

5th International Conference on Structural and Functional Intermetallics

Technical Program

Processing and Properties I

Monday AM Room: Salon 1
July 17, 2000 Location: The Westin Bayshore Hotel

Session Chairs: S. H. Whang; Shankar M.L. Sastry

8:30 AM Invited

Controlling the Properties of Some Ordered Ti-Based Alloys: *M. H. Loretto*¹; X. Wu¹; D. Hu¹; R. Botten¹; D. Horspool¹; Y. G. Li¹; ¹The University of Birmingham, IRC in Matls. for High Perform. Appls., Edgbaston B152TT UK

An extensive programme is being carried out in the IRC aimed at optimising the properties of a range of ordered Ti-based alloys. This paper will focus upon the work done on alloys based on TiAl and on alloys of the type Ti₂₅V₁₅Cr₂₋₆Al. The approach to alloy development which is being used and the range of processing techniques which have been used in an attempt to optimise the properties will be described. The work has shown that whilst it is possible to make dramatic improvements in the ductility in the alloys based on Ti₂₅V₁₅Cr₂₋₆Al by specific alloying additions, only limited improvements in TiAl-based alloys appear possible through alloy development. In this case property control is mainly obtained through the use of different processing routes. The significance of this and related work will be discussed.

9:00 AM

Lamellar Orientation Control of Ti-48Al PST Crystal by Unidirectional Solidification: *H. Morishima*¹; K. Koike¹; S. Y. Chang¹; Y. Yamamoto¹; M. Takeyama¹; T. Matsuo¹; ¹Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Control of lamellar orientation of unidirectionally solidified Ti-48 Al PST crystals has been attempted by an optical floating zone method using various seed crystals of Ti-(48-57)Al alloys. Regardless of the seed crystals, the Ti-48 Al PST crystals can be grown without any difficulty. However, the lamellar orientation can never reproducibly be controlled by using any seeds but single crystal of Ti-57 Al. In case of the 48Al PST seed, polycrystallization due to $\gamma\alpha$ phase transformation takes place in heating to melt. The seeds with composition range of a peritectic reaction ($L+\alpha\gamma$) to occur in melting exhibit a cellular liquid/solid interface, resulting in new grains there. In these cases, the growth crystal succeeds to an orientation of one of the newly formed grains. The single crystal seed of Ti-57 Al exhibits a flat liquid/solid interface in melting ($\gamma\gamma_4L$) even after making contact with 48Al alloy to grow, and the grown crystal succeeds to the seed orientation.

9:20 AM +

Microstructure Control of TiAl Alloys Including γ_2 Stabilizer by Directional Solidification: *In-Soo Jung*¹; Hong-Seob Jang²; Myung-Hoon Oh²; Je-Hyun Lee³; Dang-Moon Wee¹; ¹KAIST, Dept. of Matl. Sci. and Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea; ²KNUT, Dept. of Matl. Sci. & Eng., 188, Sinpyung-dong, Kumi 730-701 Korea; ³Changwon National University, Dept. of Metall. Eng., 9, Sarim-dong, Changwon 641-773 Korea

Phase transformation at high temperatures in the Ti-Al-X (X=Mo, Re, W) systems has been investigated in the arc-melted buttons using EDS, XRD, and DTA. The pseudo binary phase diagram of Ti-Al-2X systems has been proposed in this study. The addition of W was found to be the most effective γ_2 (bcc) stabilizer in the TiAl alloy, and the Ti-47Al-2W composition was selected for γ_2 solidification. This alloy was directionally solidified by a Bridgman-type directional solidification (DS) apparatus at the growth rate of 90 mm/hr and by a floating zone-type DS apparatus at the growth rate of 30 mm/hr. The microstructure and the lamellar orientation were observed in the directionally solidified Ti-47Al-2W alloy. Solidification microstructures have been investigated with solidification parameters by directional solidification and mechanical properties of DS ingots have been also evaluated by the tensile test at room temperature.

9:40 AM +

Stabilization of Lamellar Structure and Microstructure Control by Directional Solidification in TiAl-X-Si Alloys: *Ho-Nyum Lee*¹; David Ray Johnson²; Haruyuki Inui³; Myung-Hoon Oh⁴; Dang-Moon Wee¹; Masaharu Yamaguchi³; ¹KAIST, Dept. of Matls. Sci. & Eng., 373-1 Kusong-dong, Yusong-ku, Taejon 305-701 Korea; ²Purdue University, Schl. of Mat. Eng., West Lafayette, IN 47907-1289 USA; ³Kyoto University, Dept. Mat. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan; ⁴KNUT, Dept. Mat. Sci. & Eng., Kumi 730-701 Korea

The thermal stability of the lamellar microstructure in cast TiAl alloys containing Mo, Nb, Si and C was investigated in order to determine the processing window where the orientation of the lamellar microstructure could be controlled by directional solidification using a seeding technique. Compositions for the seeding experiments must be chosen such that original orientation of the lamellar microstructure is restored upon heating to and cooling from the melting temperature. In this study, the lamellar stability was determined by examining whether or not the lamellar structure in as-cast alloys is preserved after quickly heating into the alpha single-phase region, holding, and then cooling them to room temperature. Some prospective compositions were found in Ti-Al-Mo-Si, Ti-Al-Nb-Si and Ti-Al-Nb-Si-C systems. Adding Si was found to be crucially important to fulfil the requirements of the lamellar stability and phase equilibria. Directional solidification of these alloys will also be discussed.

10:00 AM

Directional Solidification of TiAl Base Alloys Using a Polycrystalline Seed: *S. E. Kim*¹; Y. T. Lee¹; M. H. Oh²; H. Inui³; M. Yamaguchi³; ¹Korea Institute of Machinery and Materials, Dept. of Matls. Proc., 66 Sangnam, Changwon, Kyungnam, Korea; ²Kum Oh National University of Technology, Schl. of Matls. and Metall. Eng., 188 Shinpyung, Kumi, Kyungbuk, Korea; ³Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Lamellar microstructure control and characterization of directionally solidified (DS) TiAl base alloys have been studied. DS ingots having not only fully lamellar microstructure parallel to longitudinal axis but also rotated columnar grains with respect to longitudinal axis were obtained in Ti-43Al-3Si alloys using a polycrystalline seed. It was recognized that successful seeding and growing require plane front solidification condition (very slow growth rate). DS processing type,

ram or floating zone, did not significantly influence to obtain the target microstructure. Fracture toughness of the DS alloys were superior to polysynthetically twinned (PST) alloys for the short transverse mode. Moreover, the orientation dependence of fracture toughness for the crack arrest/divide mode was improved in the DS alloys compared to the PST alloys. Such a new concept for DS technique was applied to binary and multi-component TiAl alloys and mechanical properties of the DS TiAl alloys were characterized.

10:20 AM Break

10:40 AM

Microstructures and Properties of Investment Castings of Gamma Titanium Aluminide: *Jian Ping Kuang*¹; Richard A Harding²; John Campbell²; ¹National Research Council Canada, Struct. Matls. and Propulsion Lab., Instit. for Aero. Rsch., 1500 Montreal Rd., M-13, Ottawa, Ontario K1A 0R6 Canada; ²The University of Birmingham, IRC in Materials for High Performance Application, Edgbaston, Birmingham, Birmingham B15 2TT UK

Gamma-TiAl castings have been produced by an induction skull melting (ISM) technique and their microstructures, hardness and tensile properties have been studied in the as-cast, HIPped and HIPped + heat treated conditions. HIPping and heat treatment for 24 h at 1300°C resulted in a duplex structure and decreased both the hardness and tensile properties compared with the HIPped condition. The mechanical properties also decreased with increasing bar diameter. The main fracture modes in duplex structures were transgranular in equiaxed gamma grains and translamellar in lamellar grains; the minor fracture modes were intergranular in equiaxed gamma grains and local ductile failure in lamellar grains. After deformation, there was a high density of dislocations in the gamma phase, whereas dislocations were rarely observed in the alpha-two phase. Twinning was an important deformation mode in the gamma phase.

11:00 AM

Joining between NiAl and Ni-Base Superalloys by Reactive Casting: *Kiyotaka Matsuura*¹; Masayuki Kudoh¹; ¹Hokkaido University, Div. of Matls. Sci. and Eng., Kita 13 Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628 Japan

By mixing aluminum and nickel liquids by a molar ratio of 1:1 on a base material such as Ni-base superalloys, molten nickel monoaluminide, NiAl, is exothermically synthesized in a short time of 1 to 2 s, and is joined to the base material when it solidifies. Heat generated by an exothermic reaction, Al+Ni = NiAl, leads to a high temperature exceeding 2300 K in the molten NiAl. The heat is transferred from the NiAl to the base material, and brings about the melting of the base material near the surface. The depth of the melt increases with both the preheating temperature of the base material and the thickness of the synthesized NiAl. A four-point bending test was carried out to evaluate the joint strength. Fracture did not occur at the joint interface but in the NiAl near the interface showing fracture strength of approximately 400 MPa on average.

11:20 AM

Formation of Intermetallics and Intermetallic Eutectics in Mg-Al and Mg-Al-Zn Alloys: *L. Shepeleva*¹; A. Katsman¹; B. Medres¹; M. Bamberger¹; ¹Technion-Israel Institute of Technology, Dept. of Matls. Eng., Technion City, Haifa 32000 Israel

The formation of the intermetallics in magnesium alloys determines their mechanical and corrosion properties. The intermetallics are formed in the eutectic interdendritic regions and in the matrix. The mechanism of structure formation, its stability and evolution during thermal treatment are the subjects of this paper. Specimens of Mg-Al-Zn alloys were die-cast and studied in as-cast condition and after heat treatment for 300 h and 700h at 150°C by SEM and TEM. The distribution of elements and macro-segregation were investigated by EDS SEM and EDS TEM

microanalysis. The interdendritic regions are composed of β -Mg₁₇Al₁₂- α -Mg eutectic with finesse depending on the amount of alloying elements. The coarsening of this eutectic during the heat treatment was accompanied by a change in the local concentration of Zn. The α -Mg grains were found to be enriched with the Al and Zn close to their circumference, which in its turn resulted in the precipitation of β -Mg₁₇Al₁₂ intermetallics (typical dimension 20nm) after the heat treatment. The influence of heat treatment duration on the precipitation of the b-intermetallic phase was studied. It is expected that this zone is dispersion-strengthened and influences the mechanical properties of the alloy.

11:40 AM

Properties of Eutectic Ru-Al Alloy Produced by Ingot Metallurgy: *Nenad M. Ilic*¹; Rüdiger Rein²; Flavio Andres Soldera¹; Frank Mücklich¹; ¹University of Saarland, Funct. Matls., Gebäude 22 Etage 7, P.O. Box 15150, Saarbrücken, Saarland 66041 Germany; ²Institute for New Materials, Im Stadtwald, Geb.43.2, Saarbrücken, Saarland 66123 Germany

In the Ru-Al system, intermetallic RuAl with B2 structure possesses a unique combination of properties, unusual for intermetallics such as inherent toughness at room temperature. RuAl is also characterised by high-temperature strength, high oxidation and corrosion resistance. Composite structure of RuAl and Ru solid solution is formed by eutectic reaction (~1920°C) at Ru rich portion of the Ru-Al phase diagram. According to this diagram, the eutectic point lies at 70 at.% Ru, but the microstructure of such an alloy additionally consists of RuAl primary dendrites. This could indicate that the eutectic point lies at higher Ru contents. RuAl+Ru (eutectic), combines the properties of its constituents and is suitable for application in energy generation, electrical contacts and electronics. The aim of this work was to determine eutectic composition and some of its properties at room temperature. Strength, ductility, modulus of elasticity, wear characteristic, fracture mechanism and hardness of individual phases, were determined.

12:00 PM

Prospects for Metal Injection Moulding Using a Gamma Titanium Aluminide Based Alloy Powder: *Rainer Gerling*¹; Frank-Peter Schimansky¹; ¹GKSS-Research Centre Geesthacht GmbH, Instit. for Matls. Rsch., Geesthacht 21502 Germany

Alloy powder of an advanced gamma titanium aluminide alloy (g TAB: Ti 47Al 4(Mn, Nb, Cr, Si, B) (at.%)) was produced by argon gas atomization. The fine powder (< 45 μ m) was mixed with a special binder consisting of a wax/polymer mixture and using this feedstock test specimens were moulded on a conventional moulding machine. The specimens were debinded and subsequently sintered at 1410°C. The porosity after sintering was about 3.8%. Subsequent hot isostatic pressing (hip) caused a further reduction in porosity to 0.4%. While the fine alloy powder had low oxygen and nitrogen levels (O₂: 790 mg/g, N₂: 140 mg/g), the sintered and hiped samples are characterized by a considerable increase in O₂ and N₂. Both types of impurities vary in the depth of the samples in the range of 1600 ? 8000 mg/g (O₂) and 180 ? 280 mg/g (N₂). After hip the microstructure is of duplex type. Tensile tests at room temperature resulted in a yield strength of 410 MPa, an ultimate tensile strength of 430 MPa and a plastic elongation of 0.6%.

Deformation I

Monday AM Room: Salon 3
July 17, 2000 Location: The Westin Bayshore Hotel

Session Chairs: C. T. Liu; Sharvan Kumar

8:30 AM Invited

Deformation Behavior of Differently Processed Gamma-Titanium Aluminides: Arno Bartels¹; Heinrich Kestler²; Helmut Clemens³; ¹TU Hamburg-Harburg, Matls. Sci. and Tech. Dept., Eissendorfer Str.42, Hamburg D-21073 Germany; ²Plansee AG, Tech. Ctr., Reutte A-6600 Austria; ³Universitaet Stuttgart, Institut fuer Metallkunde, Seestr. 71, Stuttgart D-70174 Germany

A short review on forging and rolling of TiAl-ingot material as well as HIPed prealloyed powder compact is presented. The ingots show a lamellar microstructure with a strong cast texture and alignment of the lamellae, resulting in strong mechanical anisotropies. Hot-forging and heat-treatment are required to obtain isotropic mechanical properties due to a more random texture. The influence of strain rate and temperature on the development of microstructure during forging was studied. The role of twinning and dynamic recrystallization will be discussed. After rolling a fine grained microstructure is observed with a strong modified cube texture which causes improved strength and creep resistance in transverse direction of the sheets. Above 1000°C the sheets can be deformed superplastically. Dynamic recrystallization produces grain sizes obeying a power law with the Zener-Hollomon parameter. In addition a bulging mechanism leads to a selection of grain orientations which intensifies the texture.

9:00 AM Invited

Deformation Mechanism Responsible for Heavy Strain of a High-Nb-Containing TiAl Alloy at Room Temperature: G. L. Chen¹; L. C. Zhang¹; ¹University of Science and Technology of Beijing, State Key Lab. for Adv. Mets. and Matls., Beijing 100083 China

At the beginning of the room temperature plastic deformation the dislocation glide is the main deformation mode of the dual-phase TiAl alloy. The 1/2[12], 1/2[110] dislocations become difficult to move because most dislocations are pinned after the 5% deformation strain. With increasing deformation strain, dislocation gliding gets more difficult and deformation twinning becomes the main deformation mode. This paper investigated the abnormal structural changes of deformation twins and twin intersections during heavy deformation at room temperature in a Ti-45Al-9Nb-2.5Mn (at. %) alloy with duplex microstructure by TEM method. Numerous bent twins can be found to propagate across the γ matrix. These twins are inclined at angles of a few degrees to the (111)_M plane. No defined habit planes of the twin boundaries can be detected. The (111)_T plane in the twins exhibits a large mis-orientation angle with the (111)_M plane in the γ matrix. Numerous 1/3[111] Frank partial dislocations, which are formed through the interfacial dislocation dissociation, were regarded as accommodating the bending of the twin boundaries. There are two types of twin intersection in γ -TiAl. Zigzag bending of the barrier twin boundary can be observed after incident twins intersected with a barrier twin. Because the incident twinning dislocations are differently oriented relative to the barrier twins both bending directions and degrees for these two types of twin intersections are quite different. Generally, the boundary bending in the type-I intersection is much larger than in the type-II intersection. For type-I twin intersection in γ -TiAl, there may form subgrains in the intersection area and a triangular region around the intersection area during heavy deformation. The triangular region around the intersection

area is supposed to be a region with the heavy lattice distortion. Under a local stress concentration, some nano-twins were observed to nucleate at the ledges on the incoherent twin boundary of the incident twin tip. These nano-twins propagate with a homogeneous 1/6[11] twinning dislocation glide mechanism within the triangular region. For type-II twin intersection in γ -TiAl, a similar triangular region, where no nano-twins exist, can also be observed around the intersection area. The much less stress concentration in type-II twin intersection may be relaxed by sending-out of dislocations from the intersection area.

9:30 AM

Stability of Ordinary Dislocations on Cross-Slip Planes in γ -TiAl: Zhijie Jiao¹; Sung H. Whang¹; Man H. Yoo²; ¹Polytechnic University, Dept. of Mech. Eng., Brooklyn, NY 11201 USA; ²Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831 USA

Ordinary dislocations in γ -TiAl were frequently observed in various cross-slip planes at high temperatures. The cross-slip behavior was repeatedly linked to anomalous hardening in γ -TiAl. In this respect, it is important to examine the stability of ordinary dislocations in terms of the line tension and the interaction energy of kink pairs or jog pairs in the cross-slip planes. The energy factors and line tension for 1/2<110> screw dislocations in γ -TiAl were calculated on the basis of the Sextic formalism. The inverse Wulff plots were applied to determine the instability of dislocations on the (111) primary glide plane. The dislocations are stable due to the convexity of the plots and always have positive line tension. The interaction energy in jog-pairs and kink-pairs on the cross-slip planes were calculated. The results show that the interaction energy was highly anisotropy in the cross-slip planes and was found to be the minimum on the (110) plane, which is consistent with the TEM observations in the literature. Temperature effects on the stability and interaction energy were also investigated using six independent elastic constants at different temperatures. The cross-slip behavior in this material at high temperatures will be discussed in terms of the line tension and interaction energy of ordinary dislocations, and the role of thermal energy. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S. Department of Energy under contract number DE-FG02-93ER45499 and DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

9:50 AM

The Role of Twinning During Room Temperature Deformation of Gamma-TiAl Based Alloys: Wilfried Thomas Marketz¹; Franz Dieter Fischer¹; Florian Kauffmann²; Thomas Bidlingmaier²; Gerhard Dehm³; Alexander Wanner²; Helmut Clemens²; ¹University of Leoben, Institute of Mechanics, Franz-Josef-Strasse 18, Leoben, Styria A-8700 Austria; ²University Stuttgart, Institut fuer Metallkunde, Seestr. 71, Stuttgart D-70174 Germany; ³Max-Planck-Institut fuer Metallforschung, Seestr. 92, Stuttgart D-70174 Germany

Deformation twinning is well known as an important deformation mechanism in gamma-TiAl based alloys. The purpose of this paper is to analyze the course of room-temperature deformation in relation to the applied stress in a polycrystalline Ti-46.5at%Al-4at%(Cr,Nb,Ta,B) alloy with near-gamma microstructure. Both, computational finite element simulations and acoustic emission measurements are performed leading to the following approaches: In the first approach twinning is incorporated into a crystal plasticity concept by considering the twin planes as additional slip planes. The second approach follows the role of twinning on the total energy consumed for a certain deformation state. This concept allows also statements regarding the twin width as well as their distance. The results of both approaches are compared and, finally, an improved understanding of twinning can be presented.

10:10 AM

Micro-Mechanics of Strengthening Effects in Lamellar TiAl Alloys: Identification of Key Microstructural Features: *B. K. Kadl¹; R. J. Asaro¹; C. T. Liu²; ¹University of California-San Diego, 409 U. Ctr., MC-0085, La Jolla, CA 92093-0085 USA; ²Oak Ridge National Laboratory, Oak Ridge, TN 37830 USA*

Fully lamellar two-phase γ -TiAl + α 2-Ti3Al alloys are strengthened primarily by three types of boundaries: grain boundaries, lamellar boundaries and domain boundaries, each of which obstruct the passage of mobile dislocations, and the volume fraction of the Ti₃Al phase in the lamellar constituent. Experimental materials processing efforts designed to produce high strength alloys till date are unable to vary these three grain sizes independently, though the current practices of refining lamellar spacing increase the yield strength. In an effort to deconvolute their respective contributions, and to identify the key microstructural parameters, yielding behavior of fully lamellar polycrystalline microstructures is simulated by Finite Element Methods. Polycrystalline stress-strain response is computed using, as independent input parameters, a range of scale dependent soft (π soft-mode) and hard (π hard-mode) mode critical resolved shear stresses, for shear parallel and perpendicular to the lamella. Our results show that increasing the hard (π hard-mode) mode CRSS, to reflect the refined lamellar spacing, by itself does not contribute to the experimentally observed strengthening. Discrepancies in such experimental versus theoretical results will be discussed, along with a ranking of key microstructural features contributing to the high yield strength

10:30 AM Break .

10:50 AM

The Mechanical Properties of Different Lamellae and Domains in PST-TiAl Investigated with Nanoindentations and Atomic Force Microscopy: *Mathias Göken¹; Markus Kempf¹; Horst Vehoff¹; ¹University of Saarland, Dept. of Matls. Sci., Geb. 43b, Postfach 151150, Saarbrücken D-66041 Germany*

A nanoindenting atomic force microscope (NI-AFM) was used to determine the hardness and modulus of polysynthetically twinned TiAl on a nanometer scale. The different properties of α and γ lamellae and domain variants in γ phase were measured with nanoindentations at load levels below 1 mN. The hardness of α lamellae is much higher than of γ lamellae, whereas different γ domains only show small hardness variations. However, the plastic anisotropy in the γ phase produces significant differences in the pile-up around the impressions left by nanoindentations. The atomic force microscope allows direct imaging of these impressions. The shapes of indents in γ lamellae deviate clearly from the triangular shape of Berkovich indenters and show a pattern which is characteristic for the orientation of the domain. In compression deformed PST crystals show a large amount of twinning in some γ domains. The results of nanoindentation measurements on these domains were compared with measurements on undeformed samples.

11:10 AM

Deformation Behavior of Large Grain, Fully Lamellar Gamma Titanium Aluminides: *W. J. Porter¹; R. John²; A. H. Rosenberger²; A. F. Lackey¹; ¹University of Dayton Research Institute, Struct. Integ. Div., Adv. Matls. Character. Grp., Dayton, OH 45469-0128 USA; ²Air Force Research Laboratory, Matls. and Manufact. Direct., AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA*

Gamma titanium aluminide (TiAl) alloys are being considered for application in rotating components of gas turbine engines. While the elastic properties of gamma and alpha-2 single-phase materials have been thoroughly investigated, the elastic response of fully lamellar two-phase materials have not received the same amount of attention. Detailed reports on the elastic property information of various gamma TiAl alloys are scarce. Colony-level properties are required for development

of accurate models in these material systems. In an effort to determine colony-level properties, samples with gage sections composed of individual lamellar grains were tested in tension to determine the elastic and plastic behavior of this two-phase structure. A variety of lamellar orientations with respect to the loading direction were investigated at room and elevated temperatures. The results of these tests and their implications to the design and use of gamma titanium components are discussed.

11:30 AM

Extension of the Concept of a Multivalley Peierls Relief for Dislocations in TiAl: *B. A. Greenberg¹; M. A. Ivanov²; ¹Russian Academy of Sciences, Instit. of Met. Phys., Ural Div., Ekaterinburg 620219 Russia; ²National Academy of Sciences of Ukraine, Instit. of Met. Phys., Kiev 252142 Ukraine*

An earlier concept (B.A. Greenberg et al., Scripta Met. 1988, 22, p. 859) included a blocking mechanism for single dislocations by immersion into deep Peierls valleys. However, the whole curve $\sigma_y(T)$ could not be explained in terms of that concept. Using TEM data on the dislocation structure evolution and comparing deformation curves for TiAl with typical curves for other materials, an attempt was made to reconstruct the shape of the potential relief for dislocations in TiAl. This relief includes potential wells of different depth, which correspond to two types of traps, and potential barriers of different height. It is emphasized that pulsation of the wide core of a dislocation, which is due to thermal fluctuations, leads to local narrowing of the core and, finally, capture of the dislocation in a deep trap. The stress-induced alteration of the potential relief near a microcrack and possible effect of this alteration on the brittle-ductile transition was analyzed.

11:50 AM

Description of a Nonmonotonic Temperature Dependence of the Yield Stress in TiAl: *B. A. Greenberg¹; M. A. Ivanov²; ¹Russian Academy of Sciences, Instit. of Met. Phys., Ekaterinburg 620219 Russia*

An explanation was proposed for the whole trend of yield stress $\sigma_y(T)$ having two extremums where $\sigma_y(T)$ changes its temperature behavior. Equations determining extremums of $\sigma_y(T)$ were established. Thermally activated blocking of dislocation sources was found to be significant. Possible shapes of the temperature dependence of the work hardening rate $\theta(T)$ in the region of an anomalous trend of $\sigma_y(T)$ were analyzed. Conditions of the anomalous behavior of $\theta(T)$ were ascertained. A paradoxical situation was explained: on the one hand, observation of an anomalous trend of $\sigma_y(T)$ and, on the other hand, TEM observation of single dislocations containing numerous pinning points and bent segments between them (S. Sriram, D.M. Dimiduk, P.M. Hazzledine & V.K. Vasudevan, Phil. Mag., 1997, A76, p.965). The discrepancy proved to be apparent and the situation could be fully described by the aforementioned equations.

Phase Stability

Monday AM
July 17, 2000

Room: Cypress 1
Location: The Westin Bayshore Hotel

Session Chairs: David Pope; Linda Horton

8:30 AM Invited

Effects of Electronic Structure on the Structural and Thermoelastic Properties of Mo-Si Compounds: *C. L. Fu¹; ¹Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831 USA*

Effects of the Mo-Mo covalent bond on the phase stability and the coefficients of thermal expansion (CTE) of Mo_3Si and Mo_5Si_3 have been examined by first principles calculations. We show that this covalent bond contributes significantly to the structural stability and the defect formation energies of A15 structure. We also address the physical origin for the high CTE anisotropy of 5-3 silicides (contrasting with nearly isotropic disilicides). The CTE anisotropy in Mo_5Si_3 is due to an elastically more rigid basal plane and a higher anharmonicity along the c-axis. As the structure of 5-3 compounds is modified from $D8_m$ to $D8\bar{y}$ by boron substitutions (Mo_5SiB_2), we predict a significant decrease in the CTE anisotropy, which is confirmed by experiments. Effects of ternary alloying additions on the CTE anisotropy will also be discussed. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S. DOE under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

9:00 AM Invited

Phase Stability of Intermetallic Compounds: *Masahiko Morinaga*¹; ¹Nagoya University, Dept. of Matls. Sci. and Eng., Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

The phase stability of intermetallic compounds is treated on the basis of electronic structure calculations by the DV-Xa molecular orbital method. First, new crystal structure maps are made since the prediction of crystal structures is indeed a first step to the design of intermetallic compounds. Two electronic parameters are introduced and used for the classification of crystal structures. One is the bond order and the other is the d-orbital energy level of elements. Both of them change following the position of elements in the periodic table. With these new parameters crystal structure maps are constructed for aluminides, silicides and some transition-metal-based compounds. There is a clear separation of the crystal structures on the maps. Second, the phase stability of hydrogen storage compounds (LaNi_5 , TiFe , ZrMn_2 , and Mg_2Ni) is treated in view of electron theory. The hydrogen absorption and desorption characteristics are very different among the compounds, but they are well understood in terms of the chemical bond between atoms and also the crystal structural evolution in the course of the hydrogenation. In addition, it is shown that the composition of hydrogen storage compounds is predictable from the ratio of chemical bond strengths between constituent metal atoms.

9:30 AM Invited

Rapid Generation of Phase Diagram Data for Development of High Performance Niobium Silicide-Based Materials: H. H. Liang¹; Minghong He²; S. -L. Chen³; M. Mendiratta⁴; *Y. Austin Chang*¹; ¹Seagate Technology, Inc., Bloomington, MN USA; ²University of Wisconsin-Madison, Dept. of Matls. Sci. and Eng., 1509 University Ave., Madison, WI 53706-1595 USA; ³National Natural Science Foundation, Beijing 100083 PRC; ⁴CompuTherm LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA; ⁵UES, Inc., 4401 Dayton Xenia Rd., Dayton, OH 45432 USA

Phase diagrams are roadmaps for materials and processing development. Yet, the traditional approach to generating these data purely based on meticulous and difficult experimental determination is time consuming and no longer done in practice. In this presentation, we will show that phase diagram data of five component niobium silicide-based materials were developed rapidly by using a phenomenological thermodynamic approach with a minimum amount of experimental phase equilibrium data from Nb-Si-Ti at one temperature. While these data may not be accurate in every detail, they do provide extremely useful guides for materials engineers to select promising alloy chemistries and processing conditions for materials development. The calculated phase diagrams of Nb-Si-Ti-Al and Nb-Si-Ti-Al-Cr will be compared with experimental measurements. This approach demonstrates a promising route to generate useful and essential phase diagram data for practical applications.

10:00 AM

The Phase Diagrams Basis for Ti-Al Based Intermetallics: *J. J. Ding*¹; P. Rogl¹; R. Podloucky¹; ¹Wien University, Institut. f. Phys. Chem., Waehringerstrasse 42, Wien A-1090 Austria

Phase relations have been derived for the ternary systems Ti-M-Al employing metallography, EPMA and X-ray diffraction techniques wherein M is a transition metal ranging from vanadium to platinum. Emphasis was lead on the solubility of M in the binary Ti-Al aluminides such as α_2 -Ti₃Al and γ -TiAl. Furthermore from Rietveld refinements, we established the crystal structures for all the ternary compounds and solutions which are in equilibrium with α_2 and γ . The following structure types were encountered: AuCu₃-type, AuCu-type, Th₆Mn₂₃₊₁-type and MgZn₂-type. Based on the isothermal sections derived as well as on the characteristic of the structure types established, systematics are presented for phase equilibria and for structure types.

10:20 AM Break

10:40 AM

Ordering and Phase Separation of BCC Aluminides in (Ni, Co)-Al-Ti System: Kiyohito Ishida¹; Hajime Mitsui¹; Ikuo Ohnuma¹; *Ryosuke Kainuma*¹; Kiyoshi Aoki²; ¹Tohoku University, Dept. of Matls. Sci., Aramaki Aoba-yama 02, Sendai, Miyagi Prefecture 980-8579 Japan

B2 aluminide in Ni-Al and Co-Al systems is expected to be applied for high temperature structural materials because of its low density, high melting point etc. It has also been reported that the precipitation of L₂₁ phase in the B2 phase increases the creep strength at high temperatures in the Ni-Al-Ti system. In the present study, the phase equilibria and ordering reaction between B2 and L₂₁ phases have been determined in the (Ni,Co)-Al-Ti quaternary system. It is shown that the two phase region of B2+L₂₁ is narrowed and then disappears by increasing the Co content or temperature, indicating that the tricritical and summit temperatures of B2/L₂₁ ordering transition of Ni-Al-Ti ternary alloys are lowered by the Co addition. The phase stability of these aluminides is also discussed and compared with those of the (Ni, Fe)-Al-Ti system.

11:00 AM

Phase Equilibria at 1100°C in the Nb-Ti-Al System: *Keith J. Leonard*¹; Vijay K. Vasudevan¹; ¹University of Cincinnati, Dept. of Matls. Sci. and Eng., 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA

The phase equilibria at 1100°C has been examined as part of a larger investigation of the phase equilibria and transformations within the Nb-Ti-Al system. Fifteen alloys ranging in composition from 15 to 40 at.% Al, with Nb:Ti ratios of 1:1.5 up to 4:1, were prepared by arc-melting. All alloys were homogenized and solution treated in the β solid solution phase field prior to treatment at 1100°C. The alloys were characterized by optical microscopy, x-ray diffraction, electron microprobe, scanning and transmission electron microscopy. The phase equilibria will be discussed along with a comparison to previous experimental work. In addition, the results of differential thermal analysis and the movement of the three-phase fields in the Nb-Ti-Al system will be addressed.

11:20 AM

The Observation of a New Tetragonal Phase in the Nb-Ti-Al System: *Keith J. Leonard*¹; Vijay K. Vasudevan¹; ¹University of Cincinnati, Dept. of Matls. Sci. and Eng., 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA

The phase equilibria and transformations within three Nb-(24-36)Ti-40Al alloys (in at.%) have been examined as part of a larger investigation of Nb-rich alloys of this system. A new phase was discovered following aging treatments of the β_0 phase at temperatures below 1100°C. The new θ -phase was determined to have a body centered tetragonal structure with the I4₁/amd space group (#141) and lattice

parameters near $a_0=5.106\text{\AA}$ and $c_0=28.168\text{\AA}$. The θ phase was observed to preferentially form with the γ -TiAl phase instead of σ -Nb₂Al through a cooperative growth mechanism, however, both long term and two-step heat treatments indicate a possible three-phase equilibrium field between these structures. In addition, retained regions of β_0 were observed to undergo an ω -type transition beyond the ω'' structure into the hexagonal D8₈ phase. The formation of the hP18 form of the D8₈ structure within the Nb-rich alloys will be discussed.

11:40 AM

The Influence of Alloying Elements on Phase Constitution and Microstructure of Mo₃M₂Si₃ (M = Cr, Ti, Ni, Nb, or Co): Erik Ström¹; Zhang Ji²; Changhai Li¹; ¹Chalmers University of Technology, Eng. Mets. Dept., Hörsalsv. 7, Gothenburg 41296 Sweden; ²Central Iron and Steel Research Institute, Dept. of Superalloys, 76 Xue Yuan Nan Rd., Beijing 100081 China

Mo₃Si₃ is considered as an ultra-high temperature material for its excellent strength retention at temperatures over 1200°C. Furthermore, it has potential to be intrinsically toughened since it has a large bulk to shear modulus ratio. At first, the phase constitution and microstructure of the alloyed systems need to be understood. In this work, 6 different alloying systems were designed, using the alloying elements Cr, Ti, Ni, Nb and Co, with Mo₃Si₃ as a reference. The materials were synthesised by arc-melting followed by annealing at 1600°C. Alloying additions introduced several non-equilibrium phases in the as-cast material. After annealing the composition gradients became less pronounced. Cr, Ti, Ni, Nb and Co introduced new phases in both the cast and the annealed microstructures with considerable amounts of alloying elements dissolved into the major phase. It is concluded that Mo₃Si₃ can dissolve meaningful amounts of the selected alloying elements. Thus, alloying seems promising for Mo₃Si₃-based materials.

12:00 PM

Platinum Alloys based on Pt-Pt₃Al for Ultra-High Temperature Use: Patricia Joan Hill¹; Ira Wolff¹; ¹Mintek, Phys. Metall. Dept., Private Bag X3015, Randburg, Johannesburg, Gauteng 2125 South Africa

Platinum-aluminium alloys based on the L1₂ compound Pt₃Al have potential as high-strength alloys with superior environmental resistance at ultra-high temperatures. Two-phase microstructures, analogous to the nickel-base superalloys, and consisting of the intermetallic compound Pt₃Al and the (Pt) solid solution, can be engineered to have the attributes of microstructural stability, environmental resistance, high-temperature strength and room-temperature ductility. Pt₃Al exists as a tetragonal phase below 1290°C, and ternary alloying is employed both to stabilise the L1₂ crystal form of Pt₃Al, and as a solid-solution strengthener of the (Pt) phase. In this investigation, the phase relations of the Pt-rich corners of eight Pt-Al-X ternary systems are characterised. The solubility of element X in (Pt) and Pt₃Al, and the effect on the phase boundaries is assessed using SEM-EDS. XRD is used to assess which crystal variant of Pt₃Al was stabilised by element X. Preliminary mechanical testing was carried out on the alloys. This assessment has been used to select the most promising systems for further characterisation.

Hydrogen Storage I and Magnetic Properties

Monday AM
July 17, 2000

Room: Cypress 2
Location: The Westin Bayshore Hotel

Session Chair: M. Yamaguchi; M. Takeyama

8:30 AM Invited

Role of Intermetallics in Hydrogen Storage Materials: M. Okada¹; T. Kuriwa¹; A. Kamegawa¹; H. Takamura¹; ¹Tohoku University, Dept. of Mats. Sci., Grad. Schl. of Eng., Sendai 980-8579 Japan

Many Intermetallics show the excellent hydrogen absorbing properties such as s AB₅, AB₂, A₂B. Specially, LaNi₅ compound has been extensively studied for the negative electrode of Ni-MH batteries. In today's use for the negative electrode of Ni-MH batteries, alloys must be single phase. But it is well-known that it is difficult to make non-stoichiometric LaNi₅ alloys as single phase because of its limited solid-solution range. The present study describes the rapid quenching effects on the solid-solution range of La(Ni, M)_x ($x=3\text{?}5$) alloys prepared by melt-spinning in discussing their hydrogen absorption properties. It is found that the single phase with CaCu₅ crystal structure extends to LaNi_{4.65} compositional alloys. For fuel cell automobile, the alloys with high content of hydrogen must be developed. Strong candidates for this will be Vanadium based BCC alloys, which will be hard to be activated. Since it is well known that the Laves phases undergo easy activation treatment, it may be possible that the BCC phases will be easily activated if the alloys contain such Laves phases. The present study also found that Zr addition is effective on forming Laves phase as grain boundary phases in BCC phases in V-Ti-Zr-Ni alloys, and on improving the hydrogen absorbing properties. The study will be also extended to future hydrogen absorbing alloys with high capacity.

9:00 AM Invited

Properties of Intermetallic Compounds Suitable for Hydrogen Storage Applications: P. Dantzer¹; ¹LEMHE, UMR 8647, Université Paris-Sud, B t415, Orsay, Cedex 91405 France

Hydrides of intermetallic compounds belong to the class of functional materials whose possible applications cover a wide temperature range, from a few Kelvin up to 500K and therefore accurate and extensive thermodynamic characterisation is the prime criterion because it determines the useful domain of the hydrogen storage system. Each application is correlated to a well defined property, i.e. chemical reactivity for purification, temperature/pressure relationship for sensors, hydrogen storage capacity converted into energy for hydride chemical engines, or converted into electrical current for hydride batteries. Up to now, only the hydride batteries have reached the consumer level. For hydrides obtained by direct reaction between hydrogen gas and the parent solid phase, applications have not been successful suggesting thus that many problems remain to be solved. We will be concerned by the factors influencing the absorption properties according to the type of application. It will also be shown that the dynamic behaviour of these highly defective materials is difficult to control due to the hysteresis accompanying the formation-decomposition of the hydrides compounds. Hysteresis reflects irreversibility and contributes to the degradation of the absorption properties over the long term behaviour.

9:30 AM Invited

Crystal Structural Studies of Ternary Metal Hydrides: Etsuo Akiba¹; Hirotohi Enoki¹; Yumiko Nakamura¹; ¹National Institute of Materials

and Chemical Research, Div. of Inorg. Matls., 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

Our recent progress on crystal structural studies on hydrogen absorbing alloys including intermetallics and solid solutions is reviewed. 1) LaNi_5 shows an extremely high equilibrium pressure at the first hydrogen absorption but not at the second one. The structural change in the first absorption was followed by in-situ X-ray diffraction. We found that the crystalline size did not change but the anisotropic strain was introduced to the lattice of the hydride formed. The strain introduced was not relaxed in the following dehydrogenation and hydrogenation. 2) The crystal structures of Laves phase related BCC alloys and their hydrides were refined by the Rietveld method using in-situ X-ray and neutron powder diffraction data. It was confirmed that these alloys had bcc structure in real. Monohydride ($H/M=1$) of the alloy had the distorted bcc structure and full hydride ($H/M=2$) had the fcc structure.

10:00 AM Break

10:20 AM Invited

Mechanical Properties of Soft Magnetic FeCo Alloys: *E. P. George*¹; A. N. Gubbi²; I. Baker³; ¹Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831-6093 USA; ²Dentsply International, 1301 Smile Way, York, PA 17404 USA; ³Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755-8000 USA

The brittle fracture of FeCo has long been a puzzle given its high-symmetry B2 crystal structure, $1/2\langle 100 \rangle\{110\}$ slip, low ordering temperature, and relatively low strength. Macroalloying with vanadium (~2%) improves ductility significantly in the disordered state but only moderately in the ordered state. Brittle fracture can be intergranular in the binary alloy but is usually by transgranular cleavage in the V-containing alloy. Microalloying with boron and carbon have been shown to produce significant ductility improvements in the V-containing alloy. The mechanism appears to be related to slip refinement by fine precipitate distributions. Significant strength improvements have also been achieved in some of these ductile alloys. So far there is no evidence of environmental effects leading to premature fracture in this alloy system. We review here the effects of alloying additions and deviations from stoichiometry on the deformation and fracture behavior of FeCo alloys. Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation and contract DE-FG02-87ER45311 with Dartmouth College, and through the SHARE program under contract DE-AC05-76OR00033 with the Oak Ridge Associated Universities.

10:55 AM

On a Model for the Strain-Induced Paramagnetic to Ferromagnetic Transition in FeAl: *Ian Baker*¹; Dongmei Wu¹; ¹Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

FeAl single crystals of various compositions were cold rolled to different strains and their magnetic susceptibility measured using a vibrating sample magnetometer at temperatures from room temperature to 77K. Cold work induces a transition from paramagnetic to ferromagnetic behavior, whose Curie temperature depends on strain. The stored energy due to the cold work was also determined by slowly heating specimens in a differential scanning calorimeter. Three exothermic peaks were observed. Annealing above the lowest temperature (~500K) peak largely returns a crystal to its paramagnetic state. (The two higher temperature peaks appear to be associated with vacancies. None of the peaks are due to recrystallization.) A model is presented which explains the ferromagnetic behavior in terms of "wrong" bonding in antiphase boundaries (APB's), principally in APB tubes. This research was supported by National Science Foundation grant DMR 9973977 and U.S. Department of Energy grant DE-FG02-87ER4311.

11:10 AM

Fabrication of NiAl Microreactors: *David E. Alman*¹; Tyler Dewey²; Rick D. Wilson¹; Brian K. Paul²; ¹U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave. S.W., Albany, OR 97321 USA; ²Oregon State University, Indust. and Manufact. Eng., Corvallis, OR 97331 USA

Chemical microreactors offer opportunities for portable powder generation, on-site waste remediation, point-of-use chemical synthesis, and heat-transfer. The material requirements for this application include chemical inertness and the ability to be fabricated into structures that contain internal features of complex geometries and small (<250 μm) dimensions. It has been recognized that materials with limited formability, like ceramics and intermetallics, will be required for high temperature applications. Reactors from these materials have been produced from powder tapes. However, problems associated with binder removal and sintering result in dimensional instability of the internal geometry which degrades the performance of the reactor. In this paper, a method for forming an array of internal microchannels in a NiAl device that avoids the dimensional instability of powder processing is demonstrated. Microchannels are precision machined (via laser ablation) into elemental Ni and Al foils. During bonding, the foils are converted into NiAl. Results show that this is a viable method for producing aluminate-based structures containing complex, internal features.

11:30 AM

Effect of Ti, V, Cr, Mn and Ni on the Structure and Magnetic Properties of Fe_3Al Alloys: *B. V. Reddy*¹; S. C. Deevi¹; P. Jena²; ¹Philip Morris USA, Rsch. Dev. and Eng. Ctr., Richmond, VA 23234 USA; ²Commonwealth University, Dept. of Phys., Richmond, VA 23284 USA

The intermetallic Fe_3Al stabilizes in the DO_3 ordered phase with two kinds of Fe sites. While Fe_I is surrounded by eight Fe neighbors, the other site, Fe_{II} has four Fe and four Al neighbors. X-ray absorption fine structure and Mossbauer experiments suggest preferential site occupancies of the additive elements Ti, V, Cr, Mn and Ni in the host Fe_3Al . The elements to the left of Fe in the periodic table seem to prefer the Fe_I site, while the elements to the right of Fe preferentially occupy the Fe_{II} sites. A systematic investigation on the effect of additives Ti, V, Cr, Mn and Ni on the geometry, energetics, electronic and magnetic structure of Fe_3Al alloys is made employing the density functional theory with generalized gradient corrections. A key objective of the study is to obtain a fundamental understanding of the bonding and site preference of these additives at the electronic level. Further an attempt is made to understand the variations in the magnetic and the electronic structure of the host alloy as a function of the impurity. Varying degree of directional charge distributions and p-d hybridizations at the Fermi level are investigated. The structural deformations, and changing magnetic character accompanying various additives are highlighted using the total energy calculations.

11:50 AM

Intermetallics with a Giant Magnetoelastic Response: *Vladimir Andreyevich Chernenko*¹; ¹Institute of Magnetism, Magnetostructure, Transformation, Vernensky str., 36, Kiev 03142 Ukraine

A novel group of magnetic field operated functional materials based on ferromagnetic intermetallics exhibiting large magnetic field induced strains ($\geq 1\%$) at the ambient temperatures is considered. In contrast to the ordinary magnetostrictive materials the distinguishing feature of these intermetallics is that they undergo a first order martensitic (Ni_2MnGa , Fe_3Pt) and magnetostructural (FeRh , MnAs , TbMn_2) phase transformations whereby the large field-induced deformations associated with the transformation strain are attainable. The magnetoelastic response and magnetic field influence on the martensitic transformation in single crystalline Ni_2MnGa alloy has been studied experimentally.

The Landau theory and concept of “equivalent stresses” has been developed to describe the giant magnetoelastic response in martensitic state and in the vicinity of transformation temperature. The dilatometric measurements of mechanically alloyed MnAs under applied magnetic field have been performed at constant temperatures. The magnetic field induced strain of 0.7% and its temperature evolution has been found in this compound as result of a large spontaneous volume magnetostriction accompanying field induced magnetostructural transition above Curie temperature. Experimental results are explained within a Landau approach.

Processing and Properties II

Monday PM Room: Salon 1
July 17, 2000 Location: The Westin Bayshore Hotel

Session Chairs: Norman Stoloff; Toshiyuki Hirano

1:30 PM Invited

EPM Method of Synthesizing Intermetallics Based on Ti: Hyun-Soon Park³; Kang-Le Park²; *Sun-Keun Hwang*¹; ¹Inha University, Matls. Sci. and Eng. Depts., 253 Yonhyun-Dong, Nam-Gu, Incheon 402-751 Korea; ²Shinhan Diamond Industrial Company Limited, 610-9 Namdong-Gu Namchon-Dong, Incheon 405-100 Korea

Elemental powder metallurgy (EPM) is a powerful means of synthesizing intermetallics of high melting points. In this study, two intermetallic compounds based on Ti were investigated using different consolidation techniques: TiAl-Mn-Mo-C by hot extrusion and Ti₅Si₃-Nb-C by electro-pressure sintering (EPS). Full density compounds were obtainable in both cases. Aided by interstitial carbon atoms, each intermetallic compound showed attractive mechanical properties such as high tensile yield strength and creep resistance in TiAl-Mn-Mo-C and high fracture toughness and transverse rupture strength in Ti₅Si₃-Nb-C. The roles of carbon atoms, being less than one atomic pct in each material system, were: refinement of the lamellar microstructure and precipitation hardening in the former, and a possible modification of the crystal structure towards easier slip and less thermal anisotropy in the latter.

2:00 PM

Mechanism of Combustion Synthesis of NiAl: *Ping Zhu*¹; J. C.M. Li¹; C. T. Liu²; ¹University of Rochester, Dept. of Mech. Eng., Matl. Sci. Pgm., Rochester, NY 14627-0133 USA; ²Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

The combustion synthesis of multilayer Ni/Al system was conducted in vacuum under a near adiabatic condition. The temperature change was continuously recorded by a thermocouple. In addition, a fast quenching technique and a microanalytical method were used to identify the intermediate phases of the reaction. The experimental results clearly show that the combustion reaction starts right after the melting of aluminum. Then the reaction goes through three stages. In the first stage, the temperature rises from the melting point of aluminum to the decomposition temperature of intermediate phase NiAl₃, i.e. 854°C. The reaction is the dissolution of nickel in liquid aluminum, with the formation of intermediate phases NiAl₃ and Ni₂Al₃ at the solid-liquid interface. In the second stage, the temperature of the system increases from 854°C to about 1300°C. The reaction is still the dissolution of nickel in liquid aluminum solution. However, because of the absence of intermediate phase at the interface, the dissolution mechanism is different from the first stage. The third stage starts at about 1300°C, and ends at the maximum reaction temperature. The temperature rise at this stage is

much faster than those of the first and second stages. The final product, NiAl, forms at this stage. This research was sponsored by the Division of Materials Science, U.S. Department of Energy under Contract DE-AC05-84OR21400 with Martin-Marietta Energy Systems, Inc.

2:20 PM

Reaction Synthesis and Sintering of Fe-40%Al: *Shalva Gedevanishvili*¹; Seetharama C. Deevi¹; ¹Philip Morris USA, Rsch. Ctr., 4201 Commerce Rd., Richmond, VA 23234 USA

Iron aluminides based on FeAl are attractive as structural materials because of their outstanding resistance to high temperature oxidation, sulfidation, and corrosion. Lack of processing techniques hampered their extensive use for structural and functional applications. In the present work, we discuss the reaction synthesis of FeAl from Fe and Al followed by pressureless sintering. Mechanism of formation of FeAl alloy from elemental powders was dependent on the temperature and rate of heating. Synthesis is accompanied by rapid expansion of the sample at heating rates >1°C min⁻¹, while the expansion is less dramatic at heating rates <1°C min⁻¹. Initial powder undergoes phase transformation through an intermediate phase Fe₂Al₅. Sintered density of up to 95% can be reached by sintering the powders for 3 hours at 1350°C. X-ray analysis, DSC measurements and microstructure were correlated to the dilatometric measurements of the thermal expansion, and shrinkage of the samples.

2:40 PM

Swelling in the Fe-Al Reactive Sintering: *Chen-Ti Hu*¹; *Hui-Zhen Kang*¹; ¹National Tsing Hua University, Dept. of Matls. Sci. and Eng., 101 Sec. 2, Koug Fu Rd., Hsinchu 30043 Taiwan

The swelling and porosity are always the problems in a stress-free sintering experiment on iron-aluminum powder mixtures. The combination of imbalanced solubilities and diffusivities leading to biased flow of aluminum into the iron matrix was suggested as the causing of this swelling. Our study on vacuum stress-free sintering of iron-aluminum mixtures with various iron powder sizes indicates a transient phase, Fe₂Al₅, which develops at low temperature. The examinations of dimensions, phases and microstructures have been carried out at various temperatures. It is strongly believed that the appearing of this transient phase plays an important role in the expansion and swelling behaviors of iron-aluminum compacts.

3:00 PM

Exothermic Behaviors of Several Elemental Powders Mixes: *Chen-Ti Hu*¹; Sanboh Lee¹; ¹National Tsing Hua University, Dept. of Matls. Sci. and Eng., Hsinchu 30043 Taiwan

The exothermic behavior study on elemental powders mixes with compositions of Ni₇₅-Al₂₅, Fe₇₅-Al₂₅ and Ni₅₀-Ti₅₀ etc. was carried out in a vacuum (8 x 10⁻⁶ torr) atmosphere and an in-situ monitored thermal couple. The maximum temperatures and the durations of exothermic process for various mixtures with or without a stainless steel sheath were measured and compared. It is observed that maximum temperature of Ni-Al compacts is effectively depressed by an equal-weight outer sheath, hence the reacted phase structure varies with the contacting of stainless steel sheath. No significant modification on either the maximum temperature or the reacted phase has been found on the Fe-Al powder compacts while contacted with the outer sheath. On the other hand, a tremendous exothermic heat was released as the Ni-Ti mixture been heated to 1100°C. These exothermic behaviors are difficultly observed with a DSC experiment.

3:20 PM

Combustion Synthesis of 25Nb75Al Powder Mixtures after High-Energy Ball Milling: *Ricardo Mendes Leal*¹; Cláudio José da Rocha¹; Fabio Luigi Hanna Venturacci¹; ¹Instituto de Pesquisas Energéticas e

Nucleares, Departamento de Engenharia e Ciência dos Materiais-MM, Travessa R 400, Cidade Universitária, São Paulo SP 05508-900 Brazil

This work investigates the influence of High-Energy Ball Milling (HEBM) on combustion synthesis of 25Nb75Al powder mixtures (NbAl₃ stoichiometry). This was accomplished by performing Differential Thermal Analysis (DTA) and by reacting compacted samples after milling. HEBM was performed in a laboratory shaker mill, changing only the milling time in order to get a refined dispersion with no reaction. A non-milled sample was added for comparing results. DTA was conducted on loose powder samples at a fixed heating rate under argon flux up to 1100°C. Compacted milled samples were reacted at the same heating rate by simultaneous combustion mode under vacuum. Pellets density was measured before and after the reaction for densification evaluation. X-ray diffraction and SEM analysis were conducted for identifying reaction products. Results shown a drastic change on the ignition temperature and on the reaction evolution of the NbAl₃ synthesis as milling time is increased. These changes are discussed along with the densification behaviour observed.

3:40 PM Break

4:00 PM

Processing, Properties, and Fabrication of Nickel and Iron Aluminides: *V. K. Sikka*¹; M. L. Santella¹; ¹Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, Oak Ridge, TN 37831 USA

The Ni₃Al-based nickel and FeAl-based iron aluminides have characteristic properties that make them suitable for a broad range of applications. The component size for the applications can vary from grams to tons and correspondingly the shape can vary from a simple cylinder to very complex shapes. This presentation will provide an update on the processing and properties of Ni₃Al- and FeAl-based aluminides with special emphasis on methods for the fabrication of components. Several applications will also be discussed. Research supported by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Advanced Industrial Materials Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

4:20 PM

HIP Temperature and Properties of Gas-Atomized-Titanium Aluminides: *Ulrike Habel*¹; Brian J. McTiernan¹; ¹Crucible Research Center, 6003 Campbels Run Rd., Pittsburgh, PA 15205 USA

Inert gas atomization is increasingly used for gamma titanium aluminides. Its inherently high cooling rate suppresses segregation and results in uniform composition. Consolidation by hot isostatic pressing (HIP) leads to fully dense solids with isotropic and homogeneous microstructure and properties. The HIP process is conducted in the alpha+gamma or alpha+gamma+beta phase region, and some work has been published on the effect of HIP temperature on mechanical properties, but it is restricted to the investigation of as-HIP microstructure and properties. It is well known that the mechanical properties of gamma-TiAl based alloys vary with their microstructure. Since the as-HIP microstructure depends on the specific location in the alpha+gamma+beta phase field, the effect of HIP-temperature can not be isolated from the effect of microstructure on properties and investigations of as-HIP gamma-titanium aluminides. In this study, Ti-47Al-2Cr-2Nb and other gamma-TiAl alloys were HIP'ed at a range of temperatures and subsequently submitted to the same heat treatment. Microstructure and tensile properties in the as-HIP and HIP plus heat treated conditions are discussed.

4:40 PM

Effect of MA on Microstructure and Synthesis Path of In-situ TiC Reinforced Fe-28at%Al Intermetallic Composites: *Se-Hyun Ko*¹; Bong-Gyu Park¹; Hitoshi Hashimoto¹; Toshihiko Abe¹; Yong-Ho Park¹; ¹Tohoku National Industrial Research Institute, Matls. Sys. Div., 4-2-1 Nigatake, Miyagino-ku, Sendai 983-8551 Japan

The microstructure and synthesis path of in-situ TiC reinforced Fe-28mol%Al manufactured by mechanical alloying-pulse discharge sintering (MA-PDS) processes were examined as a function of milling time and sintering temperature. The microstructure of composites made by MAed powders consists of three regions, that is, large TiC particles of about 5µm in size, matrix with TiC particles of submicron and particle-free region. On the other hand, the composites made by mixed powders showed homogeneously distributed TiC particles of 1-20µm, where mixed Fe₃Al+TiC phases were surrounded. The MA of elemental powders induced extended solid solution of Fe with bcc structure. In powders mixed or MAed for short times, intermediate phases, such as Al₅Fe₂ and Al₃Ti, were formed when heat-treated at 673K and 873K, while Fe₃Al+TiC phases were synthesized after heat treatment at or above 1073K. On the contrary, powders MAed at or above 200hr transformed directly from bcc solid solution to ordered Fe₃Al+TiC phases.

5:00 PM

Fabrication and Characterization of Intermetallic Matrix Composites by MA-PDS Process: *Bong Gyu Park*¹; Se-Hyun Ko¹; Toshihiko Abe¹; Yong-ho Park¹; ¹Tohoku National Industrial Research Institute, Matls. Sys., 4-2-1 Nigatake Miyagino-ku, Sendai, Japan

Iron aluminides are of considerable interest due to their low cost, relatively high melting point, relatively low density, and excellent resistance to oxidation, sulfidation and molten salts. However, poor ductility and fracture toughness at room temperature hinder their usage as a structural material. Grain refinement can reduce hydrogen induced brittle intergranular fracture. The advantage of mechanical alloying (MA) is that small particle sizes distributed homogeneously can be obtained. On the other hand, the advantage of Pulse Discharge Sintering (PDS) is that spark generated during sintering broke the surface oxide layer and sintering was much faster. This can reduce the sintering time and suppress the grain growth of final product. Therefore, combination of MA and PDS processes resulted in very fine microstructure of final product. Fe-28at.%Al matrix composites reinforced with submicron size TiB₂ particles. Room temperature mechanical properties were increased with the particle volume fraction.

5:20 PM

Fabrication of TiB₂ Reinforced Al₃Ti Composite Layer on Ti Substrate by Reactive-Pulsed Electric Current Sintering: *Toshio Matsubara*¹; Tomohide Shibutani¹; Keisuke Uenishi¹; Kojiro F. Kobayashi¹; ¹Osaka University, Dept. of Manuf. Sci., 2-1 Yamadaoka, Grad. Sch. of Eng., Suita, Osaka 565-0871 Japan

A TiB₂ reinforced intermetallic compound Al₃Ti composite was formed by reactive-pulsed electric current sintering of ball-milled powders, and was simultaneously joined with Ti substrate to improve its wear and oxidation property. By preparing the two kinds of precursor ball-milled powders starting either from mixing Al, Ti and TiB₂ or from mixing elemental Al, Ti and B, the homogeneous Al₃Ti layer with finely dispersed TiB₂ was fabricated. The desiccation behavior of the surface layer, as well as the reaction between surface layer and substrate were investigated. While the wear property of Ti substrate could be improved by the reinforcement of TiB₂, the oxidation property at high temperature was degraded by the decomposition of TiB₂.

Deformation II

Monday PM Room: Salon 3
July 17, 2000 Location: The Westin Bayshore Hotel

Session Chairs: T. G. Nieh; Fritz Appel

1:30 PM

Saturation of Yield Stress and Embrittlement in Fine Lamellar TiAl Alloy: *Kouichi Maruyama*¹; Mayumi Suzuki¹; Hiroyuki Sato²; ¹Tohoku University, Dept. of Matls. Sci., Aobayama 02, Aoba-ku, Sendai 980-8579 Japan; ²Hirosaki University, Dept. of Intell. Mach. and Sys. Eng., Hirosaki 036-8561 Japan

Mechanical properties of a Ti-39mol%Al alloy with fully lamellar microstructure were studied at room temperature and 950K paying special attention to effects of lamellar spacing. Lamellar spacing ranging from 20nm to 600nm was made by isothermal aging at various temperatures, keeping the same grain size of 90micron m. Yield stress of the alloy increases with decreasing lamellar spacing, and the Hall-Petch relation holds over the range of lamellar spacing greater than 100nm. Contrary to the beneficial effect of lamellar refinement, ductility is reduced by the refinement. The yield stress saturates at a value of about 1GPa below the critical lamellar spacing. Causes of the saturation of yield stress and the embrittlement will be discussed in conjunction with the deformation modes of the fine lamellar microstructures.

1:50 PM Invited

Mechanical Properties of Nanocrystalline Ti-Al-X Alloys: *Hector A. Calderon*¹; *Vicente Garibay-Febles*¹; Minoru Umemoto²; Jose Gerardo Cabanas-Moreno¹; ¹ESFM-IPN, Dept. Ciencia de Materiales, UPALM Ed. 9, Apdo. Postal 75-707, Mexico, DF 07338 Mexico; ²Toyohashi University of Technology, Production Systems, Toyohashi, Japan

Nanocrystalline alloys have been produced by means of mechanical milling and spark plasma sintering. Two types of materials have been obtained i.e., Ti-Al-X and TiAl₃-X alloys, X represents Cr, Mn or Fe. Sintered Ti-Al-X alloys have a two phase microstructure consisting of the γ -TiAl phase and the α_2 phase, this last one with a globular morphology. Their average grain size is approximately 150 nm. The TiAl₃-X alloys are constituted by a single phase with an L1₂ structure and an average grain size of 30 nm. Compression tests are used to evaluate the mechanical properties of these materials at temperatures ranging from 298 to 773K. a slight positive dependence of the yield strength with temperature is observed in the Ti-Al-X alloys. Surface traces develop during deformation at room temperature of these materials. Microscopic observation reveals the formation of slip bands that originate by dislocation motion in the larger grains. The TiAl₃-X alloys show no ductility and a rather high strength.

2:20 PM

The Yield Strength Anomaly in the B2 Compound CoTi: *Markus W. Wittman*¹; *Ian Baker*¹; *Pier Bove*¹; ¹Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Mechanical tests have been performed on polycrystalline stoichiometric B2-structured CoTi as a function of temperature at different strain rates. The aim was to determine whether the George-Baker vacancy-hardening model (Phil. Mag., 77 (1998) 737), which was developed for FeAl, is generally applicable to the yield strength anomaly observed in some B2 compounds. The temperature of the yield stress peak in CoTi changes with changes in strain rate but the magnitude appears to be only weakly strain rate dependent. Also, unlike FeAl, the yield stress peak does not disappear at slow strain rates. Quenching

experiments suggest that the vacancy concentrations at elevated temperature are much lower in CoTi than in FeAl, and probably too low for vacancy hardening to be appreciable. The mechanical behavior has been correlated with observations of dislocations in the transmission electron microscope. This work was funded by the National Science Foundation, Division of Materials Research through grant DMR-9812211 with Dartmouth College.

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Yield Stress Anomaly in B2 FeAl: *Olivier Calonne*¹; *Anna Fraczkiewicz*¹; *Francois Henri Louchet*¹; ¹INPG, LTPCM/ENSEEG, Domaine Universitaire, BP 75, St Martin d'Herès 38402 France

The stress anomaly of B-free and B-doped B2 FeAl alloys is investigated through mechanical testing between RT and 900°C. In both alloys, the anomaly is associated with a very low strain rate sensitivity. Boron addition significantly increases the stress peak temperature T_p and the corresponding stress maximum. TEM observations show that the slip direction changes from $\langle 111 \rangle$ below T_p to $\langle 100 \rangle$ above. $\langle 110 \rangle$ dislocations are also observed around T_p in the B-doped material, as elongated edge dipoles. The results are consistent with a model based on Yoshimi's decomposition scheme of $\langle 111 \rangle$ superdislocations into $\langle 110 \rangle$ and $\langle 001 \rangle$, resulting in superdislocation locking. The locking rate increases with temperature, and is balanced by multiplication, which yields both a stress anomaly and a zero strain rate sensitivity. Boron is supposed to segregate on $\langle 110 \rangle$ edges, which hinders multiplication and therefore shifts towards higher temperatures the alternative deformation mechanism involving $\langle 110 \rangle$ and $\langle 001 \rangle$ glide.

3:00 PM

Cold-Work Induced Phenomena in B2 FeAl Intermetallics: *Robert A. Varin*¹; *Tomasz Czujko*²; *Jerzy Bystrzycki*²; *Andrzej Calka*³; ¹University of Waterloo, Mech. Eng. Dept., 200 University Ave.W., Waterloo, Ontario N2L 3G1 Canada; ²Military University of Technology, Matls. Tech. Div., S. Kaliskiego 2, Warsaw 00-908 Poland; ³University of Wollongong, Matls. Eng. Dept., Northfields Ave., Wollongong, New South Wales 2522 Australia

Cold-workability of B2 iron aluminides (FeAl) by conventional cold-working operations such as rolling, forging, extrusion etc. is very difficult, resulting ultimately in severe cracking. This hampers any attempts to process iron aluminides by static recrystallization. In turn, unconventional methods of cold-work such as mechanical (ball) milling and shock-wave loading (explosive) can induce severe deformation to iron aluminides either in their powder or bulk form, respectively. For the last several years we have been conducting systematic studies of microstructural changes and mechanical behavior of B2 FeAl alloys, cold-worked by both ball-milling and shock-loading. It has been observed that several fundamental physical phenomena, common to all these methods of cold-working, can be induced in iron aluminides. The most typical ones are an accelerated chemical disordering, increase in the lattice parameter, non-magnetic/magnetic transformation and formation of nanocrystals in ball-milled powder particles. As a rule, a substantial increase in mechanical strength, reflected in increasing microhardness, is associated with all these phenomena. In this work, the results of our studies of fundamental phenomena observed in heavily cold-worked powders and shock-loaded bulk specimens of B2 FeAl with varying Al concentration are presented and discussed

3:20 PM Break

3:40 PM

Mechanical Properties of Mo₅SiB₂ Single Crystals: *Keisuke Ihara*¹; *Kazuhiro Ito*¹; *Lanting T. Zhang*¹; *Katsushi Tanaka*¹; *Masaharu Yamaguchi*¹; ¹Kyoto University, Matls. Sci. and Eng. Dept., Yoshidahonmachi Sakyo-ku, Kyoto 606-8501 Japan

Mo₅SiB₂ is a new refractory metal silicide with great potential for

ultra-high temperature structural applications. We have first succeeded to grow high purity single crystals of Mo_5SiB_2 by an optical floating zone method. Single-crystal elastic constants at room temperature (RT), thermal expansion coefficient (CTE) at RT-1500°C and electric resistivity at RT-800°C of the silicide were measured. Mo_5SiB_2 has less anisotropic single crystal elastic moduli and a lower shear modulus than MoSi_2 . In comparison to Mo_5Si_3 , it has isotropic CTE ($\alpha_c/\alpha_a=1.4$) and a high shear modulus. RT Vickers indentation tests and compression tests at high temperatures were also performed. Deformation mode wise, slip was observed on $\{001\}\{100\}$ for $[021]$ orientation at 1500°C. However, slip on $\langle 100 \rangle\langle 001 \rangle$, which has been reported to be activated, was not observed. Mechanical properties of Mo_5SiB_2 single crystals are discussed in terms of crystal structure and orientation dependence of shear modulus.

4:00 PM

Deformation Behavior of Mo_5Si_3 Single Crystals at High Temperatures: *K. Yoshimi*¹; *M. H. Yoo*¹; *A. A. Wereszczak*²; *S. M. Borowicz*¹; *E. P. George*¹; *E. Miura*³; *S. Hanada*³; ¹Oak Ridge National Laboratory, Mets. and Cers. Div., Oak Ridge, TN 37831-6115 USA; ²Oak Ridge National Laboratory, High Temp. Matls. Lab., Oak Ridge, TN 37831-6069 USA; ³Tohoku University, Instit. for Matls. Rsch., Sendai, Miyagi 980-8577 Japan

Compressive deformation behavior of D8m-type Mo_5Si_3 single crystals was investigated at temperatures between 1473K and 1723K in an argon atmosphere. Four compression axes, namely $[001]$, near $[111]$, near $[101]$ and $[100]$, were chosen. Plasticity occurred at and above 1573K, whereas at 1473K the crystals failed by brittle fracture before yielding. Slip traces with high-index planes were observed on the crystal surfaces. After high-temperature yielding, all crystals except the $[001]$ crystal exhibited a large yield drop, followed by plastic flow at a constant stress. Considering the constant-stress flow behavior as a steady-state creep process, it was estimated that the stress exponent is about 6 and the activation energy of deformation is approximately 450 kJ/mole. Dislocations were observed by post mortem TEM. The active slip system will be characterized, and a possible deformation mechanism will be discussed on the basis of the available bulk and defect properties.

4:20 PM

Influence of Texture and Hydrostatic Pressure on the Room Temperature Compression of NiAl Polycrystals: *Werner Skrotzki*¹; *Roland Tamm*¹; *Carl-Georg Oertel*¹; *Bernd Beckers*²; *Heinz-Guenter Brokmeier*³; *Georg Dresen*⁴; ¹Dresden Technical University, Instit. of Crystal. and Solid State Phys., Dresden 01062 Germany; ²RWTH Aachen, Institute of Metallurgy and Metal Physics, Kopernikusstr. 14, Aachen 52074 Germany; ³GKSS-Research Center Geesthacht GmbH, Max-Planck-Straße, Geesthacht 21494 Germany; ⁴Geo-Research Center Potsdam, Telegrafenberg, Potsdam 14473 Germany

Compression of NiAl polycrystals was carried out at room temperature under atmospheric as well as 0.4 Gpa confining pressure at constant strain rate. The stress-strain curves show that the strength as well as the work-hardening rate is generally higher when deformed under hydrostatic pressure. Moreover, there is a strong plastic anisotropy, that is, samples having a $\langle 100 \rangle$ crystallographic preferred orientation parallel to the compression axis are much harder than those having $\langle 110 \rangle$ and $\langle 111 \rangle$. Similarly, the frequency of microcracking shows the same tendency. It seems that the microcracks in the samples deformed under confining pressure mainly result from internal stresses released after unloading. The strength of the differently textured samples at elevated strains correlates with the Taylor energy calculated. These calculations also explain for certain textures the deviation from a homogeneous shape change during compression via plane-strain deformation. The textural changes produced by deformation are simulated with the Taylor model.

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Evaluation of Thermal Residual Stresses by Finite Element Modeling in NiAl-TiB₂ Joints: *R. D. Torres*¹; *G. G.W. Mustoe*²; *I. E. Reimanis*²; *J. J. Moore*²; ¹Catholic University of Parana, Mech. Eng. Dept., Rua Jovino do Rosario 475/506, Curitiba 82510 300 Brazil; ²Colorado School of Mines, Metall. and Matls. Eng. Dept., Golden, CO 80401 USA

The magnitude of the thermal residual stresses were determined for NiAl/TiB₂ joints. The thermal residual stresses were first calculated for a NiAl/TiB₂ sharp interface considering two situations. Firstly NiAl and TiB₂ were assumed to behave elastically and secondly assuming a small plastic deformation in the NiAl layer. It was found that all three stress components (radial, axial and shear) are reduced when NiAl undergoes plastic deformation. In a second study, a comparison in the magnitude of thermal residual stresses was made in a NiAl/TiB₂ graded joint, assessing the elastic properties and coefficient of thermal expansion by applying the linear rule of mixtures and measured values for the same properties. It was found that there is a small but non negligible difference in the radial stress when the linear rule of mixture (LROM) is used instead of the experimental measured values for the properties of the composites in the graded region. Finally thermal residual stresses were calculated for a five layer graded composite in which the architecture of the graded region was varied according to a power law model. In this models three situations can arise: i) a NiAl rich graded region, ii) same volume fraction of TiB₂ and NiAl and iii) TiB₂ rich graded region. The major finding of this specific study is that the magnitude and the location of the highest stress can change significantly with different graded region architectures.

Defects I

Monday PM

July 17, 2000

Room: Cypress 1

Location: The Westin Bayshore Hotel

Session Chair: V. Vitek; Haruyuki Inui

1:30 PM Invited

Lattice Defects and Their Influence on Mechanical Properties of TiAl Single Crystals: *Haruyuki Inui*¹; *Kazuyoshi Chikugo*¹; *Tokujiro Yamamoto*¹; *Masaharu Yamaguchi*¹; ¹Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Lattice defects in TiAl single crystals with nominal compositions of Ti-54 and -56 at.% Al have been investigated by means of transmission electron microscopy. When quenched from 1100°C following homogenization treatment at 1300°C, diffuse scattering due to short-range ordering of the Al_5Ti_3 -type appears in addition to discrete diffraction spots from the L1_0 lattice for Ti-56 at.% Al while for Ti-54 at.% Al, only discrete diffraction spots from the L1_0 lattice are observed. When aged at 400°C, discrete diffraction spots due to Al_5Ti_3 precipitates start to appear for both compositions and increase their intensity up to aging for one week for Ti-54 at.% Al and for one month for Ti-56 at.% Al. Upon further aging, the intensity of diffraction spots from Al_5Ti_3 precipitates decreases, resulting in the complete disappearance after three month aging for Ti-54 at.% Al. For Ti-56 at.% Al, however, diffuse intensity from Al_5Ti_3 precipitates remains even after aging for two years.

2:00 PM

Roles of Point Defects in Twinning of Intermetallics with FCC-Based Structures: *Man H. Yoo*¹; ¹Oak Ridge National Laboratory, Mets. and Cer. Div., P. O. Box 2008, Oak Ridge, TN 37831-6115 USA

In fcc-based superlattices, stacking fault and twin boundary energies are known to be rather low as compared to APB energies, and the kinetics of twin formation depends strongly on intrinsic and extrinsic point defects. This paper discusses possible effects of anti-site defects, thermal vacancies, self interstitials, and interstitial solutes on twin nucleation in intermetallic compounds of the L10 and C15 structures. The available experimental data relevant to the subject are summarized: namely, the profuse twinning activity in fully-lamellar Ti-Al alloys; the difficulty of twin formation in Ti-54at.% Al single crystals; the so-called radiation-induced ductility in Ti-47at.% Al alloys after neutron irradiation; and the formation of twin clusters in the Laves phase NbCr₂ and (Hf,Nb)V₂ alloys. The prominent features of experimental data will be discussed in terms of the proposed roles of point defects and their clusters in twin formation, and future research will be suggested.

2:20 PM

Bond-Order Potentials for Studies of Extended Defects in TiAl: V. Vitek¹; S. Znam¹; D. Nguyen-Manh²; D. G. Pettifor²; ¹University of Pennsylvania, Dept. of Matls. Sci. and Eng., Philadelphia, PA 19104-6272 USA; ²University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

The structure and properties of dislocations and interfaces in TiAl have been studied extensively using central-force many body potentials to describe atomic interactions in this compound. However, a variety of LDA based ab initio calculations indicate that the bonding in this alloy is not purely metallic and that the covalent component of bonding is significant. The semi-empirical method in which the required covalent character of bonding is included explicitly is the tight-binding method. In recent years this method has been reformulated in terms of Bond-Order Potentials (BOP) which is an order N, fully real space method. The cohesive energy is composed of the bond energy that contains the angular dependence of the atomic interactions, an environment dependent central-force many-body term representing the overlap repulsion arising from the valence sp electrons, and a pair potential term representing short-range repulsions. A condition of charge neutrality at individual atoms is employed as an ersatz for self-consistency. In this paper we present recently constructed BOPs for Ti-Al alloys that can be used in atomistic simulations in both TiAl and Ti₃Al. In this scheme only the valence d-electrons are retained explicitly on Ti sites and the p-electrons on Al sites and the orthogonal basis and two-center bond integrals are used. The transferability of these integrals is crucial when the method is to be used in studies of lattice defects and it is shown by studying the environmental dependence of the bond integrals using the ab initio all electron TB-LMTO method that in Ti-Al alloys the bond integrals are, indeed, transferable. Results of atomistic studies of stacking-fault type defects, ordinary dislocations and twin-like interfaces encountered in lamellar TiAl will then be presented. These will be compared with previous calculations employing central forces and the importance of non-central character of bonding for the structure and properties of these extended defects will be assessed.

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Interaction of Boron with Crystal Defects in B2-Ordered FeAl Alloys: Anna Fraczkiewicz¹; Annie-Sophie Gay¹; Michel Biscondi¹; Emmanuel Cadel²; Didier Blavette²; ¹Ecole des Mines de St-Etienne, Centre SMS, URA CNRS 1884, 158 Cours Fauriel, St-Etienne F42100 France; ²Universite de Rouen, Faculte des Sciences de Rouen, UMR 6634 CNRS, Mont St-Aignan F-76821 France

Doping with small boron additions in a well-known way to suppress the intrinsic room-temperature intergranular brittleness of intermetallic alloys. The commonly admitted mechanism, i.e. an intergranular segregation of boron, seems not to be the only important effect. An extended study of boron interactions with crystal defects (point defects, dislocations, APB, grain boundaries) was performed in FeAl (B2) alloys (40 or 50 at % Al). The intergranular segregation of boron is confirmed and

characterized; both the equilibrium and non-equilibrium (due to a solute atom/thermal vacancy interaction) segregation mechanisms are identified. Thanks to a study by 3D atom probe field ion microscopy, a strong tendency of boron to segregate to other kinds of crystal defects, like dislocations, stacking faults or APB, could be shown. Especially, the first experimental evidence of boron segregation to dislocations (with a simultaneous local depletion in Al) was obtained, thanks to an atomic-resolution 3D observation of an edge <100> segregated dislocation. These observations suggest that the elementary mechanisms of plastic deformation of FeAl crystals may be strongly modified by boron addition, and support the results presented in this conference.

3:00 PM

The Effect of Bond Energies on the Point Defect Behaviour in B2 Ordered Intermetallics: Mogadalai Pandurangan Gururajan¹; Thennathur A. Abinandanan¹; ¹Centre for Advanced Study, Dept. of Metall., Compu. Matls. Lab., Indian Instit. of Sci., Bangalore, Karnataka 560 012 India

The existing mean field theories of the point defect concentrations in ordered intermetallics employ bond energies, which are typically obtained from data on heat of sublimation, heat of formation, defect concentrations, etc. We have used a mean field formalism to study the point defect concentrations in B2 alloys with only the first nearest neighbor bond energies between the species A, B and vacancies (V). In particular, we examine the effect of AA, BB, AB, AV and BV bond energies in order to identify the range of these energies which leads to the "Bradley-Taylor" like behaviour viz., the vacancy concentration in B-rich alloys being proportional to off-stoichiometry. First, when the AV and BV bond energies are assumed to be zero and AA bond energy is negative, we show that the Bradley-Taylor like behaviour is observed only if the BB bond energy is repulsive i.e., positive or zero. On the other hand, if the AV and BV bond energies are assumed to be non-zero (specifically, they are assumed to be 25 % of AA and BB bond energies respectively), the Bradley-Taylor like behaviour is exhibited even by those systems with attractive, i.e., negative, BB bond energy. Our results are then used to rationalize the conflicting findings from the previous mean field studies.

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Electronic Structure of Carbon Doped FeAl Alloys: B. V. Reddy¹; S. C. Deevi¹; P. Jena²; ¹Philip Morris, Rsch. Dev. and Eng. Ctr., Richmond, VA 23234 USA; ²Virginia Commonwealth University, Phys. Dept., Richmond, VA 23284 USA

The effect of carbon on the geometric, electronic and magnetic structure of the FeAl host alloy is theoretically investigate using the first-principles density functional formalism within the generalized gradient approximation. The formation and the role of K-carbide phase are examined. For the perovskite K-carbide, a 15-atom unit cell is used for the calculations. The bulk environment for this unit cell is simulated by providing all the 14-nearest neighbors for the Fe-atoms in the central unit cell. The investigations on the stoichiometric and the non-stoichiometric K-carbide reveal interesting patterns of atomic relaxations. While the introduction of carbon constrains the Fe-atoms to reside on the faces of the cube, its removal drives the Fe-clustering due to the inward relaxations. Aluminum atoms occupying the corners of the perovskite structure are not perturbed significantly due to the addition or removal of carbon. It is demonstrated that the stoichiometric K-phase is non-magnetic, while the removal of one carbon neighbor induces a large moment of $\sim 1.4\mu_B$ on each of the Fe-atoms. Based on the B2(CsCl)-lattice and the characteristic triple defect structure of FeAl, a mechanism for the formation of Fe₃AlC is proposed. Further, the calculations reveal that the carbon going in to the interstitial sites prefers the octahedral sites to the tetrahedral sites. The optimal Al-C bond requirements for

these site occupations result in considerable distortions of the host lattice.

4:00 PM

Point Defect Behavior in B2-Type Intermetallic Compounds:

*Tomohide Haraguchi*¹; Mineo Kogachi²; ¹Osaka Prefecture University, Dept. of Mats. Sci., 1-1Gakuen-cho, Grad. Sch. of Sci., Sakai, Osaka 599-8531 Japan; ²Osaka Prefecture University, Dept. of Matls. Sci., College of Integr. Arts and Scis., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

As well known, material's properties are affected by their own lattice defects, particularly the point defects because they stably exist at any temperature. Point defect behavior in B2-type intermetallic compounds has been classified into two types, anti-site defect and triple defect, depending on whether they have 'constitutional' vacancies or not. Because of the important role of vacancies, we have attempted to precisely determine the vacancy concentrations for NiAl, CoAl, FeAl and AuCd alloys by means of density measurement. Further, we have tried to determine their point defect structure by means of x-ray or neutron diffractometry. Through these work, several results that cannot be explained by above-mentioned classification are found. In this report, point defect formation mechanism in B2-type alloys will be generally discussed on the basis of our both experimental and thermodynamic studies, together with the updated results for AgMg and CoFe alloys.

4:20 PM

Why Vacancies are Readily Quenched in NiAl and Related Intermetallics:

*Gary S. Collins*¹; *Matthew O. Zacate*¹; ¹Washington State University, Phys. Dept., Webster Bldg., Pullman, WA 99164-2814 USA

Furnace cooling of Ni-rich NiAl leaves behind quenched-in vacancies in concentrations of 0.2 at.%. These vacancies first become mobile over atomic length scales in 15-minute anneals at 350°C. The onset of dislocation climb by mobile, quenched-in vacancies offers a plausible explanation for the brittle-to-ductile transition. But why are vacancies so readily quenched-in? We report experiments on Ni-poor NiAl using PAC, a nuclear probe method, that demonstrate that Ni-vacancies cluster at temperatures below about 800K. A thermodynamic model fits measurements well with an association energy between vacancy and vacancy cluster equal to 0.20 eV, much greater than previously supposed. Clustering keeps vacancies from annealing out at low temperature.

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Crystal-Geometric Method of Planar Defects Analyses in Intermetallic Phases:

*Michail D. Starostenkov*¹; ¹Altai State Technical University, Gen. Phys. Dept., Lenin st. 46, Barnaul 656099 Russia

On the basis of crystal-geometric approach, developed by the author, structural filling of coordination spheres by components in superstructures of intermetallic phases of substitution and interstitial, electronic phases, Laves phases. This method allows to obtain analytical expressions for energy formation of any type of planar defects: antiphase boundaries, fault defects and their different complexes. Expressions as functions of ordering energy and energy of atom pair bonds in arbitrary number of coordination spheres. In the result of similar estimations it appears to be possible to determine strength characteristics of intermetallics, phases transformations from one type of superstructure into another, including long term superstructures.

Hydrogen Storage II

Monday PM
July 17, 2000

Room: Cypress 2
Location: The Westin Bayshore Hotel

Session Chair: M. Okada; E. Akiba

1:30 PM Invited

Applications of Flake Metal Powders as a Surface Modifier for Hydrogen Absorbing Intermetallic Electrodes of Ni/MH Rechargeable Battery: *Jai-Young Lee*¹; ¹Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., 373-1 Kusong-dong, Yusung-gu, Taejon 305-701 South Korea

Extensive studies on metal hydride have been carried out for their application as the anode in the Ni/MH battery. It is well known that the electrode performances such as rate-capability, low-temperature dischargeability, and cycle life, etc. depend on the surface properties as well as the bulk properties of MH alloy. Flake metal powders are very effective in improving the surface properties of metal hydride electrode. For example, flake Cu powder is very useful as a conductive material for MH electrode. In the case of flake Cu-compacted electrode, the rate capability and low temperature dischargeability were greatly improved. The improved electrode properties were due to Cu layers on the electrode surface deposited by the dissolution and precipitation of flake Cu powder. Also, inner cell pressure of the cell using Zr-based alloy was decreased very much by using flake Cu powder-mixed electrode, equaling to that of the cell using AB₅ type electrode. Flake Ni powder is a very useful material to modify the surface of metal hydride. The cycle life of Ti-based alloy is greatly improved by surface-alloying with Ni. After ball-milling with flake Ni powder, the alloy shows a good cycle life (8% capacity loss after 180 cycle) without decreasing the discharge capacity. On the other hand, V-Ti alloy can not charge/discharge itself in KOH solution. When the V-Ti alloy is surface-alloyed by ball-milling with flake Ni powder, the alloy shows a very high discharge capacity of 439mAh/g. These results are closely related with the high strain of flake Ni powder. The residual strain of flake Ni powder can promote the diffusion reaction in the impact event. Therefore, Flake Ni powder can modify the alloy surface without changing the bulk properties of alloy.

2:00 PM Invited

Hydrogen Absorbing Alloys with a Large Capacity for the New Energy Carrier: *Toshihiro Mouri*¹; ¹Toyota Motor Corporation, Matl. Eng. Div.1, 1 Toyota-cho, Toyota, Aichi 471-0873 Japan

In order to apply hydrogen absorbing alloy to the new energy carrier such as the hydrogen storage tank or the Ni-MH battery, a drastic increase in a hydrogen capacity of the alloy required. For years, intermetallics compounds or single phase alloys have been studied as hydrogen absorbing alloys. We have proposed the new approaches of the alloy design related to multiphase alloys. As results of these approaches, we found a new hydrogen absorbing alloy consisting of the micro structure that has a large hydrogen capacity and a good desorbing property. The hydrogen storage capacity of the newly-developed alloy was over twice that of the conventional alloy. The reaction rate or the activate condition were improved by the effect of intermetallics phase. We applied this alloy to a hydrogen storage tank of a prototype fuel-cell vehicle.

2:30 PM

Hydrogen Storage Properties of TiMn₂-Based Alloys for Metal Hydride Heat Pump: *Jeoung-Kurn Park*¹; Sang-Chol Han¹; Youn-

Seon Kang¹; Jai-Young Lee¹; ¹Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., Kusong-dong 373-1, Yusong-gu, Taejon 305-701 South Korea

TiMn₂-based alloys (AB₂-type) are promising hydrogen storage materials for their easy activation, good hydriding-dehydriding kinetics, high hydrogen storage capacity (~1.9 wt%H, LaNi₅-based alloys = ~1.5 wt%H) and low cost. But they have two critical problems for various applications, especially metal hydride heat pump. One is a high plateau pressure of alloys at room temperature about over 20 atm. The other is a poor sloping characteristics. It is important to solve both of problems simultaneously. It was known that the reasons for the sloping of the alloys were i) micro-segregation, ii) chemical energy effect, and iii) strain energy effect. To improve the sloping properties, heat treatment, and changing the stoichiometry or alloy substitution methods are adopted respectively. Non-stoichiometry and V, Cu substitution method are found to be very effective for improving sloping properties and decreasing a plateau pressure. Heat treatment for homogenization of alloys is conducted at 1100°C for 20h under Ar atmosphere. From the P-C-T curves and SEM analysis, it is found that these alloys are homogenized after heat treatment, but the sloping properties are not improved. Hydrogen forming element part is increased. The A/B ratio (x in A_{1-x}B) is changed from 0.0 to 0.15. Vanadium and copper are substituted for manganese of the Ti_{0.8}Zr_{0.2}MnCr alloy. The amount of V and Cu (x in Ti_{0.8}Zr_{0.2}(MnCr)_{1-y}MY) is changed from 0.05 to 0.2. In the case of x=0.05 and y=0.15 ((Ti_{0.8}Zr_{0.2})_{1.05}Mn_{0.8}Cr_{1.05}V_{0.05}Cu_{0.1}), the alloy shows excellent sloping properties with the plateau pressure maintained under 10 atm.

2:50 PM

Multiphase Effects on the Hydrogen Absorption/Desorption Behavior of Ti-22Al-27Nb Alloys: Kazuhiro Ito¹; Lanting T. Zhang¹; Vijay K. Vasudevan²; Masaharu Yamaguchi¹; ¹Kyoto University, Matls. Sci. and Eng. Dept., Yoshidahonmachi Sakyo-ku, Kyoto 606-8501 Japan; ²University of Cincinnati, Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

Microstructure of hydrogen absorbing alloys has been known to affect their hydrogen absorption/desorption behavior. However, there has been no systematic understanding of mechanisms of the microstructure effect. We investigated multiphase effects on the hydrogen absorption/desorption behavior at 100°C of an alloy (Ti-22Al-27Nb (at. %)) in the Ti-Al-Nb system whose multiphase structures consisting such as bcc, B2 and O phases can be easily obtained. B2 single-phase alloys form β(bcc) hydride. In the β(bcc) phase, the hydrogen pressure rises steeply with hydrogen concentration and the β-γ (bct) hydride phase transformation does not occur. However, when O phase exists in the B2 or bcc matrix phase, the β-γ hydride transformation occurs and reversible absorption/desorption of hydrogen is observed between β and γ hydride states. Roles of O phase in the reversible hydrogen absorption/desorption behavior will be discussed and future work to improve the hydrogen absorption/desorption properties of Ti-Al-Nb alloys will be suggested.

3:10 PM Break

3:30 PM

Effects of Cold-Rolling on the Hydrogen Absorption/Desorption Behaviour of Ti-22Al-27Nb Alloys: Lanting Zhang¹; Kazuhiro Ito¹; V. K. Vasudevan²; M. Yamaguchi¹; ¹Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan; ²University of Cincinnati, Dept. of Matls. Sci. and Eng., P.O. Box 210012, Cincinnati, OH 45221-0012 USA

B2 single-phase alloys with a composition of Ti-22Al-27Nb (at. %) were cold-rolled to 80% reduction in thickness at room temperature and effects of cold-rolling on their hydrogen absorption/desorption behavior were investigated. P-C-T measurements were made at 50 and 100°C.

Similarly to bcc hydrogen absorbing alloys, Ti-22Al-27Nb alloys with the B2 structure form β (bcc) hydride at a very low hydrogen pressure. However, the β hydride in undeformed specimens does not transform to γ (bct) hydride. In contrast, the β hydride in cold-rolled specimens transforms to the γ hydride with increasing hydrogen concentration, although the plateau of the transition is not flat, and the γ hydride transforms back to the β hydride with decreasing hydrogen pressure. Thus, cold-rolling gives rise to a reversible β-γ hydride transformation in B2 Ti-22Al-27Nb alloys. Mechanisms of the reversible hydride transformation will be discussed in terms of a considerable amount of dislocations introduced in cold-rolled specimens.

3:50 PM +

Effects of Lattice Defects on Hydrogen Absorption-Desorption Pressures in LaNi₅-Based Alloys: Tokujiro Yamamoto¹; Haruyuki Inui¹; Masaharu Yamaguchi¹; ¹Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Microstructural evolution in LaNi₅-based alloys during hydriding-dehydriding cycles has been investigated by means of transmission electron microscopy. A high density of dislocations are introduced in LaNi₅ particles during the first cycle of hydriding. Once such a high density of dislocations are introduced, the hydrogen absorption pressure decreases considerably from 0.9 MPa for the first cycle to 0.3 MPa for the second and further cycles. Most of these introduced dislocations have Burgers vectors of the 1/3<11-20>-type and of the edge character. These are consistent with the observation by neutron and X-ray diffraction that anisotropic line-broadening preferentially occurs for <hk0>-type reflections. When the sample once hydrogenated was annealed at 800°C so that the introduced dislocations were annealed out, the hydrogen absorption pressure increases again from 0.3 MPa before annealing to 0.6 MPa after annealing. Thus, the density of introduced dislocations seems to control the hydrogen absorption pressure of LaNi₅-based alloys.

4:10 PM

Electrochemical Behaviour of Intermetallic-Based Metal Hydrides Used in Ni/Metal Hydride (MH) Batteries: Derek O. Northwood¹; ¹Ryerson Polytechnic University, Fac. of Eng. and Appl. Sci., 350 Victoria St., Toronto, Ontario M5B 2K3 Canada

Hydrogen storage alloys are a new group of new functional intermetallics which have found multi-purpose applications. The development of Ni/MH (metal hydride) batteries based on MH negative electrodes has seen considerable activity in recent years. Batteries based on such hydride materials have some major advantages over the more conventional lead-acid and nickel-cadmium systems. These advantages include: high energy density; high rate capability; tolerance to overcharge and over-discharge; the lack of any poisonous heavy metals; and no electrolyte consumption during charge/discharge cycling. The most important electrochemical characteristics of the hydrogen storage compounds used in these batteries include capacity, cycle life time, exchange current density and equilibrium potential. These characteristics can be changed by designing the composition of the hydrogen storage alloy to provide optimum performance of the Ni/MH batteries. The electrochemical behaviour of such intermetallics depends on their structure, the nature and the amount of each element in the intermetallic compound, and the electrochemical process(es) taking place. In this paper, we present some recent results on the electrochemical behaviour of such compounds and the mechanisms of the electrochemical reactions.

4:30 PM

MOCVD Regrowth of GaN on Free-Standing GaN Substrate Prepared by Hydride Vapor Phase Epitaxy: Ig-Hyeon Kim¹; ¹Samsung Advanced Institute of Technology, Comp. Semicond. Lab., P.O. Box 111, Suwon, Korea

GaN overlayers were grown on free-standing HVPE (Hydride Vapor Phase Epitaxy) GaN substrate by metalorganic chemical vapor deposition. Surface morphology of GaN overlayer was investigated with various surface treatment processes including mechanical polishing, chemical assisted ion beam etching (CAIBE), and thermal annealing. It was found that ion beam etching process on polished free-standing GaN substrate is essential to grow high quality GaN with the defect density of about 10^6 cm^{-2} . Epi-grade surface morphology and low defect density were obtained on optimized surface condition of the substrate. The ion beam etching about 2000 Å in depth can completely remove the surface damaged caused by mechanical polishing and results in epi-grade surface of GaN overlayer. There exist an optimum etching depth for ion beam etching to obtain high quality GaN overlayer with smooth surface. Excessive ion beam etching deteriorate surface quality of free-standing substrate. The performance of LEDs grown on both substrates of Sapphire and free-standing GaN was investigated and discussed in terms of surface morphology and defect density.

4:50 PM

Gas Atomization Processing of Designed Dual-Phase Intermetallic Hydrogen Storage Alloys: *Jason Ting*¹; *Ulrike Habel*¹; *Michael W. Peretti*¹; *William B. Eisen*¹; *Brian J. McTiernan*¹; *Rosa C. Young*²; *Bao Huang*²; *Benjamin S. Chao*²; ¹Crucible Research, 6003 Campbells Run Rd., Pittsburgh, PA 15205 USA; ²Energy Conversion Devices, Inc., 1675 West Maple Rd., Troy, MI 48084 USA

The two main classes of hydrogen storage intermetallics for battery applications can be grouped according to their stoichiometry: AB_2 and AB_5 . In this study, dual phase alloys were produced by gas atomization process, containing both AB_2 and AB_5 phases. These dual-phase alloys were made with the intention of adding small amounts of AB_5 alloys to AB_2 alloys. The alloys were designed from synthesizing a matrix phase of zirconium-based AB_2 and a secondary phase of mischmetal-based AB_5 alloy. Utilizing the inherent high solidification rate of the gas atomization process, submicron dispersion of AB_5 precipitates among the AB_2 matrix were produced. The dispersion of AB_5 precipitates provided strong catalytic sites that facilitated the rapid absorption and desorption of hydrogen for high discharge capacity at high discharge rates. SEM of microstructures, wet cell discharge capacities, gas atomized powder size distributions and post-atomization of the powders will be discussed.

Processing and Properties III

Tuesday AM Room: Salon 1
July 18, 2000 Location: The Westin Bayshore Hotel

Session Chairs: James Li; Ian Baker

8:30 AM

Intermetallic Nanoparticles: Synthesis and Characterization: *Samy S. El-Shall*¹; *Y. Pithawalla*¹; *Seetharama C. Deevi*²; ¹Virginia Commonwealth University, Dept. of Chem., 1001 W. Main St., Richmond, VA 23284-2006 USA; ²Philip Morris USA, Rsch., Dev. and Eng. Ctr., 4201 Commerce Rd. D-234, Richmond, VA 23234 USA

Nanoparticles often exhibit novel properties widely different from the bulk properties of materials. Research in nanomaterials is motivated by the possibility of designing nanostructured materials that possess novel electronic, optical, magnetic, photochemical and catalytic properties. These properties show strong dependence on size, shape and surface preparation. We developed a novel technique to synthesize nanoparticles of controlled size and composition. Our technique com-

bines the advantages of pulsed laser vaporization with controlled condensation (LVCC) in a diffusion cloud chamber under well-defined conditions of temperature and pressure. It allows synthesis of a wide variety of nanoparticles of metals and alloys, and oxides, carbides and nitrides of a wide variety of metals. We will discuss the synthesis and characterization of a variety of intermetallic nanoparticles such as TiAl, and FeAl (with different Al contents). In addition, we address methods to incorporate nanocrystals into polymer films will be discussed.

8:50 AM

Spray Forming and Subsequent Forging of Gamma Titanium Aluminide Alloys: *Gerhard Wegmann*¹; *Rainer Gerling*¹; *Frank-Peter Schimansky*¹; *Jin-Xu Zhang*²; ¹GKSS Research Centre, Instit. for Matls. Rsch., Max-Planck-Str, Geesthacht 21502 Germany; ²Shanghai Jiao Tong University, Shanghai 200030 PRC

Utilizing the 'Electrode Induction Melting Gas Atomization' technology (EIGA) and a specially designed collector system spray forming experiments with a binary Ti-48.9Al (at%) alloy and an advanced γ -TiAl alloy with the composition Ti-47Al-4(Nb, Mn, Cr, Si, B), designated as γ -TAB, were carried out. Subsequently, the spray formed materials were successfully forged. The sprayed and forged conditions were characterized in terms of porosity and microstructure. Tensile properties were evaluated for room temperature and for elevated temperatures. Upon forging the porosity of the spray formed materials was reduced significantly. The microstructures turned from nearly lamellar to near γ with a grain size of $4.9 \mu\text{m}$ (Ti-48.9Al) and from duplex to near γ with a grain size of $2.2 \mu\text{m}$ (γ -TAB). The sprayed and forged γ -TAB alloy sustains an elongation of 120% at 800°C indicating the possibility of superplastic forming. The results are discussed in comparison with conventionally P/M-processed and HIP-compacted materials of the same composition.

9:10 AM

Ductile Ni₃Al Thin Foil by Cold Rolling: *Toshiyuki Hirano*¹; *Masahiko Demura*¹; *Kyousuke Kishida*¹; *Osamu Umezawa*¹; *Yozo Suga*²; ¹National Research Institute for Metals, Mech. Prop. Div., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; ²Nippon Cross Rolling Corporation, 697 Mobara, Chiba 297-0026 Japan

Honeycomb structure of intermetallic compound Ni_3Al is a promising high-temperature structural component because of its lightweight. Large-area thin foil below 200mm in thickness is required for the honeycomb structure, but this has been a problem. With boron-doped Ni_3Al it is very difficult to fabricate such thin foil by engineering technique such as cold rolling. Here we present the fabrication of thin foil by cold rolling using directionally solidified binary stoichiometric Ni_3Al . Thin foils of 70mm in thickness were successfully fabricated with 92% reduction at room temperature without intermediate annealing. The surface is smooth and shining. It is possible to bend the as-rolled foil almost double. There are no major problems in laser-beam welding. In addition, the foil exhibits more than 5% room-temperature tensile elongation after heat treatment at high temperatures above 1573K. The texture and the mechanical properties will be presented.

9:30 AM

Superposition of Grain Size and Dispersion Strengthening in ODS L1_2 -(Al,Cr)₃Ti: *Martin C. Heilmair*¹; *Holger Saage*¹; *Kabir J. Mirpuri*¹; *Jürgen H. Eckert*¹; *Ludwig Schultz*¹; *Paramanand Singh*²; ¹IFW Dresden, Instit. for Met. Matls., P.O. Box 270016, Dresden D-01171 Germany; ²IIT Madras, Dept. of Metall. Eng., Madras 600 036 India

In previous investigations we have successfully demonstrated the preparation of oxide dispersion strengthened (ODS) $\text{Al}_{67}\text{Cr}_8\text{Ti}_{25}$ -type intermetallic compounds with L1_2 -structure by mechanical alloying and ball milling, respectively. Subsequent powder consolidation by hot uniaxial pressing yielded fully dense samples with nano-scaled disper-

sions of 1 to 3 vol.% Y_2O_3 and ZrO_2 embedded in the single-phase matrix revealing a sub-micrometer grain size. As a single-phase reference samples without oxide dispersions were also prepared. While retaining the mean dispersoid size, additional thermal and thermo-mechanical treatment yielded normal and abnormal grain growth of the matrix, respectively, to produce grain sizes ranging from several microns to close to 1 mm. Constant compression tests were carried out at ambient temperature to reveal the contribution to the yield strength stemming from the dispersoid/dislocation interaction as well as from dislocation pile-up at grain boundaries. The results will be discussed in terms of current microstructurally based concepts for room temperature yielding.

9:50 AM

Synthesis and Thermal Stability of Nano-RuAl by Mechanical Alloying: *Kaiwen Liu*¹; Frank Mücklich¹; ¹Saarland University, Institut für Funct. Matls., P.O. Box 151150, Geb.22 Etage 7, Saarbrücken, Saarlandes D-66041 Germany

Single phase RuAl has been synthesized by mechanical alloying(MA). The structural evolution during MA and thermal stability of as-milled powders at elevated temperatures have been analyzed by XRD, thermal analysis and isothermal annealing. The results indicate that there are two stages of reaction between Ru and Al in MA before single phase RuAl is obtained, and that the as-milled RuAl experiences reordering, strain relaxation and grain growth at high temperatures. All three structural evolutions show signs of stagnation upon high temperature annealing. The isothermal grain growth kinetics has been analyzed by considering the influence of impurities. Upon grain growth, lattice diffusion and segregation of impurities in grain boundaries have been verified by lattice parameter variation. Reordering and strain relaxation processes could consume part of driving force for grain growth. The grain size of less than 40 nm in RuAl annealed at 900°C for 5 h indicates its strong thermal stability.

10:10 AM

Effect of NiCoCrAlYHf Overlay Coating on Performance of Ni3Al Base Alloy IC6A: *Chengbo Xiao*¹; Yafang Han¹; Jianping Li¹; Shusuo Li¹; Xihong Zhao¹; ¹Beijing Institute of Aeronautical Materials, P.O. Box 81-1, Beijing 100095 PRC

The directionally solidified Ni3Al base alloy IC6 has been recently developed as a high-temperature structural material used for advanced jet-engine vanes operating in the temperature range of 1050-1100° and 161-230°. The alloy not only has high yield strength and fairly good ductility from room temperature to 1100° and 161-230°, but also has high creep resistance in the temperature range of 760-1100° and 161-230°. However, the high temperature oxidation resistance and hot corrosion resistance at intermediate temperatures of alloy IC6 are poor for the high content of molybdenum and should be further improved for industrial application. Its high temperature oxidation resistance is substantially improved by adding proper amount of yttrium. The alloy modified by yttrium is named IC6A. A variety of experimental techniques have been used to study the effect of NiCoCrAlYHf overlay coating on the performance of alloy IC6A in the paper.

10:30 AM

Refining of Coarse Lamellar Microstructure of TiAl Alloys by Rapid Heat Treatment: *Jian N. Wang*¹; P. Zhou¹; ¹Shanghai Jiao Tong University, Sch. of Matls. Sci. and Eng., Shanghai 200030 PRC

It is now well known that the room temperature tensile strength and ductility of TiAl alloys increase with decreasing the colony size (d) of a fully lamellar (FL) structure. Therefore searching a cost-effective approach to refine the coarse FL structure becomes critical to the engineering applications of this material. In this presentation, we report our recent observation that, merely by rapid heat treatment, a FL structure with $d \geq 500$ um in TiAl alloys of Ti-48Al-2Cr and Ti-46Al-2Cr-2Nb

(at.%) could be refined to a FL structure with d being as fine as 10 um. Thermomechanical processing such as canned forging and hot extrusion, as done in many previous studies, was not required. In order to achieve the refinement, several parameters should be strictly controlled including heating rate, heating temperature, hold time and cooling rate. Mechanical testing in tension on the Ti-48Al-2Cr alloy shows that with the refinement from 500 to 10 um, the room temperature yield strength increased from 330 to 610 MPa, fracture strength from 415 to 825 MPa and ductility from 0.7 to 3.3%, although the fracture toughness decreased only slightly from 24 to 19 MPa.m^{1/2}.

10:50 AM

Designed Fully Lamellar Microstructures in a Gamma-TiAl Based Alloy: Adjustment and Microstructural Changes Upon Isothermal Exposure: *Manuel Beschliesser*¹; Anita Chatterjee²; Alexander Lorich¹; Wolfram Knabl³; Heinrich Kestler³; Helmut Clemens⁴; ¹Materials Center Leoben (MCL), Franz-Josef-Str. 13, Leoben 8700 Austria;

²Max-Planck-Institut fuer Metallforschung, Seestr. 92, Stuttgart 70174 Germany; ³Plansee AG, Techn. Ctr., Reutte 6600 Austria; ⁴Universitaet Stuttgart, Institut fuer Metallkunde, Seestr. 71, Stuttgart 70174 Germany

A balanced combination of fracture toughness and creep strength is expected for fully lamellar microstructures exhibiting a small colony size and narrow lamellar spacing. In this study, the influence of annealing temperature and time on colony size as well as the effect of cooling rate on lamellar spacing was investigated. Fine-grained Ti-46.5at.% Al-4at.% (Cr,Nb,Ta) sheets with and without boron addition were used as starting materials. In case of the boron containing alloy the presence of (Ti,Ta)-borides impede the growth of the alpha-grains and consequently a small colony size is achieved upon cooling. The cooling rate must be selected carefully in order to avoid Widmannstätten-like features as well as gamma grains at colony boundaries. Within undisturbed fully lamellar microstructures the mean interface spacing decreases with increasing cooling rate. However, high cooling rates lead to alpha₂-fractions far from equilibrium, thus providing a driving force for microstructural changes upon long-term exposure at 700°C and 800°C.

11:10 AM

Microstructural Evolution during Heat Treatments in Ti-45 and 47Al-2Nb-2Mn+0.8vol.% TiB₂XDTM alloys: *Dong-Yi Seo*¹; Linruo Zhao²; Jonathan Beddoes¹; ¹Carleton University, Dept. of Aero. and Mech. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada;

²Structures, Materials and Propulsion Laboratory, Institute for Aerospace Research, National Research Council of Canada, Ottawa, Ontario K1A 0R6 Canada

Investment cast gamma XD TiAl alloys, Ti-45 and 47Al-2Nb-2Mn+0.8vol.% TiB₂, were heat treated below and above alpha transus temperature to investigate the microstructural response. The heat treatment temperature and time range from 1290~1400°C and 0.5~24 hours. Different cooling methods, including furnace cool, air cool, and oil quench, were used to control lamellar spacing. For microstructural stabilization, the heat-treated samples were subsequently subjected to a secondary aging treatment. Single or multiple steps of aging treatments were developed to minimize microstructural coarsening. The microstructural features resulting from the heat treatments were analyzed in terms of grain size, volume fraction of lamellar grains, and interlamellar spacing. To assess the strength of the heat-treated material, hardness testing was conducted and the results were analyzed against those derived from standard XD TiAl alloys. From this research, the heat treatment conditions responsible for various microstructural features can be identified and applied to produce required microstructures.

11:30 AM

Structural Development in Sputter Deposited TiAlCr Coating Alloys: Zhiqi Wang¹; Guosheng Shao¹; Fuhui Wang²; Panayiotis Tsakiroopoulos¹; ¹University of Surrey, Sch. of Mech. and Matls. Eng., Guildford, Surrey GU2 5XH UK; ²Institute of Metal Research, Corrosion and Protection, 62 Wencui Rd., Shenyang 110015 PRC

Intermetallic alloys based on TiAl are considered for structural applications at elevated temperatures. However, these alloys need to be protected from oxidation above 973K. Alloys based on the Ti-Al-Cr system and containing the Laves Ti(Cr,Al)₂ and Gamma TiAl phases exhibit excellent oxidation resistance up to 1273K under both static and cyclic thermal exposures. In this work, Ti-50Al-xCr (x = 0-20 at%) coatings have been produced by magnetron sputter deposition. Microstructures of as-deposited and annealed samples have been studied using analytical electron microscopy. The results show that the typical morphology of the crystalline phase/s in the coatings is columnar and that the nucleation of crystalline phases can be completely suppressed by controlling processing parameters. The as-deposited structures have great influence on the morphology of the final microstructures in the annealed samples.

11:50 AM

Mechanical Characterisation of γ -TiAl Thin Films Obtained by Two Different Sputtering VIA: Maria Teresa Freire Vieira¹; Bruno Trindade¹; Sofia Ramos¹; José Valdemar Fernandes¹; Manuel Vieira²; ¹Coimbra University, Mech. Dept., Polo II, Pinhal de Marrocos, Coimbra 3030 Portugal; ²Oporto University, Faculty of Eng., Rua dos Bragas, Oporto 4099 Portugal

Sputtering has been used as a predictive way to produce new bulk materials with enhanced properties since it easily enables to cover a wide range of composition. In this study (TiAl)-based films were magnetron sputtered using two different methods: two targets (Ti + Al) and a composite target (TiAl). In the first case, the as-deposited films were constituted by nanometric multilayers of Ti and Al. In the second case, f.c.c. disordered TiAl was obtained. In both cases, the as-deposited films had to be heat treated in order to obtain the intermetallic γ -TiAl. The mechanical characterisation of the films included hardness, Young's modulus and ductility evaluations. A tensile sample with a special geometry was developed for this purpose. The residual stresses were evaluated by laser curvature measurements using a Stoney equation and by X-ray measurements considering the material's texture. The effect of the addition of chromium on the structure and mechanical properties was studied.

12:10 PM

Influence of Packing Treatment on Mechanical Properties of Fe-18Al and Fe-18Al-5Cr Alloys: Liu-Ho Chiu¹; Pee-Yew Lee²; ¹Tatung University, Dept. of Matls. Eng., 40 Chungshan N. Rd., 3rd Sec., Taipei, Taiwan 10451; ²National Taiwan Ocean University, Inst. of Matls. Eng., Keelung, Taiwan

The influence of a pack treatment on the mechanical properties of the Fe-18Al and Fe-18Al-5Cr alloys was investigated. The crystal structure analyses of the surface layer and the mechanical properties of Al₂O₃ powder pack treated specimens for two FeAl alloys at a temperature range of 700°C to 1000°C are investigated. The ductility of the specimen of Fe-18Al alloy conducted by packing treatment at 700°C for 8 hours had the lowest elongation (0.8%) among those specimens studied. The specimen of Fe-18Al alloy conducted by packing treatment at 1000°C for 8 hours had the highest elongation (19.6%) among those specimens studied. The significant improvement in the tensile ductility of Fe-18Al and Fe-18Al-5Cr alloys by pack treatment are related to the forming of aluminum oxide layer and Al-depleted alloy layer on the specimen surface.

Alloying Effect I

Tuesday AM
July 18, 2000

Room: Salon 3
Location: The Westin Bayshore Hotel

Session Chair: M. Akinc; E. A. Loria

8:30 AM Invited

The Effect of Stoichiometry on the Properties of C15 Laves Phases: Dan J. Thoma¹; Katherine C. Chen²; Paul G. Kotula³; K. A. Nibur²; E. J. Peterson¹; ¹Los Alamos National Laboratory, Mail Stop G770, Los Alamos, NM 87545 USA; ²California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA; ³Sandia National Laboratory, Mail Stop 1405, Albuquerque, NM 87185 USA

The effect of stoichiometry in binary (HfCo₂ and NbCr₂) and ternary (NbCr₂-TiCr₂) C15 phases properties have been investigated. The HfCo₂ phase was chosen as a model binary system because binary solubility occurs on both sides of stoichiometry. The dominant constitutional defect mechanism appears to be anti-site substitution, and the intermetallic phase displays a maximum of elastic modulus and hardness values at stoichiometry. Moreover, from hardness testing, the Hf-Co Laves phases become less brittle on the Co-rich side of stoichiometry. While ternary NbCr₂-TiCr₂ Laves phases also are governed by anti-site constitutional defects, the alloys display a maximum in properties in the center of the ternary phase field (and a minimum of toughness). Although the binary behavior is contrary to many intermetallic systems, the property response is consistent with bonding characteristics in Laves phases. However, the ternary phase field has features that are more characteristic of solid-solution strengthening mechanisms. Support of DOE-OBES, Division of Materials Sciences is gratefully acknowledged.

9:00 AM Invited

Design of High Temperature Alloys Strengthened by Coherent Interfaces: H. Harada¹; ¹National Research Institute for Metals, High Temp. Matls. 21 Project, 1-2-1 Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan

Introducing coherent interphase interface is a very effective way for strengthening high temperature materials. In the present paper, firstly, Ni-base superalloys are discussed as a typical example. Depending on the fcc/L12 interfacial lattice misfit, the morphology of L12 precipitate as well as the dislocation network density on the semi-coherent interface changes to affect the creep strength. Our recent alloy design work with a careful attention to the lattice misfit toward the development of 4th generation single crystal superalloys is presented. New alloy systems with having coherent interfaces are being searched for and investigated in our Project. Some results with Ir- and Rh-base refractory superalloys both having the fcc/L12 coherent structure are introduced and compared with Ni-base superalloys.

9:30 AM

Effect of Alloying Addition and Hot Extrusion on Microstructure and Tensile Properties of TiAl Alloys: C. T. Liu¹; S. C. Deevi²; J. L. Wright¹; ¹Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831-6115 USA; ²Philip Morris USA, Rsch. Ctr., 4201 Commerce Rd., Richmond, VA 23234 USA

Alloying additions of Cr and V are commonly added to two-phase TiAl alloys for ductility improvement; however, these elements cause some concerns when the alloys are used for health related applications. The objective of this study is to achieve good ductility from TiAl alloys containing no health-concern elements through control of microstruc-

tures, alloying additions, and processing parameters. TiAl alloys containing 2.0 to 4.0 Nb, 0.5 to 1.0 W, 0 to 0.5 Mo, and 0.08 to 0.2 B (at.%) were prepared by arc melting and drop casting, following by hot extrusion at 1295 to 1400°C. A variety of microstructures, including nearly duplex to fully lamellar microstructures and uniform to non-uniform grain structures, were produced by control of hot extrusion temperature and post-extrusion heat treatment. High ductility (~4%) and high strength (~100 ksi) were obtained in these alloys at ambient temperatures, and the results will be correlated with microstructural features observed in these alloys. Research was sponsored by Philip Morris, Inc, under the Work for Other Program under contract No. DE-AC05-96OR22464 with Lockheed Martin Research Corporation.

9:50 AM +

Effects of Refractory Metals on Microstructure and Mechanical Properties of Directionally-Solidified TiAl Alloys: *Shinji Muto*¹; Takamitsu Yamanaka¹; David R. Johnson²; Haruyuki Inui¹; Masaharu Yamaguchi¹; ¹Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan; ²Purdue University, Sch. of Matls. Eng., West Lafayette, IN 47907-1289 USA

We have shown that in the Ti-Al-Si ternary system, the lamellar microstructure of two-phase TiAl alloys can be aligned parallel to the growth direction by directional solidification (DS) with the use of a seed crystal with appropriate compositions and the resultant DS ingots exhibit mechanical properties superior to conventional TiAl alloys. In the present study, we have investigated effects of refractory metals such as Mo, W and Re on microstructure and mechanical properties of directionally-solidified TiAl-Si alloys. Lamellar microstructure as well as room-temperature strength and ductility and high-temperature creep properties were evaluated. The maximum alloying amount where the alignment of the lamellar microstructure is possible by DS-processing is found to be about 2 at.% for all the refractory metals. The creep properties are found to be improved upon alloying with these metals, with the significant improvement occurring for the Re additions.

10:10 AM

Effect of Addition of Rare Earth Element Gd on Grain Growth in Ti-44Al Based Alloys: *Wenhong Li*¹; *Kenong Xia*¹; ¹University of Melbourne, Dept. of Mech. and Manufact. Eng., Victoria 3010 Australia

Grain growth in the single alpha region was studied both in a Ti-44Al-0.15Gd alloy and in a binary Ti-44Al alloy. Cast ingots were solution treated at 1623K for various times from 2 minutes to 20 hours and furnace cooled to obtain fully lamellar microstructures. Grain growth was much slower in the Gd containing alloy. The kinetics of grain growth can be described by the traditional power-law relationship between the grain size (in micrometers) and time (in seconds) with a proportionality constant of 320 and a time exponent of 0.043 for the Gd-containing alloy, and 315 and 0.17 for the binary alloy, respectively. Some ingots were solution treated at temperatures from 1543 to 1713K for 1 hour and the grain size was plotted against the reciprocal temperature. The activation energy obtained was 50 kJ/mol for the Gd-containing alloy and 81 kJ/mol for the binary alloy. The implications of these values were discussed.

10:30 AM

Thermal Stability of Ti-46Al-5Nb-1W Alloy: *Ze-Wen Huang*¹; Wayne Voice²; Paul Bowen¹; ¹The University of Birmingham, IRC in Matls. for High Perform. Appl./Sch. of Metall. and Matls., Edgbaston, Birmingham B15 2TT, UK; ²Rolls-Royce plc, Aerospace Group, P.O. Box 31, Derby DE24 8BJ UK

The TiAl-based alloy Ti-46Al-5Nb-1W has been exposed at a temperature of 700°C for up to 5000 hours in air. The decomposition processes of all major phases in this alloy: gamma, alpha-2, beta and omega, have been investigated in detail by transmission electron mi-

croscopy and have been correlated with tensile properties. It was found that considerable amounts of beta phase were retained in the as-cast condition and these readily decomposed into omega phase after exposure. Along with this transformation, beta was also consumed by gamma and gradually disappeared with increasing exposure time. During exposure, alpha-2 lamellae were altered very slightly at first but began to change significantly after 3000 and 5000-hour exposures, while gamma lamellae remained essentially unchanged throughout this process. The ductility, yield stress and tensile strength are discussed with reference to these underlying microstructural changes.

10:50 AM Break

11:10 AM Invited

The Effect of Cr Addition on Mechanical and Chemical Properties of the Ni₃Si Alloys: *Takayuki Takasugi*¹; Yasuyuki Kaneno¹; ¹Osaka Prefecture University, Dept. of Metall. and Matls. Sci., Gakuen-cho 1-1, Sakai, Osaka 599-8531 Japan

The microstructures of the Cr-containing Ni₃Si alloys were firstly characterized by an optical microscope, a scanning electron microscope (attached with WDS) and a transmission electron microscope. Next, the mechanical properties of the Cr-containing Ni₃Si alloys were evaluated as a function of temperature by tensile and compression tests, and also SEM fractography. Also, the chemical properties such as oxidation behavior in air and corrosion behavior in sulfuric acid were evaluated, and correlated with the microstructure and chemistry of the Cr-containing Ni₃Si alloys. It was found that by the Cr addition to the Ni₃Si alloy, the strength was enhanced, and also the chemical properties were substantially improved.

11:40 AM Invited

The Evolution of Microstructure Change and Mechanical Properties for a Nickel Silicide Based Alloy Doped with Carbon and Boron: *Jason S.C. Jang*¹; T. Z. Ou¹; C. Y. Cheng¹; ¹I-Shou University, Dept. of Matls. and Eng., Kaohsiung, Taiwan

A Ni-19Si-3Nb alloy was selected as the based alloy and microalloyed with a series amount of carbon and boron for improving their mechanical properties. The specimens were prepared by arc melting and drop casting under argon atmospheres, annealing in the vacuum, and then machined for the mechanical testing. The evolution of microstructure change and mechanical properties of the Ni-19Si-3Nb-xC alloys were characterized by the x-ray diffraction, DTA, SEM, EPMA, microhardness test, and tensile tests respectively. The results of x-ray diffraction and DTA could not resolve any change of phase content for the Ni-19Si-3Nb alloy with varied carbon and boron content. But, the consequences of the fracture behavior of the alloys of Ni-19Si-3Nb with varied carbon and boron addition exhibit significant change from brittle mode to ductile mode. The optimum additions of carbon and boron for improving the mechanical properties of the based alloy will occur at the combination of 100 ppm carbon and 300 ppm boron addition. The relation among the effect of carbon and boron addition, the microstructure change, and the mechanical behavior on these Ni-19Si-3Nb-xC alloys will be discussed in this paper.

12:10 AM

Effects of Stoichiometry and Fe Addition on Crystal Structure of Ni₃Nb: A. Yamauchi¹; T. Ueyama¹; M. Takeyama²; T. Matsuo²; ¹Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., Grad. Student, 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan; ²Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

The location of atoms and lattice parameters of Ni₃Nb with orthorhombic DO structure have been examined by powder X-ray diffraction. Rietvelt analysis on the stoichiometric Ni₃Nb revealed an asymmetric arrangement of Ni atoms on the closepacked (010) plane. The asymme-

try can be interpreted by the difference in total binding energy between the Ni atom and its next nearest neighbor atoms (NNNA), since there are two types of a-sublattice (Ni) site in terms of the kind of NNNA: one with four Ni and two Nb atoms (Ni-1 site) and the other with six Ni atoms (Ni-2 site). The excess Ni atoms occupy b-sublattice (Nb) site, and Fe atoms are found to preferentially occupy the Ni-1 site, rather than Ni-2 site. In both cases, the lattice parameter ratios b/a and c/a decrease, in good agreement with calculation based on hard sphere model. The results indicate that combined addition of these elements is effective for relaxation of asymmetry in the structure of Ni_3Nb .

11:50 AM

Influence of Chromium Content and Heat Treatments on Microstructure and Mechanical Behaviour of Fe3Al Based Ordered Intermetallic Alloys: *Antonio Augusto Couto*¹; Paulo Iris Ferreira¹; ¹IPEN-CNEN/SP, Matls. Sci. and Eng. Dept., Travessa R, 400, Cidade Universitária, São Paulo, São Paulo 05359-001 Brazil

The effect of chromium additions and two heat treatments, TT1 (800°C/1 h) and TT2 (800°C/1 h + 500°C/9 d), on the microstructure and on the mechanical properties obtained in tension tests, was investigated for initially hot worked Fe-30Al-0.15Zr-0.2B-(0-4.5)Cr (at.%) alloys. The presence of the D03 ordered phase, resulting from heat treatment TT2, is response for the reduction in the yield strength of these alloys when compared with the results obtained after heat treatment TT1. The decrease in room temperature yield strength with the increase in the amount of chromium observed for all alloys investigated, independently of heat treatments condition, is probably due to the enhancement of cross slip resulting from superdislocation dissociation. The reduction in the anomalous peak temperature with the increase in chromium content in the alloys seems to favor the explanation proposed for this phenomenon, based in superdislocation dissociation and climb-lock in temperatures below the peak temperature.

Ductility, Fatigue and Fracture I

Tuesday AM Room: Cypress 1
July 18, 2000 Location: The Westin Bayshore Hotel

Session Chair: Horst G. Vehoff; Dongliang Li

8:30 AM Invited

Fatigue and Fracture Mechanisms in Intermetallics-Based In-Situ Composites: *Kwai S. Chan*¹; ¹Southwest Research Institute, Mech. and Matls. Eng. Dept., 6220 Culebra Rd., P.O. Box 28510, San Antonio, TX 78228-0510 USA

The pertinent fatigue and fracture mechanisms in TiAl-based alloys and NbCr₂-based in-situ composites are summarized, with a particular emphasis focused on the role of alloy composition in the fracture process. It is demonstrated that the matrix composition exerts a significant influence on the static crack growth (resistance-curve) behavior of both materials, but considerably less is known about the effects of alloying on the fatigue crack growth response. A recent modeling effort to increase the fracture resistance of NbCr₂-based in-situ composites via a computation-based composite design approach is described. This approach relies on computation methods to design the matrix compositions and microstructures for optimum fracture resistance and ductility. Extension of this approach from NbCr₂-based composites to TiAl-based intermetallic alloys is discussed.

9:00 AM Invited

Design of High-Temperature Creep/Fatigue-Resistant Gamma TiAl Alloys: *Young-Won Kim*¹; ¹UES, Inc., Matls. and Process. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The fatigue properties of wrought gamma TiAl alloys with and without carbon additions were investigated in air at RT, 600°C, 800°C and 870°C for fine duplex and fully-lamellar microstructural forms. Tests on hourglass specimens show that both microstructures exhibit flat S-N curves at RT and 600°C. At and above 800°C, both materials exhibit a two-stage behavior, a gradual decrease of fatigue strength within the low cycle regime and a more rapid drop at higher cycle regime. The fatigue behavior was found to be dependent on microstructural form, grain size, tensile deformation behavior and creep resistance. For example, alloys containing carbon in the form of fine carbides appear to retard the occurrence of the second stage SN behavior. Their interrelations are discussed. Fatigue crack-initiation and subsequent propagation was related to the scale and distribution of microstructural features, and the effect of microstructure on fatigue life was temperature dependent. These aspects are analyzed and the implications are discussed.

9:30 AM Invited

Microstructural Effect on Moisture-Induced Embrittlement of the Ni₃(Si,Ti)-Based Alloys: *Takayuki Takasagi*¹; Yasuyuki Kaneno¹; Hirohumi Inoue¹; ¹Osaka Prefecture University, Dept. of Metall. and Matls. Sci., Gakuen-cho 1-1, Sakai, Osaka 599-8531 Japan

The effects of grain size, grain morphology and the second-phase dispersions on the moisture-induced embrittlement of the Ni₃(Si,Ti)-based alloys were investigated as a function of strain rate and temperature by tensile test in air, and SEM fractography. It was found that the fine grains, the unidirectionally solidified grains and the Nb-containing second-phase dispersions are effective in reducing the moisture-induced embrittlement of the Ni₃(Si,Ti)-based alloys. Possible mechanisms accounting for the beneficial effect of such microstructures on the moisture-induced embrittlement were discussed in association with the dynamic interaction of hydrogen with the microstructure as well as the chemistry of the Ni₃(Si,Ti)-based alloys.

10:00 AM

Crack Propagation in Binary Lamellar TiAl: Experiments and Computations: *Sharvan Kumar*¹; Jonathan Arata¹; Jessica Onstott¹; Alan Needleman¹; William Curtin¹; Kwai Chan²; ¹Brown University, Div. of Eng., P.O. Box D, Providence, RI 02912 USA; ²Southwest Research Institute, San Antonio, TX 78238 USA

Crack propagation behavior in a binary Ti-46.5Al alloy with a lamellar microstructure was examined by conducting fracture toughness tests within a scanning electron microscope equipped with a loading stage. Crack path was primarily interlamellar within a colony. Whereas crack extension across similarly oriented colonies was relatively easy, the K level of the approaching crack had to be increased significantly to extend the crack into an unfavorably oriented colony. Crack propagation across such a colony frequently required the renucleation of a microcrack at a location away from the crack tip, resulting in the formation of an interconnecting ligament that had to be fractured to further crack growth. Other features such as multiple cracks within a colony and propagation of cracks in the reverse direction were also observed. Finite Element Analysis (FEA) has been used to study crack nucleation and growth numerically. The analysis has been carried out within a framework where the continuum is characterized by two constitutive relationships: one relating the stress and strain in the bulk material and the other relating the traction and separation across a specified set of cohesive surfaces. These calculations were initially performed assuming that the material is fully elastic but they have since been extended to the elastic-plastic situation. The elastic-plastic calculations have been successful in capturing details that were not recognized in the elastic analysis but

were experimentally observed. This includes crack renucleation in a new colony and multiple crack nucleation within a given colony.

10:20 AM Break

10:40 AM

Modeling Thermal Exposure Effects in Gamma Titanium Aluminides: *P. Kennard Wright*¹; Andrew H. Rosenberger²; ¹GE Aircraft Engines, MPED, 1 Neumann Way, Mail Drop G50, Cincinnati, OH 45215 USA; ²Air Force Research Laboratory, Materials & Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Aircraft engine applications of gamma alloys require these materials to undergo extended periods of high temperature exposure in air. Therefore the effects of long time air exposure need to be defined, understood, and quantified. To date, the most complete investigation in this area has been conducted on cast Ti-48Al-2Nb-2Cr and wrought Alloy 395, Ti-46Al-2Nb-2Cr-1Mo-0.2B (at%). Both alloys showed reductions in fatigue capability that peaked in the intermediate temperature (~540°C) range, with smaller or no reductions at lower or higher temperatures. This paper examines the nature of this fatigue behavior and develops a model for it based on propagation of surface cracks initiating from an embrittled surface layer. The model successfully predicted the maximum effect at intermediate temperature and provides agreement with the post-exposure fatigue capability. Sponsored by NASA Contract NAS3-26385, and Air Force Contract F331615-97-C-5291.

11:00 AM

Effect of Microstructural Parameters on the Fatigue Crack Propagation in Fully Lamellar Gamma-TiAl Alloys: *Chong Soo Lee*¹; C. Choi²; H. J. Kim²; Y. -T. Lee³; Y. -W. Kim⁴; ¹MIT, Dept. of Matls. Sci. and Eng., Rm. 8-139, Cambridge, MA 02139 USA; ²Pohang University of Science and Technology, Ctr. for Adv. Aero. Matls., Pohang, Korea; ³KIMM, Changwon, Korea; ⁴UES, Dayton, OH USA

The effects of colony size and lamellar spacing on the fatigue crack propagation (FCP) behavior of gamma TiAl alloys have been investigated. It has been found that the overall crack growth rates of fully lamellar microstructure are not largely affected by the variation of the colony size up to about 400µm, though its respective roles on the intrinsic and extrinsic nature of crack growth resistance are quite different. However, in the coarse colony microstructure (~1400µm), the fatigue crack initiation resistance is markedly decreased while the crack growth resistance remains constant. The lamellar spacing is proved to be the more important factor to control both the fatigue crack initiation and growth resistance at room temperature. The fine lamellar spacing (0.2~0.7µm) microstructures represent superior fatigue crack initiation and growth resistance as compared to the coarse lamellar spacing (~5.5µm) microstructure. This superior fatigue resistance is mainly attributed to the higher number of lamellar interfaces resistant to crack advance as well as to the higher extrinsic closure effects. The colony boundaries and the lamellar interfaces play an important role in retarding the advancing crack at room temperature, serving as barriers for the dislocation movement and as sinks for dislocation pile-ups.

11:20 AM

Influence of Impact Damage on the High Cycle Behavior of Gamma-TiAl Sheet Material at Elevated Temperature: *Georg Glitz*¹; Hartmut Baur¹; Helmut Clemens²; ¹DaimlerChrysler, Struct. Metall. Div., Research Center-Ulm, Wilhelm-Runge-Str. 11, Ulm 89081 Germany; ²Universität Stuttgart, Institut für Metallkunde, Seestrasse 71, Stuttgart 70174 Germany

Domestic object damage (DOD) events are likely to happen in the turbine section of aeroengines through release of particles from sealing components and/or ceramic heat-shields of the combustion chamber. In this study the influence of DOD on the HCF behavior at 700°C of gamma-TiAl alloy sheets was investigated. In order to include the influ-

ence of the microstructure, sheet specimens with fine-grained near-gamma as well as coarse-grained fully lamellar microstructures were used. Prior to HCF testing the gauge area of prestressed specimens was impacted either by steel spheres or irregularly shaped ceramic particles of identical mass. The projectile velocities were chosen to be similar to those occurring in aeroengine turbines. Prior to HCF testing the damaged area has been examined with regard to indentation depth and diameter of the crack network. From this results a correlation between damage size and fatigue life behavior was established.

11:40 AM

Fatigue Crack Growth Resistance in Coarse-Grained and Fine-Grained Fully Lamellar Ti-Al-based Alloys: *Ze-Wen Huang*¹; Paul Bowen¹; ¹The University of Birmingham, IRC in Matls. for High Perform. Appl., Sch. of Metall. and Matls., Edgbaston, Birmingham B15 2TT UK

The fatigue crack growth behaviour of both coarse and fine-grained fully lamellar (FL) TiAl-based alloys has been studied. It has been found that mechanisms of fatigue crack growth change as the colony size is reduced significantly. For example, FL microstructures of 600 µm colony size are found to be highly orientation and microstructure-sensitive in near threshold and intermediate growth regimes. Crack growth resistance can thus be varied significantly by controlling the lamellae orientation, lamellae thickness and lamellae spacing. In contrast, fine-grained FL microstructures of, for example, 50 µm colony size display crack growth resistance which is relatively orientation and microstructure-insensitive. Controlling factors for operative mechanisms have been identified and these are discussed with reference to a detailed microstructural characterisation of the crack profile, fractography and studies of deformation local to crack-tips.

12:00 PM

Fracture in PST Crystal of TiAl: Experiments and Numerical Simulations: *Alfred Cornec*¹; Malte Werwer¹; Volker Heitmann¹; ¹GKSS Research Center, Instit. of Matls. Rsch., Max-Planck-Strasse, Geesthacht 21502 Germany

The fracture behavior of PST-crystals of TiAl has been investigated by in situ SEM experiments as well as by finite element simulations of micro-bend specimens with different orientations (crack divider, crack arrestor) of the lamellar plane. For the simulations, a representative volume element consisting of each one alpha-2, gamma-matrix and gamma-twin lamellae has been used as a material sub-model. The six different orientation variants of the gamma phase are also considered. The constitutive behavior of the phases is described by crystal plasticity in a rate-dependent viscoplastic formulation. The damage behaviour has been implemented by means of a cohesive zone model which describes fracture by a traction-separation law. The fracture behavior is well described by the simulations. By comparison with the experimental data, the cohesive parameters (cohesive stress, cohesive energy) have been determined.

Shape Memory

Tuesday AM Room: Cypress 2
July 18, 2000 Location: The Westin Bayshore Hotel

Session Chair: Easo George; Mathias Goken

8:30 AM Invited

Shape Memory Alloys: Multi-Purpose Functional Intermetallics: *Jan Van Humbeeck*¹; ¹MTM-KULEUVEN, De Croylaan 2, Heverlee 3001 Belgium

Shape memory alloys (SMA) are not exclusively derived from intermetallic compounds. Also solid solutions based on an element having allotropic phases or specific Fe-based alloys with a $g \ll e$ transition or certain alloys showing a cubic to tetragonal phase have clearly revealed similar functional properties as some definite intermetallic solid solutions with a bcc-parent phase. However so far only the intermetallic structures became successful industrial alloys. The first condition for good shape memory effects (SME) is the occurrence of a reversible (thermo-elastic) transformation. Other important factors for successful SME is the ability of matrix strengthening by ordering, precipitation or cold deformation. The combination of those requirements are excellently fulfilled by the NiTi intermetallic compound, being presently the far most applied system. Also b-Hume-Rothery phases such as the b Cu-, Ag-, Au-based alloys, or intermetallic phases such as b Ni-Al and b Cu-Zr are very much investigated. This paper will describe and compare those important intermetallic SMA and the alloys derived there of.

9:00 AM Invited

Recent Development of Ti-Ni Shape Memory Alloys: *Shuichi Miyazaki*¹; ¹University of Tsukuba, Instit. of Matls. Sci., Tsukuba, Ibaraki 305-8573 Japan

Ti-Ni shape memory alloys have many important attractive characteristics for practical applications, i.e., strength, ductility, corrosion resistance, biocompatibility, etc. in addition to excellent shape memory and superelastic functions. Recently, a variety of manufacturing methods have enabled us to produce fine or thin shapes and unique structure of Ti-Ni shape memory alloys: e.g., (a) rolled thin plates with a thickness less than 100 μm , (b) drawn fine wires with a diameter less than 50 μm , (c) drawn microtubes with an outer diameter less than 350 μm , (d) sputter-deposited thin films with a thickness less than 1 μm , (e) rapidly solidified ribbons of several tens μm thickness and (f) sintered porous structured materials. The characteristics and applications of these TiNi-based alloys are reviewed.

9:30 AM +

Multi-stage Martensitic Transformation in Ni-Rich Ti-Ni Alloy: *Jae-il Kim*¹; *Shuichi Miyazaki*¹; *Eiji Abe*²; ¹University of Tsukuba, Instit. of Matls. Sci., Tsukuba, Ibaraki 305-8573 Japan; ²National Research Institute for Metals, Tsukuba, Ibaraki 305-0047 Japan

The mechanical properties and martensitic transformation behavior of Ni-rich Ti-Ni alloys are strongly affected by aging, because the Ni-content of the matrix, the size and density of Ti_3Ni_4 precipitates vary depending on aging condition. It has been found that the multi-stage martensitic transformation (MSMT) occurs when the Ti-Ni alloys are subjected to some aging treatments. In order to clarify the cause for the appearance of the MSMT, the Ti-50.9at%Ni alloy was solution-treated at 1073K for 3.6ks followed by aging at 748K for periods between 0.3ks and 3600ks. The MSMT appeared in the specimens with aging time between 0.6ks and 18ks. TEM observation revealed that the Ti_3Ni_4 precipitates were formed in all specimens and their size increased with

increasing aging time. Two factors were considered to be the causes for the MSMT, i.e., the variations of stress and composition in the matrix around the precipitates.

9:50 AM

Thermomechanical Behavior of Ti-25Pd-24Ni-1W Shape Memory Alloy Fiber Reinforced Ti Matrix Smart Composite: *Kiyoshi Mizuuchi*¹; *Kanryu Inoue*²; *Ken-ichi Hamada*³; *Kiyoshi Yamauchi*⁴; *Kazuyuki Enami*⁵; *Masami Sugioka*¹; *Masao Itami*¹; *Yoshihira Okanda*¹; ¹Osaka Municipal Technical Research Institute, Mech. Eng. Dept., 1-6-50 Morinomiya, Joto-ku, Osaka, Osaka 536-8553 Japan; ²University of Washington, Mats. Sci. and Eng., 302 Roberts Hall, FB-10, Seattle, WA 98195-2120 USA; ³Tokushima University, Sch. of Dent., Dental Eng., 3-18-15, Kuramoto-cho, Tokushima, Tokushima 770-8504 Japan; ⁴Tokin Corporation, Mats. Devel. Lab., 6-7-1, Koriyama, Taihaku-ku, Sendai, Miyagi 982 Japan; ⁵Ryukoku University, Mech. and Sys. Eng., 1-5, Yokotani, Setaooe-cho, Otsu, Shiga 520-2123 Japan

A Ti matrix composite reinforced by a Ti-25Pd-24Ni-1W shape memory alloy (SMA) was fabricated by sheath-rolling of a SMA plate sandwiched with a pair of Ti plates. A reaction region was formed to be multi-layered in the vicinity of the interface between Ti matrix and TiPd SMA by heat treatment following sheath rolling. Tensile tests were performed at various temperatures for the composite, and it was found that the yield stress of the composite increased gradually with increasing temperature at temperatures higher than As(0). This stress increase is mainly caused by changes in residual compressive stress in the matrix. The change of this residual stress is associated with the martensite-to-austenite transformation of TiPd-based SMA during loading. The yield stresses were calculated by a one-dimensional analytical model and the predictions are in good agreement with the experimental results.

10:10 AM Break

10:30 AM +

Shape Memory Behavior and Microstructure in Sputter-Deposited Ti-Rich Ti-Ni Thin Films: *Junpei Sakurai*¹; *Jae-il Kim*¹; *Shuichi Miyazaki*¹; ¹University of Tsukuba, Instit. of Matls. Sci., Tsukuba, Ibaraki 305-8573 Japan

Unique microstructure is formed in amorphous Ti-rich Ti-Ni shape memory alloy thin films during the crystallization process. The crystallization temperature increased with increasing the Ti-content. In the crystallization process in an amorphous Ti-48.2at%Ni thin film, B2 nanocrystals were formed by annealing at about 673K. During the formation of nanocrystals, Ti-rich thin plate precipitates were formed along the interfaces of the nanocrystals. The thin plate precipitates improve the shape memory characteristics, achieving 6% recovery strain. While in an amorphous Ti-45.4at%Ni thin film, Ti_2Ni precipitates with random orientations were formed in an amorphous by annealing at about 700K, followed by the formation of B2 phase in the amorphous. Thus, the Ti_2Ni precipitates in the B2 phase are randomly distributed. The thin plate precipitates weren't observed in the Ti-45.4at%Ni thin film. The recovery strain was only 2% in the Ti-45.4at%Ni thin film due to the Ti_2Ni precipitates.

10:50 AM +

Texture and Microstructure of Ti-51.0at%Ni Melt-Spun Shape Memory Alloy Ribbons: *Anak Khantachawana*¹; *Koutaro Yamazaki*¹; *Hiroshi Mizubayashi*¹; *Shuichi Miyazaki*¹; ¹University of Tsukuba, Instit. of Matls. Sci., Tsukuba, Ibaraki 305-8573 Japan

The near equiatomic Ti-Ni is well known for the unique functions such as shape memory effect and superelasticity. These functions were studied in rapidly quenched melt-spun Ti-51.0at%Ni ribbons. As-spun ribbons of 15 μm thickness were annealed at 673K and 1073K, respectively, for 3.6ks. The texture was determined by X-ray diffraction pole figure measurements. A strong $\langle 100 \rangle$ fiber texture was found both in

the as-spun ribbon and the ribbon annealed at 673K. The specimen annealed at 1073K showed two types of textures, i.e. {001}<130> and {113}<031>. The internal structure of each specimen was investigated by TEM. Nonequilibrium disk precipitates of 10nm length located uniformly on {100}_{B2} planes inside grains, while Ti₂Ni precipitates of 25nm in diameter were also observed on grain boundaries. The disk precipitates could not be found and Ti₂Ni growth was confirmed when the ribbon was annealed at temperatures above 673K.

11:00 AM

Improvement of the Corrosion Resistance of TiNi Shape Memory Alloy by DC Plasma-Polymerized Hexamethyldisilazane Coatings: *Mu-Rong Yang*¹; *Shyi-Kaan Wu*¹; ¹National Taiwan University, Institut. of Matls. Sci. and Eng., 1 Roosevelt Rd., Sec. 4, Taipei, Taiwan 106

The DC plasma-polymerized hexamethyldisilazane (PHMDSN) films were deposited on the Ti₅₀Ni₅₀ shape memory alloy. The higher energy density input used in plasma polymerization will develop a film with a more inorganic nature from the IR spectrum analysis and water contact angle measurements. The corrosion performance of PHMDSN films in Ringer's solution at 37°C was evaluated by DC electrodynamic polarization and electrochemical impedance spectroscopy (EIS). The corrosion current density of the TiNi alloy can be lowered by as many as 4 orders of magnitude. The pitting potential and re-passivation potential can also be enhanced to nobler potential and exhibit good reproductivity after PHMDSN coatings. The EIS analysis indicates that the films have high impedance and less water uptake, which can be attributed to the hydrophobic and pinhole-free properties of the films, leading to the improvement of TiNi corrosion behavior.

11:30 AM

Effect of Temperature on Erosion Behavior of Pseudoelastic TiNi Alloy: *Tiancheng Zhang*¹; *D. Y. Li*¹; ¹University of Alberta, Dept. of Chem. and Matl. Eng., Edmonton, Alberta T6G 2G6 Canada

It has been observed that TiNi shape memory alloy exhibits excellent resistance to erosion, benefiting from its pseudoelasticity associated with a thermoelastic martensitic transformation. The pseudoelasticity helps to absorb the impact energy and thus diminish the erosion damage. Since the martensitic transformation is strongly dependent on temperature, the erosion behavior of this alloy varies as temperature changes. In general, TiNi alloy may perform well when the martensitic transformation can be induced by stress. However, it is not clear in which temperature range the alloy performs the best; or in other words, which of the martensitic transformation and the reorientation of martensite variants under stress is more beneficial to the erosion resistance is a question. In addition, how wide the temperature range is for maintaining good erosion resistance needs to be determined. In this work, erosion loss of Ti-51at%Ni alloy against temperature was evaluated using an air-jet erosion tester. The martensitic transformation temperature was determined by differential scanning calorimetry (DSC). Worn surfaces respectively eroded at different temperatures were analyzed using scanning electron microscope (SEM). The mechanism responsible for the erosion performance of the TiNi alloy as a function of temperature is discussed.

11:50 AM

Vacancy Migration and Long-Range Ordering due to Aging in AuCd Shape Memory Alloys: *Hiroki Ishibashi*¹; *Takio Hikono*¹; *Mineo Kogachi*¹; *Takuya Ohba*²; *Xiaobing Ren*³; *Kazuhiro Otsuka*³; ¹Osaka Prefecture University, Dept. of Matls. Sci., Gakuen-cho 1-1, Sakai, Osaka 599-8531 Japan; ²Teikyo University, Dept. of Mats. Sci. and Eng., Toyosatodai 1-1, Utsunomiya, Tochigi 320-8551 Japan; ³University of Tsukuba, Insti. of Mats. Sci., Tennoudai 1-1-1, Tsukuba, Ibaraki 305-8573 Japan

The martensite ageing effect in AuCd alloys is believed to be developed by a short-range atom diffusion without changing the average

structure. Since the diffusion process strongly correlates with point defects such as vacancies, it is important to examine their behavior. We have examined vacancy migration during ageing in martensite and parent B2 phases for furnace-cooled and quenched samples of Au_{1-c}Cd_c alloys (0.47 < c < 0.51) by measuring the lattice constants by X-ray diffraction. The long-range ordering (LRO) is also examined by measuring the integrated intensities. For example, in the sample of c = 0.498, the martensite ageing does not bring about an effective migration and a detectable LRO change. For the B2-phase of the quenched sample, the quenched-in vacancies are drastically annealed-out by ageing, which accompanies a promotion of the LRO. The difference in the vacancy migration is due to the difference in the crystal structure.

Industrial Applications

Tuesday PM
July 18, 2000

Room: Salon 1
Location: The Westin Bayshore Hotel

Session Chairs: Rod Judkins; V. K. Sikka

1:30 PM Invited

Alloy Design and Industrial Processing of Fe-40Al Sheets: *S. C. Deevi*¹; *D. G. Morris*²; *V. K. Sikka*³; ¹Philip Morris USA, Rsch. Ctr., Richmond, VA 23264 USA; ²CENIM, Dept. of Phys. Metall., CSIC, Avda Gregorio del Amo, Madrid 28040 Spain; ³Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37830 USA

Intermetallics based on iron aluminides are attractive candidates for high temperature applications due to their high strength to weight ratio, and excellent oxidation and corrosion resistances. Environmental embrittlement, low room temperature ductility, and lack of economical processing techniques hampered the commercial utilization of iron aluminides based on FeAl in cast and wrought forms. As part of the Philip Morris program on intermetallics, we carried out alloy design of FeAl with the addition of boron, carbon, and molybdenum to optimize the room temperature ductility and high temperature strength of FeAl alloys. Alloys with an optimum combination of properties were powder processed to obtain thin sheets of FeAl by employing a combination of ceramic and metallic processing techniques. At present, excellent sheets of FeAl are commercially manufactured by roll compaction followed by sintering/annealing techniques using water atomized FeAl powders. In this paper, we will discuss the evolution of microstructure during the rolling process, and the importance of intermediate and final sintering/annealing steps for the recovery and recrystallization processes. In addition, we summarize the physical, electrical, tensile, creep, and fatigue properties of FeAl sheets obtained by roll compaction, and will be compared to the properties of hot extruded FeAl.

2:00 PM Invited

Development of TiAl Turbocharger for Passenger Vehicles: *Toshimitsu Tetsui*¹; ¹Mitsubishi Heavy Industry Limited, Nagasaki R&D, Fukahori-machi 5-717-1, Nagasaki 851-0392 Japan

Weight reduction of the turbine wheel is the most effective way to improve the response ability of turbochargers for passenger vehicles. However no such lightweight materials possessing the requisite heat-resistance and formability have been available until now. Accordingly, a new high-performance alloy based on a lightweight TiAl intermetallic compound has been developed together with the required manufacturing technology. Applicability for turbochargers has been confirmed by engine tests, leading to successful practical application. High-response turbochargers have been produced by Mitsubishi Heavy Industries Ltd.

using this new material, and the decision has been made to use them in Lancer Evolution VI automobiles made by Mitsubishi Motors Cooperation. from January 1999.

2:30 PM Invited

New Surface Treatment Using Shot Blast for Improving Oxidation Resistance of TiAl-Base Alloys: *Hiroyuki Kawaura*¹; Hiroshi Kawahara¹; Kazuaki Nishino¹; Takashi Saito¹; ¹Toyota Central Research & Development Laboratories, Inc., Mats. Div. 1, 41-1 Yokomichi, Nagakute, Aichi 480-1192 Japan

A new surface treatment by the shot blast using WO₃ powder has been developed, which has been most effective in improving the cyclic oxidation resistance of TiAl-base alloy in air and an exhaust gas atmosphere of diesel engine up to 1223K. This effect is contributed to the formation of Al₂O₃ layer during oxidation atmosphere. The above layer is supposed to be formed as follows: Since the WO₃ powder was shot repeatedly by the surface treatment, the WO₃ layer adhering to the substrate was formed on the surface. This layer causes the preferential oxidation of aluminum at the early stage of oxidation, resulting in the formation of a sound and continuous Al₂O₃ layer. The developed surface treatment gives the same oxidation resistance as that of nickel-base superalloy Inconel 713°C up to 1223K, and is applicable to complex-shape parts with the as-cast skin surface, such as turbocharger wheels.

3:00 PM

Development of Alumina Forming Coatings for Titanium and Titanium Aluminides: *L. Niewolak*¹; U. Flesch¹; V. Shemet¹; L. Singheiser¹; W. J. Quadackers¹; ¹Research Centre Juelich, IWV-2, Juelich 52425 Germany

In the development of advanced gas turbines towards higher efficiencies and less environmental impact, there is an increasing demand for lightweight materials which are suitable for rotating and oscillating components. Lightweight γ -TiAl alloys are promising high-temperature materials that may replace conventional heat-resistant steels and superalloys in engines. The good strength and low density make these materials attractive for intermediate temperature (600-800°C) applications in gas turbines. One important hindrance for the use of γ -TiAl alloys at high temperatures is its relatively poor oxidation resistance and sensitivity against environmental embrittlement. The latter effect also occurs in elevated temperature application of Ti and Ti- alloys. These problems can principally be avoided by the application of oxidation resistant alumina forming coatings. One alloy system which seems to possess suitable properties for this purpose is Ti-50Al-2Ag. This alloy exhibits excellent oxidation resistance at 800°C in air, due to formation of a protective alumina based scale. The effective use of Ti-50Al-2Ag as coating system depends on two crucial issues, i.e. the thermal expansion mismatch and interdiffusion between coating and base alloy. In the present study coatings of nominal composition Ti_{47.6}Al₅₀Ag_{2.4} have been applied to both Ti- and γ -TiAl- based alloys by the magnetron sputtering processes. Subsequently, the coatings were subjected to long term cyclic oxidation tests. The significance of the results in terms of selecting the process parameters for developing an optimal coating system will be discussed.

3:20 PM

Nickel Aluminides: A Step Forward Towards Industrial Application: *Frank Scheppe*¹; Peter R. Sahn¹; Wolfgang Hermann²; Uwe Paul²; Jürgen Preuhs³; ¹RWTH Aachen Giesserei-Institut, Intzestr. 5, Aachen, NRW 52056 Germany; ²Siemens AG KWU, Wiesenstr. 35, Mülheim a.d. Ruhr, NRW 45473 Germany; ³Doncasters Precision Castings-Bochum, Bessemerstr. 80, Bochum 44793 Germany

NiAl-Ta-Cr alloys are subject of an ongoing development of new high-temperature materials for application in gas turbines. Simple component were assembled in production size clusters for investment castings. The high temperature, thermal expansion and chemical behavior

of the mold system has to be compatible with the properties of the cast alloy. A special ceramic mold system was used to prevent casting defects and excessive mold-metal reactions. The ductile-to-brittle transition requires a rather small cooling rate to avoid shrinkage and tears. The numerical simulation has still to be adapted to the special parameter of the investment casting process as to the behavior of the NiAl-Ta-Cr-Alloys. Results of numerical simulations for different casting conditions were compared with results of casting trials in aspects of porosity, mold filling and temperature-field development during the casting process. The influence of convection during the mold filling of the segregation will be shown in the work.

3:40 PM Break

4:00 PM

An Assessment of the Elevated Temperature Erosion Resistance of Iron-Aluminide Cermets: *David E. Alman*¹; J. H. Tylczak¹; J. A. Hawk¹; J. H. Schneibel²; ¹U.S. Department of Energy, Albany Rsch. Ctr., 1450 Queen Av. S.W., Albany, OR 97321 USA; ²Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831 USA

The resistance to wear by the erosion of hard particles of FeAl based cermets was evaluated. Cermets were produced by infiltrating either WC, TiC or TiB₂ powder preforms with molten FeAl. Erosion testing was performed in accordance with ASTM standard G76-83, as Al₂O₃ particles entrained in a N₂ gas stream impinged the surface of the wear specimens. Test were performed at 25, 180, 325, 500 and 700°C, and the wear was measured as material loss after a fixed erosion time. The wear of the FeAl-cermets was compared to the wear of WC-Co. It was found that for WC-Co wear resistance decreased (i.e. magnitude of wear/material loss increased) with increasing temperature; where as, for the FeAl-cermets, the magnitude of wear essentially remained constant with temperature. These results are discussed in terms of the mechanisms of material removal during the erosion process.

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Role of Laves Intermetallics in Nuclear Waste Disposal: *Daniel P. Abraham*¹; ¹Argonne National Laboratory, Chem. Tech. Div., CMT-205 Rm. A-167, 9700 S. Cass Ave., Argonne, IL 60439 USA

Laves intermetallics play a non-traditional role in the disposal of metallic nuclear waste resulting from the electrometallurgical treatment of EBR-II spent fuel. These ZrFe₂-type intermetallics incorporate and immobilize highly radioactive and long-lived constituents that are present in waste forms based on the stainless steel ? 15 wt% zirconium (SS-15Zr) alloy. This paper reviews the metallurgy of SS-15Zr alloys with emphasis on Laves intermetallic behavior. SEM studies have shown that all actinide elements and most fission product elements are present only in the ZrFe₂-type intermetallics; moreover, both actinide-rich and actinide-deficient areas are found within the Laves compound. These observations have been correlated to the simultaneous presence of multiple Laves polytypes, each with a different preference for the actinide elements. Recent experiments have been aimed at determining the corrosion behavior of these intermetallic compounds. Results from Auger Electron Spectroscopy and Transmission Electron Microscopy of corrosion layers that form on these intermetallic phases will be presented.

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Prototype Plant for the Economical Mass Production of TiAl-Valves: *Matthias Blum*²; Peter Busse¹; Georg Jarczyk²; Harald Scholz²; Stefan Pleier²; Hans-Josef Laudenberg³; Klaus Segtrop³; Rainer Simon⁴; ¹ACCESS, Intzestr. 5, Aachen 52072 Germany; ²ALD Vacuum Technologies AG, Rückinger Straße 12, Erlensee 63526 Germany; ³TRW Deutschland GmbH, Hannoversche Strasse 39, Barsinghausen 30881 Germany; ⁴BMW AG, Forschungs- und Ingenieurszentrum, Knorrstrasse 147, Munich 80788 Germany

Initiated by ALD Vacuum Technologies AG the technology for an economical production of TiAl-Valves has been developed by a joint research project during the last 4 years. The integration of a modified induction cold crucible and a gradient heatable permanent metallic mold in a vacuum centrifugal casting unit is the main feature of this process. Due to their fine and full lamella as cast microstructure the produced valves impressively outperform the required mechanical properties of the automotive companies. The valves passed successfully a 500h motor test in a BMW M4 engine. As next step of process development a new joint research project with participation of five German automotive companies will evaluate the feasibility of mass production at a competitive price level. Therefore a prototype plant with a productivity of approximately 600 000 valves/year had been designed and will be build and run in during the next 2 years. The currently and the precursor project are financially supported by the German Ministry for Education and Research. The quality of the valves will be discussed and the design concept of the prototype plant presented.

preferentially for aluminum, and Cr and Cu are expected to substitute equally for both nickel and aluminum sites.

2:10 PM

Influence of Order Parameters and Alloys Concentration of Alloy Components on Energy Formation of Planar Defects: *Michail D. Starostenkov¹; Veronika V. Romanenko¹; Evgenya V. Chernyh¹; Michail A. Baranov¹; Eugenii A. Dubov¹; ¹Altai State Technical University, Gen. Phys. Dept., Lenin st. 46, Barnaul 656099 Russia*

It was carried out investigation of energy formation dependence of planar defects on values of alloy order parameters and different concentrations of alloy components of D03 type ordered materials by the method of computer simulation. Fe₃Al, Fe₃Si and Cu₃Al alloys were studied. Atoms interaction in alloys were determined by properties of pure components and experimental values of vacancies energy formation, energy sublimation and energies of antiphase boundaries in studying alloys. Two types of energy formation dependence of planar defects on values of alloy order parameters and different concentrations of alloy components were obtained: (1) energy formation of complex fault defect is higher than energy formation of superstructure fault defect in narrow interval near stoichiometric composition of ordered solutions. The same happens when long order parameter is changed: energy formation of complex fault defect is higher than energy formation of superstructure fault defect only in cases of small deviations from ordered alloys. (2) energy formation of complex fault defect is higher than energy formation of superstructure fault defect in wide interval when alloy composition and long order parameter are changed.

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Mechanical Properties of L₁ Modified Titanium Trialuminides Alloyed with Chromium, Iron and Vanadium: *Tohru Takahashi¹; Koji Tominaga²; Yasuhiko Tsuchida²; Shinji Motizuki²; Fumiaki Kawai²; Tadashi Hasegawa¹; ¹Tokyo University of Agriculture and Technology, Dept. of Mech. Sys. Eng., Naka-cho 2-24-16, Koganei, Tokyo 184-8588 Japan; ²Tokyo University of Agriculture and Technology, Graduate Student*

Mechanical properties have been investigated on L₁ modified titanium trialuminides ternary or quaternary alloyed with chromium, iron and vanadium. Arc-melted materials were converted into equi-axed polycrystals by isothermal forging and annealing. Lattice parameter in iron-modified material was a little smaller than that in chromium-modified one. Vanadium substitution into chromium- and iron-modified materials slightly increased the lattice parameters, which should be in close relationship with the solution strengthening observed in the materials quaternary alloyed with vanadium. Compressive strain at room temperature varied from 0.05 to 0.3. Flow stress fluctuation of about a few per cent was observed in an intermediate temperature range around 600K. Strengthening effect of vanadium was maintained up to higher temperatures and also under creep conditions. Dynamic recrystallization was observed at grain boundaries in the materials deformed at higher temperatures than 1200K.

2:50 PM

Drastically Improved Ductility of Ir-Base Alloy by Mixing Ir-Nb with Ni-Al: *X. H. Yu¹; Y. Yamabe-Mitarai¹; S. Nakazawa¹; Y. Ro¹; H. Harada¹; ¹National Research Institute for Metals, High Temp. Matls. 21 Pjct., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan*

As a matrix of high temperature structural materials Ni₃Al has some inherent advantages and the new developed high performance DS cast Ni₃Al base alloy IC6 has been used for gas engine turbine blades and vanes operating in the temperature range of 1000~1100°C. However, it may be fair to say that the Ni-base superalloys has almost reached its limitation in temperature capability as long as Ni (T_m=1453°C) is used as the base metal. A novel type of refractory superalloys, using Ir

Alloying Effects II and Defects II

Tuesday PM Room: Salon 3
July 18, 2000 Location: The Westin Bayshore Hotel

Session Chairs: Man H. Yoo; Mino Doi

1:30 PM

Site Occupancy Preferences in the B2 Ordered Solid Solution Phase of Nb-Rich Nb-Ti-Al Alloys: *Keith John Leonard¹; Vijay K. Vasudevan¹; ¹University of Cincinnati, Dept. of Matls. Sci. and Eng., 515 Rhodes Hall, P.O. Box 210012, Cincinnati, OH 45221-0012 USA*

The β-(Nb,Ti) solid solution phase in the Nb-Ti-Al system has recently been observed to extend to Al concentrations as high as 40 at.%. Fifteen alloys ranging in composition from 15 to 40 at.% Al with Nb:Ti ratios from 1:1.5 to 4:1 were investigated. The β-transus temperatures, lattice parameters and B2 ordering temperatures of the β phase are reported. Site occupancy preferences of the B2 lattice as a function of alloy composition were examined through the Atom Location by Channeling Enhanced Microanalysis (ALCHEMI) technique with results presented as ordering tie lines. Ti substitution for Nb on Nb sublattice sites was observed within all of the alloys, with the strength of partitioning between Ti and Al atoms dependant on alloy composition. A correlation between the partitioning behaviors of the atomic species and the Nb:Ti ratio of the alloy compositions was observed.

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Site Preference in NiAl: *Yoshihiro Terada¹; Kenji Ohkubo¹; Tetsuo Mohri¹; Tomoo Suzuki²; ¹Hokkaido University, Div. of Matls. Sci. and Eng., Grad. Sch. of Eng., Sapporo 060-8628 Japan; ²Kochi University of Technology, Tosayamada, Kochi 782-0003 Japan*

Contour map of thermal conductivity in ternary intermetallic phase is characterized by the direction of the contour ridge. It has been demonstrated in Ni₃Ga-X alloys that the direction of ridge in thermal conductivity contours in γ single phase field agrees with that of solubility lobe of γ phase in the ternary phase diagrams. This indicates that the ridge direction is a reliable indication of site preference in intermetallic compound. Site preference of fifteen kinds of ternary elements in NiAl is determined from the ridge direction of thermal conductivity contours in β single phase field. V, Nb, Ta, Mo, Fe, Ru, Co and Pt substitute preferentially for nickel in NiAl, Ti, Mn, Ga, Si and Ge substitute

($T_m=2447^\circ\text{C}$) as base metal with L12 precipitates. prepared by mixing Ir-base alloy with Ni-base alloy was investigated. The object is to balance the high temperature strength of Ir-base alloy with high ductility of Ni-base alloy. Microstructure was consisted of fcc/L12 coherent structure formed by cuboidal L12-Ir3Nb with fcc matrix. And the alloys exhibited very high 0.2% flow stress at ultra-high temperatures, e.g., 1050MPa at 1200°C with a drastically improved ductility (around 20%). The compression test at 1500°C and 1800°C was also carried out. The effect of cooling rate on morphology of Ir3Nb and time dependence of growth rate of Ir3Nb at 1600°C in Ir-base refractory superalloys were investigated. The diffusion coefficient of Ir in Ni3Al was also investigated. The potential usage of Ir-base refractory superalloys as a high temperature structural material is discussed.

3:10 PM Break

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Ductile L12 Rh3Ti Intermetallics for Ultra-High Temperature Applications: *Shankar M.L. Sastry*¹; Rabindra Nath Mahapatra²; Andrew W. Davis²; ¹Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; ²Naval Air Systems Command, Matls. Lab., Code 4.3.4.2, Patuxent River, MD 20670 USA

Rh-25 Ti has a melting point $> 1700^\circ\text{C}$ and is a single phase ordered intermetallic compound having a Cu3Au type crystal structure and therefore is an attractive candidate for developing “ductile” intermetallics for service temperatures in excess of the use temperatures of nickel base super alloys. The coefficient of thermal expansion, thermal conductivity, and room temperature specific strengths and hardnesses of Rh-Ti alloys are comparable to or superior to that of nickel base superalloys. Two phase gamma/gamma' type microstructures found in nickel-based superalloys can also be produced in Rh-Ti alloys. All of these features render the Rh-Ti system attractive for exploration as candidate structural materials for ultra high temperature applications. In the present investigation, the microstructures and deformation behavior at 25-1000°C of Rh-x Ti (x=15, 20, and 25 at.%) alloys were studied. The alloys were prepared by non-consumable electrode-arc melting. The arc-melted buttons were isothermally forged at 1100°C, and the mechanical properties at 25-1000°C were evaluated by microhardness measurements, compression testing, and 3-point bend testing. Unlike many other high temperature intermetallics, the Rh-Ti alloys do exhibit room temperature ductility and the high temperature strength and creep resistance are superior in Rh-Ti alloys to some of the other high temperature intermetallics. The deformed specimens were examined by scanning electron microscopy and atomic force microscopy for the determination of the extent and nature of slip and by transmission electron microscopy for the identification of dislocation activity.

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Effect of Quaternary Additions on Microstructure and Mechanical Properties of Orthorhombic Ti₂AlNb-Based Alloy: *Feng Tang*¹; Shizuo Nakazawa¹; Masuo Hagiwara¹; ¹National Research Institute for Metals, 3rd Rsch. Grp., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan

The orthorhombic Ti₂AlNb-based (O-based) alloys are potential lightweight materials for high-temperature applications. Among them, Ti-22Al-27Nb is said to have good balance of tensile, creep and fracture toughness properties. However, the O-based alloys exhibit shorter 1% creep strain life than IN 718. As an attempt to enhance the mechanical properties of O-based alloy further, six new alloys were prepared by substituting Mo, W or V for a part of Nb in Ti-22Al-27Nb alloy at the rates corresponding to their β phase stabilizing abilities. The effects of these quaternary additions, as well as heat treatment, on microstructure, hardness, tensile properties and creep behavior were investigated. The experimental results showed that W and V were effective on enhancing

the strength at temperature over 650°C, while V was beneficial to the room-temperature ductility. W modified alloys showed the longest 1% creep strain life which is comparable to the density normalized value of IN 718.

4:10 PM Invited

Effects of Minor Addition of Ni on Hot-Deformation Behaviors of Gamma TiAl Alloy: *Ji Zhang*¹; ¹Central Iron and Steel Research Institute, Dept. of Superalloys, No.76 Xueyuan Nan Lu, Beijing 100081 China

The gamma TiAl alloys normally exhibit acceptable hot-deformability over 1200°C. However, this temperature is beyond the common industrial productions. Our previous work showed that the Ti-46.2Al-2.0V-1.0Cr-0.5Ni (at%) alloy with an equiaxed near gamma microstructure has much wider deformable conditions (so-called process window) compared to the reference lamellar microstructure in Ti-46.5Al-2.5V-1.0Cr (at%) alloy. The contribution of the Ni addition suggested in that study is to promote the microstructure transformation in the cast TiAl alloy for reducing the deformation anisotropy. In this research, the effects of the minor addition of Ni on the hot-deformation behaviors at the temperatures below 1050°C are investigated by comparing the isothermal compressive tests of the Ni-bearing and Ni-free alloys with similar and different microstructures. It is found that the as-cast Ni-bearing alloy also has a much larger process window compared to the Ni-free alloy with the similar lamellar microstructure. In the same deformation conditions, the peak stresses of the as-cast Ni-bearing alloy are 40°C 60% lower than those of the Ni-free alloy but 8°C 10% higher than those of Ni-bearing alloy with the equiaxed microstructure. While, there is no big flow-stress difference among the tested alloys. The microstructure changes and the defects produced by the hot-deformations will be examined in addition for analyzing the deformation mechanisms.

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Hardness Behaviour in B2 Pseudo-Binary Systems: *Jeanette Hohls*¹; P. J. Hill¹; I. M. Wolff¹; ¹MINTEK, Adv. Matls. Grp., Phys. Metall., Private Bag X3015, Randburg 2125 South Africa

Distinguishing the various possible contributions to solid solution hardening in strongly ordered systems is problematic. Earlier work (1-3) reported on the mechanical properties of pseudo-binary alloys based on the B2 RuAl system, in which substitutional additions of Ni, Ti and Co were examined. Despite comparable misfit strain in the B2 lattice, Co, Ti and Ni additions differed profoundly in their effect on the hardness. Whereas the Ru1-xCoxAl and RuAl1-xTix series exhibited close to ideal behaviour, strong hardening effects occurred in the Ru1-xNixAl system. Similar hardening effects reported in related systems, notably RuTi-NiTi (4) and FeAl-NiAl (5-7), led to the conjecture that the hardness effects might be intrinsic to Ni in the B2 structure. In order to further characterise hardening behaviour in B2 compounds, room-temperature hardness profiles were additionally obtained for the systems IrAl-RuAl, RuNb-RuAl, and IrAl-NiAl. Possible origins for the divergent hardening behaviour, including ternary ordering, are examined.

Deformation III and Fatigue and Fracture II

Tuesday PM Room: Cypress 1
July 18, 2000 Location: The Westin Bayshore Hotel

Session Chairs: Amo Bartels; Kwai S. Chan

1:30 PM

Effect of Temperature and Alloy Composition on Deformability of HfV₂ + Ta C15 Laves Phase Alloy: Won-Yong Kim¹; David E. Luzzi¹; David P. Pope¹; ¹University of Pennsylvania, Dept. of Matls. Sci. and Eng., LRSM Bldg., Philadelphia, PA 19104 USA

The occurrence of twinning in Laves phase alloys is sensitive to alloy composition and deformation conditions such as strain rate and temperature. However, it remains unclear based on the existing work in the HfV₂+Nb system whether the twinning is solely stress-driven or whether there exists a critical temperature for twinning. Results in the HfV₂ system are somewhat ambiguous due to fairly complex phase equilibria and the predicted presence of a low temperature phase instability. We have recently found that alloying HfV₂ with Ta yields more extensive room temperature ductility by twinning in ternary Laves phase alloys that are more easily studied. In the present paper, Ta is again chosen as the ternary-alloying element in HfV₂. HfV₂+Ta Laves phase based arc melting and float-zone-melting produce alloys with various compositions. X-ray diffraction is used to analyze the crystal structures and lattice parameters for each sample investigated. Compression tests are conducted at temperatures down to near liquid helium temperatures using a specially designed testing apparatus. The effect of temperature and composition on mechanical properties is investigated to gain a better understanding of the mechanisms of twinning. Alloy microstructures are characterized using conventional and high-resolution TEM. Based on the obtained results, the deformation mechanisms will be discussed with particular attention given to the nucleation of twinning in the C15 cubic Laves phase.

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Design of Laves Phase Strengthened Ferritic Heat Resisting Steels: Keisuke Yamamoto¹; Yoshisato Kimura²; Yoshinao Mishima²; ¹Tokyo Institute of Technology, Matls. Sci. and Eng. Depts., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan; ²Tokyo Institute of Technology, Matls. Sci. and Eng. Dept., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

A new class of heat resisting ferritic steels is investigated in the Fe-Cr-Nb and Fe-Cr-Nb-Ni systems in which the major strengthener is the Fe₂Nb Laves phase. Ferritic steels strengthened by Laves phase are expected to show the excellent high temperature strength, while the brittleness of Laves phase may lower the toughness of the alloy. The α -Fe/Fe₂Nb two-phase microstructure is selected to improve mechanical properties through changing volume fraction and morphology of Laves phase. Effects of Nb and Ni contents on the microstructure and mechanical properties of the alloys, fixed at 10 at%Cr, have been systematically investigated. Mechanical properties were evaluated by tensile tests conducted at room temperature, 873 and 973K. The tensile test revealed that the room temperature ductility decreases with increasing Nb content. The alloys with 1.0 to 1.5 at%Nb are found to exhibit a good balance between room temperature ductility and high temperature strength.

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Orientation Dependent Deformation of Single Crystal V₃Si: Zhijie Jiao¹; Sung H. Whang¹; Easo George²; ¹Polytechnic University, Dept. of Mech. Eng., Brooklyn, NY 11201 USA; ²Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831 USA

A15-type V₃Si has been perceived as a promising high-temperature structural material because of its high melting point, high strength and low bulk density. But, the downside is its brittleness up to relatively high temperatures. To date, the micro-mechanisms of deformation aren't well understood. In current experiments, the compressive deformation of V₃Si single crystal was conducted at 1673K with strain rate, 4.0x10⁻⁵s⁻¹ in three different orientations to study possible orientation dependence in CRSS. The line and planar defects in the postmortem specimens were investigated by transmission electron microscopy under bright field and weak beam modes. The results on dislocation dissociation, interaction and cross-slip will be reported. The relationship between the orientation dependence and defect structure will be discussed. Research sponsored (at ORNL) by the Division of Materials Sciences, U.S. Department of Energy under contract DE-FG02-93ER45499 and DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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Microstructure and Mechanical Properties of Molybdenum Disilicide Alloys Made by MA-PDS Process: Manabu Matsubara¹; Yong-Ho Park¹; Hitoshi Hashimoto¹; Toshihiko Abe¹; ¹Tohoku National Industrial Research Institute, Matl. Eng. Dept., 4-2-1 Nigatake, Miyagino-ku, Sendai, Miyagi 983-8551 Japan

In order to improve the mechanical properties of molybdenum disilicide alloys as a promising high temperature structural material, the alloys where boron, aluminum or niobium added were prepared by an advanced consolidated process which combined mechanical alloying with pulse discharge sintering (MA-PDS). Their microstructure and mechanical properties were investigated. The samples which were prepared by MA-PDS had very fine and dense microstructures compared with the sample made from commercial molybdenum disilicide powders. Both monolithic molybdenum disilicide and niobium added alloys fabricated by MA-PDS showed a significant amount of plastic deformation in a bending test at 1073K. The yield stress of niobium added alloy was higher than that of monolithic molybdenum disilicide. It could be caused by the reduced grain size of niobium added alloy. The high temperature tensile test is studying in the case of another elements added alloy.

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Computation of the Fracture Stress of Notched NiAl Polycrystals: Horst Gerhard Vehoff¹; Manfred Grosse Gehling¹; ¹Universitaet des Saarlandes, Dept. of Matl. Sci. and Eng., Im Stadtwald Bldg. 43 B, Saarbruecken., Saarland D-66041 Germany

Computer simulations were performed to investigate the influence of the geometry and the microstructure of macroscopic specimens on the brittle-to-ductile transition in NiAl. A new model is proposed that permits to bridge the gap between the length scales of mesoscopic and macroscopic modeling. Considering a Finite Element model that exhibits power law hardening the fracture mode of the simulated specimen depends on the stability of microcracks of given length and orientation that are assumed to exist within the microstructure. Cleavage fracture occurs when indicated by a simulation routine on the mesoscopic length scale. A dislocation emitting crack loaded in mode I and II, generally, is simulated. Instead of the semi-infinite crack constitutive equations are derived for a finite crack in NiAl polycrystals. Finally, fracture loads are calculated with the FE-model and their scatter observed experimentally is discussed and explained physically.

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High-Temperature Bending Fracture Properties and Deformation Structures of an Ti-47at.%Al Intermetallic Compound: *Yoichi Kishi*¹; *Zenjiro Yajima*¹; *Ken'ichi Shimizu*¹; *Koichi Morii*²; ¹Kanazawa Institute of Technology, AMS R&D Ctr., 3-1 Yatsukaho, Matto, Ishikawa 924-0838 Japan; ²Daido Steel Co., LTD, 2-30 Daido, Minami-ku, Nagoya, Aichi 457-0081 Japan

The bending fracture behavior and deformation structures of as-casted and heat-treated Ti-Al intermetallic compounds were examined at 973K. Microstructure of the as-casted Ti-Al consisted of a nearly fully lamellar structure, and that of the heat-treated Ti-Al was lamellar-colony and g-grain. The bending fracture strength of the as-casted Ti-Al was superior to that of the heat-treated Ti-Al. On the specimen surface near the fracture, many micro-cracks were observed and they showed a tendency to concentrate on lamellar colony boundaries and at their triple points. When internal microstructures near the fracture were observed by using a transmission electron microscope, there were observed many dislocations parallel to the interfaces between γ and α_2 plates and deformation twins with an angle to the interfaces. On the basis of the above results, discussion was done on the bending fracture mechanism of as-casted and heat-treated Ti-Al intermetallic compounds at 973K.

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Microstructures and Fracture Behaviors of B-Free and B-Doped Ir3Nb (L12) Intermetallic Compounds: *Yue Feng Gu*¹; *Y. Yamabe-Mitarai*¹; *S. Nakazawa*¹; *H. Harada*¹; ¹National Research Institute for Metals, HTM 21, 1-2-1 Sengen, Tsukuba-shi, Ibaraki 305-0047 Japan

A large number of L12-type compounds show substantial increases in flow strength with increasing temperature. This behavior makes these compounds attractive as new high temperature structural materials. However, these materials are often subject to intergranular embrittlement. Takasugi and Izumi investigated the grain-boundary fracture behaviours of the binary L12-type A3B ordered alloys and found that failure behaviour in the alloys fundamentally depends on the heterogeneity of bonding between A and B two atoms. This model suggests that the binary L12-type A3B ordered alloys, which consists of two similar transition atoms, should have high grain boundary strength and normally fracture in transgranular mode. This study was designed to investigate the fracture behaviors of B-free and B-doped Ir3Nb alloys, where bonding environment between Ir and Nb atoms is less heteropolarity than that between Co and Ti atoms, and thus superior mechanical properties are expected for Ir3Nb intermetallic compound than that of Co3Ti alloy. Our results showed that L12-type Ir3Nb intermetallic compound fractured in intergranular mode in air and under vacuum ($< 10^{-4}$ Pa). Adding boron can change the fracture mode from intergranular fracture for the B-free compound to transgranular cleavage for the B-doped compound. But more boron addition, above 200 wppm for Ir3Nb, would cause iridium-boride phases with lower melting temperature to be formed, which made a rapid falloff in high-temperature strength. Basing on the investigation, the effective mechanisms for boron in Ir3Nb were suggested.

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Mechanical Behaviour of Orthorhombic Ti2AlNb Based Intermetallic Alloys: *Vikas Kumar*¹; *Luc Remy*¹; *Jean-Loup Strudel*¹; *Ashok Kumar Gogia*²; ¹Ecole National Supérieure des Mines de Paris, Centre des Matériaux, UMR CNRS 7633, B.P.87, Evry, Cedex 91003 France; ²Defence Metallurgical Research Laboratory, P.O. Kanchanbagh, Hyderabad, AP 500058 India

Ti3Al based titanium aluminides have been investigated extensively because of their potential for aero-engine applications. Alloys hardened by O phase (Ti2AlNb) yield better combination of strength, ductility, creep and fracture toughness. In this paper, effect of alloy processing,

composition and microstructure on tensile, creep, fatigue crack growth (FCG) and low cycle fatigue (LCF) and thermo-mechanical fatigue (TMF) behaviour of Ti2AlNb based Ti-Al-Nb-Mo-Si alloys will be presented. Investigations revealed that Mo and Si both have beneficial influence on strength and creep resistance. Further, a two-step isothermal forging sequence, followed by an appropriate heat treatment schedule, yields an optimum microstructure having controlled distribution, morphology and volume fraction of primary α_2 , α_2 /O lath fineness and prior β grain size. Several microstructures presenting various combinations of tensile and creep properties were achieved. FCG and LCF resistance are influenced by microstructure, alloy ductility as well as test frequency and temperature. The TMF loading, especially out-of-phase cycle, appears to be more detrimental than isothermal LCF loading.

4:30 PM

Influence of Morphology on Mechanical Behavior of an Orthorhombic Ti-22Al-20Nb-7Ta Alloy: *Shiqiong Li*¹; *Yong Mao*²; *Jihua Pen*¹; *Jianwei Zhang*¹; *Dunxu Zou*¹; ¹Central Iron and Steel Research Institute, High Temp. Matls. Rsch. Div., Beijing 100081 China

A new type of Ti2AlNb base alloy, Ti-22Al-20Nb-7Ta (at.%), developed in CISRI exhibits good combinations of strength and ductility at room and elevated temperatures. The tensile properties were found to strongly depend on morphologies of a 2, O and B2 phases, which were controlled effectively by thermomechanical processing (TMP) performed. The microstructure containing a mixture of O and B2 phases with coarse and crisscross lath-like morphologies and the retained large prior β grain boundaries obtained by (a 2+B2) region rolling followed by β solution and (O+B2) aging results in poor elongation ductility and in lower strength. The microstructure containing a small volume fraction ($< 10\%$) of fine a 2 particles distributed in fine and crisscross lath-like O and B2 phases obtained by (a 2+O+B2) region rolling followed by (a 2+B2) solution and (O+B2) aging also results in poor elongation ductility and in a relatively lower strength. However, upon obtaining about 30% of volume fraction of fine and equiaxed prior a 2/O particles randomly distributed in extremely fine and crisscross lath-like (O+B2) structure produced by (a 2+O+B2) region rolling followed by (O+B2) solution and (O+B2) aging, improvements occur in elongation ductility without any sacrifice in strength. The yield strengths of such microstructure obtained are 1200 MPa and 970 MPa, and the elongation are 9.8 % and 14% at room temperature and 650°C respectively. Detailed fracture analysis included in Situ fracture behavior and metallographic examination of sections of the fracture specimens, shows that different fracture mechanisms operate for the different morphologies, and the critical microstructural unit for arresting fracture also varies, depending on the morphologies.

Microstructure and Properties I

Tuesday PM

Room: Cypress 2

July 18, 2000

Location: The Westin Bayshore Hotel

Session Chairs: Y. Mishima; G. Chen

1:30 PM Invited

Mechanisms of Creep Deformation in Titanium Aluminides: *G. B. Viswanathan*¹; *S. Karthikeyan*¹; *P. G. Gouma*¹; *Y. W. Kim*²; *M. J. Mills*¹; ¹The Ohio State University, Dept. of Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; ²UES, Inc., Dayton, OH 45432 USA

Alloys based upon γ -TiAl, with slightly sub-stoichiometric compositions, have attractive strength and low density for structural applications. This talk will focus on the mechanisms of creep deformation, and the sources of creep strengthening, in these alloys. The lamellar microstructure consists of fine laths of the intermetallic γ and α_2 phases. Creep deformation in the γ phase is dominated by twinning and by motion of $1/2\langle 110 \rangle$ type, ordinary dislocations, which are frequently jogged. A model for creep of the γ phase will be presented which is based on the premise that motion of ordinary dislocations is controlled by climb of these jogged segments. During creep of the two-phase lamellar structures, an additional process of importance is dissolution of the α_2 phase. General aspects of the evolution of the lamellar structure will be discussed, as will the strengthening effects of carbon and silicon additions, which lead to precipitation at lamellar boundaries. This work is supported by the GOALI Program of the National Science Foundation.

2:00 PM

Stability of Lamellar Microstructure Consisting of γ/γ Interfaces in Ti-48Al-8Nb Single Crystal at Elevated Temperatures: *Y. Yamamoto*¹; S. Y. Chang¹; M. Takeyama¹; T. Matsuo¹; ¹Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Based on our previous phase diagram study on Ti-Al-Nb ternary system, unlike the α_2 γ lamellar structure in binary Ti-48Al, Ti-48Al-8Nb can exhibit fully lamellar structure consisting of only γ plates with three types of γ/γ interfaces, i.e., perfect-twin, pseudo-twin and variant. In this study, change in the lamellar microstructure with aging in this alloy at 1473K has been examined using the single crystal (PST), in order to identify the role of γ/γ interfaces in thermal stability of lamellar microstructure. Macroscopically, the lamellar microstructure collapses rapidly and turns so equiaxed γ grain structure. The TEM analysis in the early stage of aging revealed that the variant interfaces are disappearing, leaving mainly the perfect-twin boundaries. The instability of the variant interfaces is due to high interfacial energy, and this is responsible for the collapse of the lamellar structure. The mechanism of the process to collapse will be discussed in conjunction with the case of α_2/γ lamellar structure in the binary alloy.

2:20 PM

Interfacial Dislocations and Deformation Twinning in Fully Lamellar TiAl: *L. M. Hsiung*¹; T. G. Nieh¹; B. W. Choi¹; ¹Lawrence Livermore National Laboratory, Chem. and Matls. Sci. Dept., L-369, P.O. Box 808, Livermore, CA 94551-9900 USA

This paper further reports the result of interfacial phenomena observed in fully lamellar (FL) TiAl. Deformation twinning, taken place abnormally within FL-TiAl subjected to creep at strain rates as low as 10^{-7} , is found to be intimately related to the motion, pile-up and dissociation of interfacial (Shockley partial) dislocations. Interfacial dislocation motion and pile-up are verified using in-situ TEM. Since Shockley partial dislocations are energetically infeasible to undergo cross-slip or climb, they can only move conservatively along the interfaces. Accordingly, the pile-up configuration once generated can remain in place even at elevated temperatures. The pile-up further leads to the emission of deformation twins from the interfaces into γ lamellae. $\{111\}\langle 112 \rangle$ - and $\{112\}\langle 111 \rangle$ -type twins, both generate $\Sigma 3$ boundaries in γ lamellae, have been identified. Based upon the pile-up configuration of interfacial dislocations, the critical stress for the “interface-activated” deformation twinning is calculated and discussed.

2:40 PM

Defect Structures in the Massive gm Phase in Quenched Ti-46.5Al Alloy: Ping Wang²; Mukul Kumar³; Vijay K. Vasudevan¹; ¹University of Cincinnati, Matls. Sci. and Eng. Dept., Cincinnati, OH 45221-0012 USA; ²Brown University, Div. of Eng., Box D, 182 Hope St., Provi-

dence, RI 02912 USA; ³Lawrence Livermore National Laboratory, 7000 East Ave., M/S L-370, Livermore, CA 94550 USA

The defect structures in the massively formed gamma (gm) grains in a Ti-46.5 at.% Al alloy, rapidly quenched from the high-temperature alpha-phase field, have been studied by conventional and high-resolution TEM. Defect structures composed of dislocations, stacking faults (SFs) and antiphase boundaries (APBs) intimately associated with dislocations or stacking faults were observed. Analysis indicates that both $1/2\langle 110 \rangle$ and $1/2\langle 101 \rangle$ unit dislocations were present in the gm phase, with the latter linked by highly curved APBs. Comparison of experimental and computer simulated TEM images established that wide SFs, which are created by the dissociations of $1/2\langle 101 \rangle$ unit dislocations, lie on $\{111\}$ planes and are bound by $b = 1/6\langle 121 \rangle$ Shockley partial dislocations of all possible types. In addition, APBs are found to commence or terminate on the SFs at the partial dislocations with $b = 1/6\langle 121 \rangle$, but not those with $b = 1/6\langle 112 \rangle$. Confirmation for both the latter, as well as the intrinsic nature of the stacking faults was obtained using atomic models and analysis of the HREM images. Based on the observations and subsequent analyses, a model for the formation of these defects and defect configurations is proposed.

3:00 PM

Transfer of Shears Across Polytwinned Interfaces in Intermetallic L10-Phases: *Jörg M.K. Wiezorek*¹; William A. Soffa¹; ¹University of Pittsburgh, Matls. Sci. and Eng. Dept., Sch. of Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Polytwinned (PT), lamellar microstructures can be readily established in alloys based on intermetallic phases with the ordered tetragonal L10-structure, e.g. TiAl and FePd. The PT-composition planes are common $\{111\}$ in TiAl and common $\{101\}$ in FePd. The mechanical properties of PT-grains are anisotropic with “hard” and “soft” orientations. The deformation modes active in these L10-phases involve slip of super- and ordinary dislocations, as well as typically significant amounts of ordered twinning. For “hard” oriented grains shear transfer across PT-interfaces must occur and appears to be of particular importance to the development of quantitative descriptions of the behavior of this type of compounds. Methods of optical, scanning and transmission electron microscopy have been used to investigate details of these transfer processes. Using crystallographic analyses based on the Thompson-tetrahedron adapted to the L10-structure possible mechanisms for shear transfer across the $\{111\}$ - and $\{101\}$ -conjugated PT-interfaces have been identified. These theoretically derived transfer mechanisms are compared to experimental observations in TiAl and FePd. Results of this study are related to the physical and mechanical behavior of PT-L10-intermetallics.

3:20 PM Break

3:40 PM

Anisotropic Mechanical Behavior of Textured γ -TiAl Caused by the Directionality of Twinning: *Wolfram Schillinger*¹; Bjoern Lorenzen¹; Arno Bartels¹; ¹TU Hamburg-Harburg, Matls. Sci. and Tech. Div., Eissendorferstr. 42, Hamburg 21073 Germany

After the rolling process of γ -TiAl a strong texture similar to the cube textures of fcc-metals with low stacking fault energy occurs. In addition the tetragonal c-axis tends to evade the flow direction during forming, i.e. the c-axis is aligned in transverse direction of the sheet. At 700°C compression tests were performed in transverse, rolling and normal direction as well as tensile tests in rolling and transverse direction. The density of deformation twins observed in optical microscopy and TEM-studies increases with decreasing yield stress and strain hardening. The highest density of deformation twins is observed after compression in transverse direction and the lowest density is observed after tensile tests in transverse direction. It is concluded that the directionality of

twinning in the ordered structure is the main cause for the mechanical anisotropy found in textured γ -TiAl.

4:00 PM

Influences of Molybdenum and Boron Additions to the Microstructure and Properties of Near- γ Alloys: *J. A. Christodoulou*¹; H. M. Flower²; ¹NSWC, Carderock Div., Code 681, 9500 MacArthur Blvd., West Bethesda, MD 20817-5700 USA; ²Imperial College of Science, Dept. of Matls., Tech. and Med., London SW7 2BP UK

Molybdenum and boron additions to near- γ titanium aluminides influence microstructure and mechanical properties in profound ways, and may extend use temperatures by 50°C or more. In an early step toward realizing these goals, microstructural developments and phase transformations during solidification and subsequent thermomechanical processing and heat treatment were carefully evaluated in two alloys: Ti-50Al-5Mo + 8vol% TiB₂ and Ti-44.5Al-8Mo + 11.5 vol% (Ti_{0.8}Mo_{0.2})B. The understanding developed enabled manipulation of the microstructures, and processing routes that yielded desired microstructures were then identified. Compression testing was conducted to provide preliminary indications of mechanical properties relative to the more mature near- γ alloys. The results of these efforts are introduced here.

4:20 PM

Effect of Al Content and Nb Addition on the Fault Energy and Strength of Ti-Rich TiAl Alloys: *Wei-Jun Zhang*¹; Fritz Appel²; ¹Philip Morris USA, RD&E Ctr., 4201 Commerce Rd., D202, Richmond, VA 23234 USA; ²GKSS-Research Centre Geesthacht GmbH, Institut. for Matls. Rsch., Geesthacht 21502 Germany

Variations in planar fault energies with composition can be expected to have an important influence on the ductility, strength and creep resistance of TiAl-base alloys. In this paper, the SISF energy in Ti-45Al, Ti-49Al, Ti-45Al-10Nb and Ti-49Al-10Nb (at.%) alloys was determined by weak-beam TEM analysis of $1/2\langle 112 \rangle$ superdislocation core configurations. It was found that the SISF energy greatly decreased (from 97 mJm⁻² to 64 mJm⁻²) as the Al concentration in gamma phase decreased from 49.6% to 48%. Nb addition reduced the SISF energy in gamma phase containing high Al content (49%) but had little effect at lower Al concentrations. Based on these results, the strengthening mechanism of Nb alloying in TiAl alloys was discussed.

4:40 PM

Precipitation Behavior and Hardening in Ag-Modified L12-Al3Ti and L10-TiAl: *Wen Huai Tian*¹; ¹University of Science and Technology Beijing, Dept. of Matls. Phys., No.30 Xue Yuan Rd., Haidian District, Beijing M4X 1G7 China

A detailed transmission electron microscopy (TEM) study has been made of the crystallographic characteristics of precipitation of L10-TiAl and D023-Al11Ti5 in Ag-modified Al3Ti with an L12-ordered structure as well as the precipitation of L12-Al3Ti in L10-TiAl matrix in Ti-Al-Ag ternary system. TEM observations revealed that plate-like L10-TiAl precipitates form a kinked plate and D023-Al11Ti5 precipitates form a multi-domain structures which can be considered as a twin related structures with the tetragonal axis of twin I parallel to [100] direction and that of twin II parallel to [010] direction of the matrix. The particular habit plane, orientation relationship and twin plane variants were carefully determined. It is found that these crystallographic and morphological features can be explained excellently by adopting the linear elastic theory developed primarily by Khachaturyan. The coherency stresses across the precipitate/matrix interface is considered to be the main factor controlling the precipitate morphology.

Physical and Metallurgical Properties I

Wednesday AM

Room: Salon 1

July 19, 2000

Location: The Westin Bayshore Hotel

Session Chair: M. H. Loretto; Toshiharu Noda

8:30 AM Invited

Overview of Research on Mo-Mo Silicide Composites: *M. G. Mendiratta*¹; T. A. Parthasarathy¹; D. M. Dimiduk²; ¹UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; ²Air Force Research Laboratory, Matls. and Manfact. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Alloys consisting of aMo in equilibrium with silicide/boride intermetallics, MoSi₃ and Mo₅SiB₂ are being investigated for high temperature structural applications. The intermetallic phases provide oxidation resistance at high temperatures (1000-1350°C) by the formation of a protective borosilicate glass scale which retards volatilization of the Mo phase, and together with aMo, provide a balance in the mechanical properties including creep resistance, strength and toughness. The research is focused on investigations of multicomponent phase equilibria, microstructural evolution, static and cyclic oxidation behavior and mechanical behavior. The results and operative mechanisms of oxidation behavior from 600-1350°C will be presented. Strength, and toughness (25-1400°C), and creep (1200-1300°C) are also being determined. The results will be discussed in comparison to the behavior of other potential high temperature material systems.

9:00 AM

Single Crystal Elastic Constants of Non-Stoichiometric Ordered Alloys: *Craig S. Hartley*¹; ¹U.S. Department of Energy, Div. of Matls. Sci., 19901 Germantown Rd., Germantown, MD 20874 USA

Elastic constants of single crystals are direct consequences of the nature and type of interatomic bonds in crystalline materials. Not only do they determine many properties of defects in crystals but also technologically important quantities such as the bulk modulus and Young's modulus of polycrystalline materials. These material properties thus represent one of the principal examples of modeling where the characteristics of atoms can be related directly to the properties of bulk material through appropriate averaging procedures. Experimental data on single crystal constants is available for many elemental materials and some alloys. Substantially fewer instances exist of experimental determinations of single crystal elastic constants of stoichiometric and non-stoichiometric ordered compounds. Where experimental studies are not available, first principles calculations of elastic constants of stoichiometric alloys are possible, although computationally intensive. However, this technique is difficult to apply to non-stoichiometric alloys. Lattice models of crystals, consisting of atoms held together by interatomic forces extending to a limited number of neighbor shells, have proved successful in representing elastic constants as linear combinations of interatomic force constants, defined in terms of appropriate derivatives of an interatomic pair potential. It has been shown that in disordered alloys, an effective interatomic pair potential can be constructed using the quasi-chemical model, from which the composition dependence of elastic constants can be determined in terms of parameters characterizing pair potentials for the species constituting the alloy. The pair potentials determined from disordered alloys can be used as a first approximation to construct effective potentials for ordered alloys

by taking account of the appropriate changes in pair probability and coordination number associated with the different structure and assuming a model for site occupancy of the excess component. Elastic constants can then be calculated from these effective potentials. Examples are presented for elastic constants of non-stoichiometric alloys based on both B2 and L12 structures calculated from effective potentials constructed from data on disordered binary alloys. The approach provides a technique for calculating elastic constants of single crystals of intermetallic compounds at temperatures and compositions where experimental determination is difficult or infeasible.

9:20 AM

Thermal Expansion Of Ti5Si3 With Ge, B, N Or O Additions: J.J. Williams¹; M.J. Kramer¹; M. Akinc¹; ¹ Iowa State University; Ames Lab. and Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA

The crystallographic thermal expansion coefficients of Ti5Si3 from 20? to 1000?C as a function of B, C, N or Ge content were measured by high temperature x-ray diffraction using synchrotron sources at Cornell University (CHESS) and Argonne National Laboratory (APS). Whereas the ratio of the thermal expansion coefficients along the c- and a-axes was approximately three for pure Ti5Si3, this ratio decreased to about two when B, C, or N atoms were added. Additions of O and Ge were less efficient at reducing this thermal expansion anisotropy. The extent by which the thermal expansion is changed when B, C, N, or O atoms are added to Ti5Si3 correlates with their expected effect on bonding in Ti5Si3.

9:40 AM

Theoretical Study of Substitutional and Interstitial Defects on the Elastic Constants of Intermetallics: Peter C. Schmidt¹; Bodo Mayer¹; Juergen Sticht¹; Michael Methfessel²; John Harris³; ¹University of Technology, Phys. Chem. Dept., Petersenstr. 20, Darmstadt D-64287 Germany; ²Institut fuer halbleiterphysik, Frankfurt/Oder Germany; ³Institut fuer Festkoerperforschung, Forschungszentrum Juelich, Juelich, Germany

We have studied all components of the elastic strain tensor of binary C14/C15 Laves phases AAl₂, A = Ca, Sc, Y, La; ACr₂ with A=Ti,Zr; ternary Laves phases NbNi_xAl_{1-x} and metal-hydrogen systems NbH_x, PdH_x and HfCr₂H_x using a new variant (NFP) of the linear muffin-tin orbital density functional method. The method treats the full crystal potential and allows the computation of forces and therefore of the local relaxation of the atoms as the unit cell is deformed. With the aid of mean-values due to Hill, bulk moduli, Young's moduli, Poisson's ratios and sound velocities are calculated for polycrystalline samples. Using Debye-Grüneisen theory, the Debye temperatures, specific heats and linear thermal expansion coefficients were also estimated. Where experimental data are available for these quantities, theoretical and experimental values are generally in reasonable agreement. The correlation between the trends in the elastic constants and the chemical bond is discussed.

10:00 AM

Oxidation Behavior of Functionally Graded MoSi2-Si3N4 Composites: K. Natesan¹; S. C. Deevi²; ¹Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; ²Philip Morris USA, P.O. Box 26583, 4201 Commerce Rd., Richmond, VA 23112 USA

High temperature structural materials are critically needed for improving the thermal efficiency and reliability of energy conversion systems and advanced engine systems. Molybdenum silicides and their composites are particularly attractive among the potential candidates due to their high thermal and electrical conductivities, mechanical strength, and reasonable oxidation resistance. Approach based on functionally graded compositions provides an unique method for designing

composite materials tailored from the standpoint of the properties of individual components such as MoSi2 and Si3N4. In this paper, we focus our attention on the oxidation behavior of composite materials from the standpoint of pesting reaction at low temperatures and protective oxidation at high temperatures. Results are presented on oxidation studies conducted in air at 500 to 1400°C for exposures up to 2000 hours. We discuss the oxidation rates of composites, stability of the oxide films, role of sintering additives, nanohardness, and modulus of the oxide films at room temperature, 600°C, and at 1400°C. We compare the oxidation rates of the composites with those of MoSi2 and B-containing Mo5Si3 alloys. *Work supported by the U.S. Department of Energy, Office of Fossil Energy, Advanced Research and Special Technologies Materials Program, under Contract W 31 109 Eng 38.

10:20 AM Break

10:40 AM

Long Term Static and Cyclic Oxidation of Ti-44Al and Ti-44Al-xNb Alloys: S. K. Varma¹; R. N. Mahapatra²; Alan Chan¹; E. Corral¹; ¹The University of Texas at El Paso, Dept. of Metall. and Matls. Eng., El Paso, TX 79968 USA; ²Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20670 USA

Binary Ti-44Al and ternary Ti-44Al-xNb alloys have been subjected to oxidation in air for periods of up to one week in a range of temperature from 800 to 1000°C. Both static and cyclic (one cycle consists of 55 minutes of heating and 5 minutes of cooling in air) oxidation behavior has been monitored through the weight gain measurements. Oxidation curves at different temperatures have been obtained. Binary alloy shows a much greater extent of oxidation than ternary alloys. Enhanced oxidation resistance by the addition of Nb is well exhibited by the oxidation curves. Addition of 4 and 8 atomic percent Nb shows different oxidation characteristics compared with alloys containing 11 and 12% Nb as revealed by SEM and EDX analysis. TEM of oxidized samples indicate the features that are also different in these two classes of ternary alloys. An overview of the experimental results will be presented with emphasis on the effect of Nb on the oxidation behavior of these alloys.

11:00 AM

High Temperature Oxidation Behavior of Ti3Al-Nb Alloys Prepared PDS: Wei Fang¹; Se-hyun Ko¹; Hitoshi Hashimoto¹; Toshihiko Abe¹; Yong-ho Park¹; ¹Tohoku National Industrial Research Institute, Matls. Sys. Sect., 4-2-1 Nigatake Miyagino-ku, Sendai, Miyagi 983-8551 Japan

Titanium aluminides have received considerable attentions as the basis for new high temperature structural materials. Several compositions based on Ti3Al-Nb have been developed to achieve specific strength and stress rupture properties which exceed those of Ni base alloys such as INCO 718 over temperature range 823-973K. Recently, pulse discharge sintering (PDS) process technique was successfully developed to manufacture net shape TiAl valves. This research was aimed at investigating the high temperature oxidation of Ti3Al-Nb alloys prepared by PDS. The oxidation rates were measured from 1073-1273K in air. The kinetics and activation energy for the isothermal oxidation of Ti3Al-Nb alloys revealed that Ti3Al added Nb tended to form finer oxide grain and denser oxide layer and that oxidation rate could be clearly reduced by increasing Nb content (up to 13 at% in the study). The parabolic rate law was adequate to describe the high temperature oxidation behavior for Ti3Al-Nb alloys in air.

11:20 AM

Improvement of the High-Temperature Oxidation Resistance of Ti-50Al by Anodic Coating in the Phosphoric Acid: Mu-Rong Yang¹; Shyi-Kaan Wu¹; ¹National Taiwan University, Instit. of Matls. Sci. and Eng., 1 Roosevelt Rd. Sec. 4, Taipei, Taiwan 106

The high temperature cyclic oxidation resistance of Ti-50Al can be improved by the anodic coatings in phosphoric acid. This current density of anodization of TiAl in phosphoric acid aqueous solution (4wt% H_3PO_4) at 18°C decreases monotonically during the course of anodization. The spark will occur sporadically on the TiAl as the voltage is over 300V and the instantaneous current density after 45 min of anodization increases with increasing voltage. The anodic films are amorphous and contain substantial amount of phosphorus. Cyclic oxidation test results indicate that anodization in 4wt% H_3PO_4 at 18°C for 45 min can remarkably reduce the oxidation at 800°C and the improvement increases with increasing the anodizing voltage up to 400V, at which the parabolic rate constant can be reduced to 1/600 of that for as-homogenized TiAl. From the Raman spectrum analysis, we can attribute the improvement to the retardation of the formation kinetics of titanium oxide.

11:40 AM

Microstructure, Wear and High-Temperature Oxidation Resistance of Nitridized TiAl Based Alloys: Jian Sun¹; Jian Shen Wu¹; ¹Shanghai Jiao-tong University, Schl. of Matl. Sci. and Eng., Shanghai 200030 PRC

TiAl based alloys are considered to be used in various high-temperature structural components. Therefore, the wear and high-temperature oxidation resistance are very important, in addition to the ductility of the alloys. In this work, the nitridation process of a Ti-47Al-2Nb-2Cr-0.2Si alloy fabricated by cast and subsequent heat-treatment to refine the microstructures has been investigated in purified ammonia at temperatures from 900°C to 1100°C. The scales and sub-scales of the nitridized alloy were characterized by x-ray diffraction, SEM and EPMA, which showed that TiN and AlN were detected on the surface of the alloys after nitridation. The wear and high-temperature oxidation resistance were studied both for the nitridized alloy and for the untreated alloy. It has been concluded that nitridation can markedly improve the wear and high-temperature oxidation resistance of the TiAl based alloy. By means of the SEM observations and EPMA measurements of the tested alloys, the beneficial effect of the nitridation on wear and high temperature oxidation resistance of the TiAl based alloy will be discussed.

12:00 PM

High Temperature Oxidation of Mechanically Alloyed NiAl-Fe-AlN-Al₂O₃: Dong-Bok Lee¹; Gi-Young Kim¹; Soon-Chul Ur²; Sang-Whan Park³; Soon-Chul Ur²; ¹Sungkyunkwan University, Sch. of Metall. and Matls. Eng., Chunchundong 300, Changanku, Suwon 440-746 South Korea; ²Chungju National University, Dept. of Mats. Eng., 123 Geomdanri, Iryumyon, Chungju, Chungbuk 380-702 South Korea; ³KIST, Cheongryang, Seoul 136-791 South Korea

A new NiAl-Fe alloy containing uniformly distributed ultra-fine AlN and Al₂O₃ dispersoids was produced by mechanical alloying, and its oxidation behavior was studied over the range of 1073-1473K in air. The oxidation rates, the oxide scales formed and the oxidation mechanism were discussed with an emphasis on the effect of dispersoids.

12:20 PM

The Effect of Microalloying Y on the Microstructure and Oxidation Resistance of Nb₅Si₃ Intermetallics: Y. F. Han¹; S. Y. Qu¹; R. M. Wang¹; ¹Institute of Aeronautical Materials, Beijing 100095 PRC

Nb₅Si₃ Intermetallics have the high melting point (2753K) and low density (7.2g/cm³). Recent studies have shown that the two-phase structure of Nb/Nb₅Si₃ exhibits excellent thermochemical stability and resistance to coarsening up to 1500°C. In addition, these Nb/Nb₅Si₃ composites exhibit a good balance in mechanical properties, having a reasonably strength up to 1500°C and fracture toughness at room temperature. These fairly good comprehensive properties of the composites are attractive for developing high-temperature structural materials. However, one major barrier to the use of Nb₅Si₃ Intermetallics for high-

temperature application is their catastrophic oxidation behavior. Therefore, the effect of microalloying on the oxidation resistance of Nb/Nb₅Si₃ composites have been carried out in this investigation. Two basic compositions of Nb-10at.%Si and Nb-18.7at.%Si and microalloying elements of Y have been used in the present study. The materials were prepared by arc-melting Nb and Si elements in the form of about 100g buttons, then heat-treated in vacuum at the temperature range of 1300-1600°C for 24-100 hours. The temperature and time were chosen to ensure that the microstructure is consisted of equilibrium Nb+Nb₅Si₃ phases. The microstructure was characterized using metallography, scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM). The oxidation resistance tests were carried out in air at 1200°C up to 100 hours. The experimental results showed that the oxidation resistance of Nb/Nb₅Si₃ composites was improved by microalloying Y. The effect of microalloying Y on the mechanical properties has also been studied.

Creep I

Wednesday AM

July 19, 2000

Room: Salon 3

Location: The Westin Bayshore Hotel

Session Chair: R. E. Smallman; J. N. Wang

8:30 AM Invited

Role of Lamellar Plates in Creep of TiAl Alloy with Fully Lamellar Structure: T. Matsuo¹; T. Nozaki¹; T. Asai¹; M. Takeyama¹; ¹Tokyo Institute of Technology, Dept. of Metallu. and Cer. Sci., Meguro-ku, Tokyo 152-8552 Japan

Nearly γ Ti-48at%Al alloy with fully transformed lamellar structure (designated as FL) is believed to have the superiority in creep and in rupture ductility. To investigate the role of the lamellar plate, the difference in creep between the FL specimen and the γ single phase specimen was elucidated, conducting the creep tests at 850°C under the low stress of 68.6 MPa. The superiority of FL specimen is derived not from decreasing creep rate, but from suppressing the onset of accelerating creep. The suppression of accelerating creep is interpreted by suppressing the appearance of dynamic recrystallization along the grain boundary. The α_2 lamellar plate has larger effect to suppress the dynamic recrystallization than the γ lamellar plate. According to the above suppression, the dynamic recrystallization would be fully suppressed in the Ti-Al PST crystal, because of removing the grain boundary. By conducting the creep tests of PST crystals with different lamellar orientations to stress axis, the role of α_2 plate on creep rate is elucidated.

9:00 AM Invited

Creep Deformation in Two-Phase Titanium Aluminide Alloys: Fritz Appel¹; ¹GKSS-Research Centre Geesthacht GmbH, Instit. for Matls. Rsch., Geesthacht 21502 Germany

Insufficient creep resistance and structural stability are critical issues for high temperature applications of titanium aluminides. Despite the fact that there exists a large amount of creep data the present understanding is still limited and a matter of controversy. Conventional and high resolution electron microscope observations have been performed to sort out the mechanism limiting the creep strength under various experimental conditions. The major areas of investigation involve: creep processes at low and modest stresses which are close to the intended design requirements; dislocation processes contributing to primary creep; phase stability and transformations diffusion assisted dislocation multiplication; contribution of twinning; degradation of strength properties due to

long-term creep. Particular emphasis will be placed on metallurgical techniques for improving the creep resistance of gamma-base alloys. These involve stabilization of the microstructure by thermal treatments and the implementation of solid solution and precipitation hardening.

9:30 AM

Effect of Grain Size on the Minimum Creep-Rate of Ti-50mol%Al Intermetallic Alloy: Norihiko Hamada²; Jun-ichi Koike³; Hiroshi Oikawa¹; ¹College of Industrial Technology, Amagasaki 661-0047 Japan; ²Aichi Steel Works Limited, Rsch. and Dev. Lab., Tokai 476-0003 Japan; ³Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Sendai 980-8579 Japan

Creep characteristics have been studied on a stoichiometric γ -phase material of stable microstructures having 56 and 100 μm in grain size. Compressive creep tests were performed at 1100K under 100 and 126MPa. Under these conditions (in region III) a stagnation stage appears on the creep curve after small initial deceleration stage, and then, an acceleration stage appears to reach the (near) steady-state stage. The minimum creep-rate (during the stagnation period) decreases with increasing the grain size. The grain-size exponent for the minimum creep-rate is about -2.5 in region III. The steady-state creep rate depends little on the grain size. In conjunction with the reported results for regions of higher stresses (Takashi and Oikawa, 1991), the influence of stress level on the effect of (initial) grain size on the minimum creep-rate of near stoichiometric γ -phase intermetallics has been discussed in some details.

9:50 AM

Effect of Microstructure on Creep Behavior of Ti-48Al-2W Intermetallic Compounds: Soon Hyung Hong¹; ¹Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., 373-1 Kusung-dong, Yuseong-gu, Taejon 305-701 Korea

The creep behaviors of Ti-48Al-2W intermetallic compounds have been investigated by constant stress creep tests at temperature of 750-850°C under 170-440MPa. The effects of grain size and volume fraction of lamellar phase on creep behavior of Ti-48Al-2W intermetallic compounds were analyzed. The minimum creep rates were almost independent on lamellar grain size in the range of 135-400 μm , while were sensitively dependent on the lamellar volume fraction. The minimum creep rate decreased with increasing the lamellar volume fraction in the range of 70-99%. The activation energies were measured as 298kJ/mol for the near γ structure, 340kJ/mol for the near lamellar structure and 345kJ/mol for the fully lamellar structure. The stress exponents were measured as 6.4 in near γ structure, 4.9 in fully lamellar structure and 5.0-5.5 in near lamellar structure having the lamellar volume fraction ranged 70-95%. The microstructures of Ti-48Al-2W intermetallic compounds after creep deformation were analyzed. The creep rates of near lamellar structure could be analyzed by the rule-of-mixtures from creep rates of lamellar phase and single γ phase.

10:10 AM Break

10:30 AM

Effect of Stress Axis on the Creep Deformation Behavior of Ti-48Al Polysynthetically Twinned (PST) Crystals: Hee Y. Kim¹; Gerhard Wegmann²; Kouichi Maruyama¹; ¹Tohoku University, Dept. of Matl. Sci., Aobayama 02, Sendai 980-8579 Japan; ²GKSS Forschungszentrum, Inst. for Matls. Res., Max-Planck-Strasse, Geesthacht D-21502 Germany

The creep deformation behavior of PST crystals Ti-48Al was investigated at 1150K under 100-400MPa. Two hard orientations with the lamellar plates parallel or perpendicular to the compression axis and a soft orientation with the lamellar plates oriented 35° to compression axis were compared. The soft PST orientation exhibited the highest creep rates and larger primary creep strain than those of the hard PST orientations. The stress exponents were evaluated as 7.0 in the hard

PST orientations. In the soft PST orientations, the stress exponent increased with decreasing stress from 4 at high stress regime to 8 at low stress regime. The primary creep strain increased with decreasing stress. Ordinary dislocations with Burgers vector $\frac{1}{2}[1-10]$ and $\frac{1}{2}[110]$ were found to be the principal deformation mode. The rotation of lamellar and interface sliding played important role to accommodate the creep deformation in the soft PST orientation.

10:50 AM

Microstructure and Creep Strength of Fully-Lamellar TiAl Alloys Containing Beta-Phase: Sadao Nishikiori¹; ¹Ishikawajima-Harima Heavy Industries Company Limited, Matl. Tech. Dept. Rsch. Inst., 3-1-15 Toyosu, Koutou-ku, Tokyo 135-8732 Japan

Effect of microstructure on creep strength in TiAl-Fe-V-B alloys with fully transformed lamellar structure has been investigated using the Larson-Miller parameter. Since these alloys are characterized by addition of iron and vanadium to improve castability, the fine grains of the beta phase surrounding gamma grains is present in as-cast materials. This work has focused on the optimization of metallurgical features to improve creep strength though understanding of the beta phase. Fully lamellar TiAl alloy with the beta phase surrounding lamellar colonies showed poor creep strength. Iron works as strong beta stabilizers and causes a peak in amount of the beta phase at approximately 1523K. In order to improve creep strength, an effectual heat treatment process was investigated to disappear the beta phase. On the basis of these experimental results, further alloy development was also discussed. Fully-lamellar TiAl-Fe-V alloys without the beta phase which lead to a creep rupture life of over 3600ks at 1033K and 220MPa has been developed. Moreover, fully-lamellar TiAl in another alloy system shows a creep rupture life of well over 2160ks at 1073K and 220MPa.

11:10 AM

The Influence of Aging on the Microstructure and Creep Behavior of Heat Treated PM γ -TiAl+W Alloys: Dong-Yi Seo¹; Jonathan Beddoes¹; Linruo Zhao²; ¹Carleton University, Dept. of Aero. and Mech. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada; ²National Research Council of Canada, Structures, Matls. and Propul. Lab., Insti. for Aero. Res., Ottawa, Ontario K1A 0R6 Canada

Titanium aluminide powder of nominal composition Ti-48at%Al-2at%W was consolidated by hot isostatic pressing at 1250°C and 200 MPa for 2 hours. A solution heat treatment involving a stepped cool was developed and applied to provide a favorable microstructure with fine lamellar spacing and free of Widmanstatten or massively transformed γ . Subsequently samples were aged for up to 192 hours at 950°C resulting in β_0 formation along lamellar interfaces. Creep tests were conducted at 760 °C and 276 MPa. Aged materials showed lower primary creep strain and less tertiary creep than unaged materials. Heat treated microstructures were analyzed, as well samples obtained from the grip and gage section of crept samples after different level of creep strains. The microstructural features such as lamellar and grain boundary morphology, and β_0 precipitates are characterized. Dislocation activities that influence the creep behavior of these alloys are investigated and the creep deformation mechanisms at different creep stages are elucidated. The results indicate that the lamellar microstructure of the aged material having β_0 precipitates is stable during creep deformation, but β_0 precipitates along grain boundaries are not beneficial to tertiary creep resistance.

11:30 AM

Different Tensile and Compressive Creep Properties in a Ti-44Al-1Mn-2.5Nb-0.15Gd Alloy: Xiaolin Wu¹; Kenong Xia¹; ¹University of Melbourne, Dept. of Mech. and Manufact. Eng., Victoria 3010 Australia

Creep behaviour of a Ti-44Al-1Mn-2.5Nb-0.15Gd alloy was investigated in both tension and compression. The alloy was cast and heat treated to have a fully lamellar microstructure with an average grain size of 265 μm . Creep tests were conducted in vacuum at a constant temperature between 750 and 900°C and under a constant stress ranging from 20 to 400 MPa. Although the normal three stage creep was observed in both tension and compression, the secondary creep rate in tension was significantly higher than that in compression. The secondary creep strain was higher in tension than in compression. However, the creep time in the secondary stage was longer in compression owing to slower creep rates. The stress exponent values were mostly between 5 and 7 although a lower one of about 3 was recorded in tension at 900°C. Possible reasons for such differences were discussed.

11:50 AM

Microstructure in Ti-48at%Al PST Crystal Subjected to Creep Deformation: *Tetsuya Asai*¹; *Seiki Hirata*¹; *Masao Takeyama*¹; *Takashi Matsuo*¹; ¹Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., Meguro-ku, Tokyo 152-8552 Japan

Tensile creep tests of the Ti-48at%Al PST crystals with different lamellar orientation angles with respect to the stress axis ϕ were conducted at 1148 K-68.6 MPa and the ϕ dependence of creep was investigated. Creep of PST crystal strongly depends on ϕ . The minimum creep rate for the PST crystal with $\phi = 63^\circ$ is three orders of magnitude larger than that for the PST crystal with $\phi = 3^\circ$, and the rupture life for the PST crystal with $\phi = 63^\circ$ is about one tenth of that for the PST crystal with $\phi = 3^\circ$. The marked differences in creep between the PST crystal with $\phi = 63^\circ$ and that with $\phi = 3^\circ$ would be ascribed to the role of α_2 plate to the operative slip systems. Based on the supposition, creep interruption tests were conducted for the PST crystals with $\phi = 63^\circ$. The microstructure of the creep interrupted specimens gives a few evidences to understand the role of α_2 plate to creep property.

Phase Transformation

Wednesday AM Room: Cypress 1
July 19, 2000 Location: The Westin Bayshore Hotel

Session Chairs: S. C. Deevi; V. K. Vasudevan

8:30 AM

Morphological Changes of Ti_3Al_5 Phase Formed by Phase-Decomposition of TiAl Intermetallics: *Minoru Doi*¹; *Toshiyuki Koyama*¹; *Takeshi Taniguchi*¹; *Shizuo Naito*²; ¹Nagoya Institute of Technology, Dept. of Matls. Sci. and Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan; ²Gifu Shotoku Gakuen University, Faculty of Economics and Information, Nakauzura, Gifu 500-8288 Japan

When TiAl intermetallics containing Al of 56-60 at.% are aged at 973 K, TiAl phase decomposes into the two-phase state of Ti_3Al_5 and TiAl. Morphological changes of Ti_3Al_5 precipitates in TiAl matrix observed with TEM are as follows: 1) in Ti-56Al having lower volume fraction, at first Ti_3Al_5 precipitates are thin plates having (001) faces, but then they coalesce with each other to form large rafts each of which has uneven faces consisting of different planes tilted from (001); 2) in Ti-58Al and Ti-60Al having higher volume fraction, at first the tweed structure appears and then the coalescences of Ti_3Al_5 precipitates take place, which results in the formation of large rafts similar to the case 1). The computer simulations based on Cahn-Hilliard non-linear diffusion equation can reproduce the above morphological changes.

8:50 AM

Investigation of $003_2 \gamma$ Phase Transformation Mechanism Under the Direction of Dislocation with Lamellar Interface in Primary Creep: *Soo Woo Nam*¹; *Seung Jin Yung*; ¹Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. and Eng., 373-1 Kusong-dong Yusong gu, Taejeon 305-701 Korea

Under creep condition, the micro-structural behavior of lamellar TiAl alloy is different from that of general materials. At as-received state, the amount of initial dislocations is very high and intrinsic dislocations, $b = \langle 110 \rangle$, are exist in lamellar interface and interface morphology is irregular. In early primary stage, the amount of dislocations density in α matrix is rapidly decreased. These dislocations are gliding on lamellar interface during primary creep. When they reach interface ledge, atomistic deformation caused by gliding dislocations can change the stacking sequence and the composition of α_2 -ledge. As a result of this process, α_2 is transformed to α phase. During this phase transformation process, a new unstable phase, Ti_2Al , is observed. This new phase is intermediate and disordered phase between α_2 and α ordered phases. The atomistic model for α_2 - Ti_2Al - α phase transformation process is newly proposed.

9:10 AM

Deformation-Induced Phase Transformations and Microstructural Degradation of Two-Phase Gamma Titanium Aluminide Alloys: *Michael Oehring*¹; *Fritz Appel*¹; ¹GKSS-Research Centre Geesthacht GmbH, Institut. for Matls. Rsch., Max-Planck-Str., Geesthacht 21502 Germany

To date, the high-temperature-strength of two-phase gamma titanium aluminide alloys is not sufficient for some of the intended engineering applications. In this paper an electron microscopy study of deformation-induced microstructural changes of the material is reported. The analyses were performed on specimens with lamellar microstructures since alloys of this microstructural condition exhibit superior creep resistance. The observed processes in the microstructure involve the formation of ledges at lamellar interfaces, the dissolution and spheroidization of alpha-2 lamellae and dynamical recrystallization. These processes were found to be closely related to the glide and climb of interfacial dislocations. Their driving forces probably arise from non-equilibrium phase compositions and coherency stresses and the applied external stress. Similar processes as observed at lamellar interfaces during the destruction of the lamellar microstructure might also occur when this microstructural condition is formed on cooling from the alpha field, and this relation will be discussed in the paper.

9:00 AM

Effect of Cooling Rate and Manganese Concentration on Phase Transformation in Ti-45 at%.Al Based Alloys: *Uttara Prasad*¹; *Qiang Xu*²; *Mahesh C. Chaturvedi*¹; ¹University of Manitoba, Dept. of Mech. and Indust. Eng., Winnipeg, MB R3T 5V6 Canada; ²Industrial Technology Centre, 1329 Niakwa Rd. East, Winnipeg, MB R2J 3T4 Canada

The present work is an attempt to study microstructural evolution in various T-45Al based alloys during varying heat treatment conditions. Small samples of the alloys were annealed in a-phase field (1350°C) for 0.5 hours and subsequently cooled at different cooling rates. Detailed microstructural investigation of the heat-treated samples suggested that the alloy's susceptibility to massive transformation increases with increasing the severity of cooling. However, this transformation is quenched by a@a2 disorder-order transformation at very high cooling rates (water quenching). It has also been observed that cooling rate dependence of transformation modes depends on the composition of the alloy. It has been found that higher the amount of Mn in the alloy, higher is the cooling rate required for complete massive transformation. Based on the results obtained so far, schematic CCT diagrams will be pro-

posed to demonstrate the effect of alloying elements on phase transformations of titanium aluminides.

9:50 AM

Prediction of Omega Phase Formation in Ti-Based Intermetallic

Alloys: *Guosheng Shao*¹; Panayiotis Tsakiroopoulos¹; ¹University of Surrey, Sch. of Mech. and Matls. Eng., Guildford, Surrey GU2 5XH UK

The omega phase is detrimental to the mechanical properties of Ti-based alloys, when a precursor high temperature bcc type phase (beta phase: A2 or B2 structure) is involved. This is particularly important for the design of the new-generation TiAl-based alloys, where the addition of beta-stabilising transition metal elements have been considered for improving the low temperature ductility. The interaction between Al and transition metals in these alloys could lead to the stabilisation of the beta phase via chemical ordering and the formation of ordered omega structures in the ordered parent beta phase. In this paper general rules are proposed for the prediction of omega phase formation in Ti-based intermetallic alloys. The omega phase stability with reference to the parent beta phase will also be discussed, on the basis of new experimental and theoretical evidence.

10:10 AM

Phase Transformations and Ordering from β -Ti to α_2 -Ti₃Al in Ti-Al Binary System:

*A. Suzuki*¹; *M. Takeyama*¹; *T. Matsuo*¹; ¹Toyko Institute of Technology, Dept. of Metall. and Cer. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Phase transformations along the pathway of β -Ti(A2) α_2 -Ti₃Al (DO₁₉) in a Ti-35 at.% Al alloy have been examined by means of direct quenching/aging from the high-temperature β single phase region to 1473-1373 K, and the TTT diagram of this pathway were made based on microstructural observations. All specimens exhibit massive and/or acicular martensite structures together with very fine anti-phase domains indicating that the $\beta\alpha$ structural change by either massive or martensite transformations takes place first, followed by $\alpha\alpha_2$ ordering. The start and finish lines of massive transformation show C curves and their noses exist at very short time less than a second around 1373K. The martensite transformation starts to occur at round 1273K. The ordering temperature is identified to become C curve as well. The results suggest that the massive transformation is diffusion controlled but competitive with martensite transformation, depending on the degree of supercooling. Role of alloying elements in microstructure control for some ternary Ti-Al-M systems will be discussed based on this diagram.

10:30 AM Break

10:50 AM

The Beta to Alpha Phase Transformation in Ti-(25-35)Al Alloys:

*Uwe Pilchowski*¹; *Veer Dhandapani*²; *Vijay K. Vasudevan*³; ¹Daimler-Chrysler, Airbus, Bremen, Germany; ²VLSI Technology, 9651 Westover Hills Blvd., San Antonio, TX 78251 USA; ³University of Cincinnati, Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA

The kinetics of the $\beta\alpha$ phase transformation and microstructure evolution during continuous cooling in Ti-25Al and Ti-35Al alloys was studied using a computer-controlled temperature and electrical resistivity measurement system. Samples of the alloys were heated to the β region in the device, as well as separately in a furnace, and cooled at various rates either by controlling the flow of a helium jet quench in the former or the quenching media (furnace, air, oil, water) in the latter. Using the in situ resistivity and thermal arrest data, the start and finish temperatures of the transformation were determined as a function of cooling rate and correlated with post-mortem light and transmission electron microscopy observations to establish the continuous cooling transformation diagrams. By coupling the data with physical models the enthalpies/driving forces associated with the transformation were deter-

mined. A transition in reaction mode from diffusional to diffusionless was observed with an increase in cooling rate, this change being accompanied by a change in the α (α_2) morphology from Widmanstätten plates to massive-like to martensitic structures at low, intermediate and high cooling rates. TEM analysis revealed that the martensite substructure was composed of twinned primary plates with microtwins within them. The twin system in both was determined to be $(\sim 2201)[1\sim 104]$, which has not been observed before in titanium martensites. These results are presented and discussed.

11:10 AM

Nucleation of Embryos of a Second Phase by Individual Impurity

Atoms: *Matthew O. Zacate*¹; *Gary S. Collins*¹; *Luke S.-J. Peng*¹; ¹Washington State University, Phys. Dept., Webster Bldg., Pullman, WA 99164-2814 USA

Perturbed angular correlation was used to monitor atomic environments of indium atoms in Ni-Al alloys at temperatures and compositions in the two-phase region between Ni₂Al₃ and NiAl. In previous room temperature measurements, the probability for observing indium atoms in the Ni₂Al₃ phase greatly exceeded lever-rule predictions, hinting at embryo formation; however, segregation of indium to the Ni₂Al₃ phase could not be ruled out. In measurements now made at high temperature, signals from Ni₂Al₃ phase have been observed over a range extending into the NiAl phase 1 at.% beyond the phase boundary published by Okamoto. Assuming the boundary is accurate, the observations demonstrate nucleation of small crystals of a second phase by individual solute atoms.

11:30 AM

Effect of Heat Treatment on Martensitic Transformation in Ni₂MnGa Alloys:

*Hideki Hosoda*¹; *Tsuyoshi Sugimoto*¹; *Shuichi Miyazaki*¹; ¹University of Tsukuba, Instit. of Matls. Sci., Tennodai 1-1-1, Tsukuba, Ibaraki 305-8573 Japan

It is well known that a large amount of frozen-in vacancies easily remain in bcc-base intermetallics due to low formation energy and high migration energy of vacancies. Besides, the degree of long-range order is also widely changed by A2-B2 and B2-L₂₁ order-disorder phase-transformations. In this work, the effects of vacancies and the degree of order on martensitic transformation are investigated in Ni₂MnGa alloys, where vacancy concentration and degree of order are controlled by changing heat treatment condition. Alloys were fabricated in a wide compositional range by Ar arc-melting method. Homogenization was carried out at 1273K for 24hrs, followed by 673K for 168hrs to remove excess vacancies. Then, alloys were heat-treated between 773K and 1173K for 24hrs. Based on XRD analysis, DSC measurements and Vickers hardness measurements, it is revealed that the phase transformation, magnetic transformation and hardness are influenced by the heat treatment.

11:50 AM

The Observation of Non-Equilibrium Transformations and Metastable Structures in Ternary Titanium Aluminide:

*A. Tokar*¹; *A. Katsman*¹; *A. Berner*¹; *L. A. Levin*¹; ¹Technion-Israel Institute of Technology, Dept. of Matls. Eng., Technion-City, Haifa 32000 Israel

Titanium aluminides are promising materials for aerospace applications. Their usage is restricted by low ductility at ambient temperatures. Microstructural optimization by thermo-mechanical treatment and alloying is employed to improve materials' performance. Iron is a potential candidate for property improvement due to the presence of several ternary and quasi-binary cubic phases in the Al-Ti-Fe system. Available data on phase formation in this system is limited by several cross-sections of equilibrium ternary phase diagram. The knowledge on phase formation under non-equilibrium conditions, which is essential for alloys' development, is lacking. In the present work, non-equilibrium transformations and formation of metastable structures in the iron-al-

loyed titanium aluminide was investigated. The observed microstructures were characterized by X-ray diffraction, scanning and transmission electron microscopy, electron probe microanalysis. The particular emphasis was made on formation of ternary Al_2FeTi phase (t_2 , $\text{Mn}_{23}\text{Th}_6$ type) and its metastable modifications as well as on formation and decomposition of quasi-binary FeTi(Al) phase (CsCl type). The influence of high temperature phase content and cooling conditions on structural state of the examined alloy at ambient temperatures was analyzed and discussed.

12:10 PM

Phase Formation in Iron Containing Titanium Aluminides during a Two-Step Heat Treating: A. Tokar¹; A. Katsman¹; A. Berner¹; L. Levin¹; ¹Technion-Israel Institute of Technology, Dept. of Matls. Eng., Technion-City, Haifa 32000 Israel

Non-equilibrium transformations and formation of metastable structures in an iron containing titanium aluminide were investigated. A particular emphasis was made on decomposition of the quasi-binary high-temperature FeTi(Al) -phase (CsCl-type) during two-step heat treatments. The FeTi(Al) -phase quenched from 1300°C to intermediate temperatures (700°C, 800°C, 900°C and 1100°C) becomes unstable. It was found that this phase decomposes by two competitive mechanisms: a) into a mixture of the known stable α_2 and t_2 phases, b) into iron enriched FeTi(Al) phase, and γ . At 800°C only the first transformation was observed, allowing for a fine mixture of α_2 and t_2 phases. At all the other temperatures products of both transformations were found. The iron enrichment in FeTi(Al) is limited by lateral stresses on the FeTi(Al) high-temperature/ FeTi(Al) iron enriched border, which are a result of the FeTi(Al) lattice parameter dependence on iron concentration. It was shown that the requirement of the equality between diffusion chemical potential $\mu_{\text{Al}}-\mu_{\text{Fe}}$ of the parent phase and that of the low temperature FeTi(Al) , lead, within the quasi-chemical approximation, to the observed iron enrichment of the latter.

Microstructure and Properties II

Wednesday AM Room: Cypress 2
July 19, 2000 Location: The Westin Bayshore Hotel

Session Chairs: M. Mills; L. M. Hsiung

8:30 AM

Fine Structure of C-Component Dislocations Associated with Pyramidal Slip Activity in Ti3Al: Jörg M.K. Wiezorek¹; Hamish L. Fraser²; ¹University of Pittsburgh, Matls. Sci. and Eng., Sch. of Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA; ²The Ohio State University, Matls. Sci. and Eng. Dept., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Ti3Al is a major constituent in super-alpha-2 alloys and a minor constituent in two-phase lamellar TiAl-based alloys, which are currently considered for high temperature structural applications in transport system. The lamellar grains in the latter consist of thin slabs of TiAl and Ti3Al and exhibit anisotropic mechanical properties. Single crystals of Ti3Al are also mechanically anisotropic. The (0001)<11-20> and {1-100}<11-20> slip systems can be activated with relative ease in Ti3Al, whereas c-component dislocation slip, {2-201}<11-26> and {11-21}<11-26>, operates only for loading close to the c-axis. The latter slip system is associated with anomalous yielding and the deformation behavior of lamellar grains in two-phase TiAl may be affected by the behavior of the minority phase to a significant degree. Thus, the fine structure of c-component dislocations activated during c-axis load-

ing of lamellar grains in TiAl-alloys has been studied by conventional and high-resolution transmission electron microscopy. In addition to the large amounts of debris characteristically associated with pyramidal slip in Ti3Al non-planar configurations of c-component dislocations have been observed. The results are discussed in relation to the mechanical behavior of Ti3Al and lamellar TiAl. This research is supported by NSF (DMR-9622497), Dr. Bruce MacDonald acting as Program Manager.

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Effect of Nd on Microstructure and Strengthening of TiAl Alloy: Fusheng Sun¹; Chunxiao Cao¹; Minggao Yan¹; S. E. Kim²; Y. T. Lee²; ¹Beijing Institute of Aeronautical Materials, Lab. of Ti Alloys, P.O. Box 81-15, Beijing 100095 China; ²Korea Institute of Machinery and Materials

Effect of Nd on the microstructure, tensile property, and creep property of Ti-47.5Al-0.25Nd and Ti-46.5Al-2Cr-2Nb-0.25Nd alloys has been studied. The addition of Nd results in the formation of neodymium oxides. By adding Nd, the cast microstructure of Ti-46.5Al-2Cr-2Nb-0.25Nd alloy is remarkably refined after conventional heat treatment, showing the fine colony grain size of about 15-30 micrometer. Both the tensile strengths and ductility at room and high temperature are significantly improved. The strengthening mechanism of Nd-bearing TiAl alloys can be explained by the superposition of second phase strengthening by big neodymium oxide particles, dispersion strengthening by fine precipitated neodymium oxides and fine colony grain size strengthening.

9:10 AM

Nucleation and Growth of the Ferromagnetic L10 Phase in Manganese-Aluminum Permanent Magnet Alloys: Evolution of Microstructure and Defect Structure: Gagatay Yanar¹; Jörg M.K. Wiezorek¹; Velemir R. Radmilovic²; William A. Soffa¹; ¹University of Pittsburgh, Matls. Sci. and Eng., Sch. of Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA; ²National Center for Electron Microscopy, Berkeley; & University of Belgrade, Dept. of Phy. Metall., Belgrade 11001 Serbia

Manganese-aluminum alloys in the vicinity of the equiatomic composition exhibit an attractive combination of magnetic properties for application in various devices including thin films. These technical magnetic properties derive from the formation of a metastable L10 intermetallic phase (τ -MnAl) characterized by a high, uniaxial magnetocrystalline anisotropy with an easy "c-axis." Carbon is generally added to stabilize the tetragonal τ -MnAl phase with respect to the formation of the stable phases in the system. The magnetic hysteresis behavior of these permanent magnet alloys is extremely sensitive to the microstructure and defect structure produced during the formation of the τ -phase (L10) within the parent high-temperature epsilon-phase (hcp). In this study modern metallographic techniques including high-resolution electron microscopy (HREM) have been applied to elucidate the nature of the phase transformation and evolution of the unique microstructure and defect structure governing the resultant structure-property relationships of these materials. The atomic processes occurring at the migrating interphase interfaces during transformation are shown to play a critical role in the generation of the so-called polytwinned microstructures and the profusion of planar defects characteristic of the MnAl-base ferromagnets.

9:30 AM

Microstructure Control in Two-Phase (B2+L12) Ni-Al-Fe Alloy by Adding of Carbon: Myung-Hoon Oh¹; Soo-Hyun Kim²; Je-Hyun Lee³; Dang-Moon Wee²; ¹Kumoh National University of Technology, Mat. Sci. and Eng. Dept., Kumi 730-701 Korea; ²KAIST, Mat. Sci. & Eng., Taejon 305-701 Korea; ³Changwon National University, Metall. Eng., Changwon 641-773 Korea

Carbon and titanium were added in NiAl-Fe/Ni3Al-Fe two-phase alloys for carbides precipitation, and the additions of 0.2wt.%C and 3at.%Ti have been found to be the most adequate for formation of carbides. Carbon doped alloys showed more refined microstructure than other carbon free alloys. The carbides, which were formed in the as-cast microstructure, appeared to play a role to suppress the grain growth and the coarsening of second phases. The NAF29-10 alloy including no carbides showed the lamellar type microstructure, while the NAF29-10-C alloy including carbides showed much more refined 'mesh' type microstructure. When the carbon free alloys were quenched into water, cracks occurred at the grain boundaries probably due to martensitic transformation, however, these cracks were not observed in the carbon doped alloy. Therefore, the carbon doped alloys showed good R.T. ductility. They also showed much higher yield strength than other alloys including no carbides through the whole temperature range.

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Microstructural and Mechanical Properties of a (Ni, Cu)Al Intermetallic Compound in Its Rapidly Solidified Condition: Sergio Garcia-Galan¹; Carlos Gonzalez-Rivera¹; Julio Alberto Juarez-Islas¹; ¹Instituto De Investigaciones En Materiales-Unam, Matls. Metal. Y Ceram., Circuito Exterior S/N, Cd. Universitaria, Mexico, D.F. 04510 Mexico

Intermetallic compounds have long been recognized as potentially useful structural materials for high temperature applications, however their utilization have been limited by the lack of low temperature ductility. Solidification processing of intermetallic compounds upon equilibrium phase diagram appears to be quite limited. With conventional melt processing one or more peritectic reactions and wide liquidus-solidus separation are encountered and lead to large-scale composition segregation which is difficult to eliminate during post-solidification treatments. At the same time the characteristics which make intermetallic compounds unattractive for conventional processing qualify them as suitable candidates for rapid solidification processing. In this work, we report the results obtained after characterization of a (Ni, Cu)Al intermetallic compound after been rapidly solidified, giving emphasis to microstructure and mechanical properties by means of scanning transmission electron microscopic observations, X-ray and electron diffraction patterns and strength versus strain curves.

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Microstructural Control for Strengthening the γ -Fe/E₂-(Fe,Mn)₃AlC_x Alloys: Yoshisato Kimura¹; Kunio Hayashi¹; Kazuyuki Handa¹; Yoshinao Mishima¹; ¹Tokyo Institute of Technology, Matls. Sci. and Eng. Dept., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

The E₂ type intermetallic compound (Fe,Mn)₃AlC_x can be an excellent strengthening phase for the fcc-based γ -Fe alloys aiming high-temperature application, exceeding 950 K, since the E₂ ordered crystal structure is quite similar to that of the L1₂, well-known strengthener of Ni-base superalloys. The E₂ has an interstitial carbon atom on the body-center site of the L1₂. Typical microstructures of the γ -Fe/E₂-(Fe,Mn)₃AlC_x two-phase alloys are that the E₂ phase precipitates as cubes or plates in the γ -Fe matrix depending on the lattice parameter mismatch. To improve the mechanical properties of the alloy through the microstructural control, we have modified the lattice parameter mismatch with two strategies: (1) choosing the appropriate compositions in the Fe-Mn-Al-C system, and (2) the addition of transition metal elements substituting for Fe and Mn. Scanning and transmission electron microscopic observations and mechanical tests were performed to investigate the relationship among microstructures, lattice parameter mismatch and mechanical behaviors.

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Microstructure of Al₃Sc with Ternary Transition-Metal Additions: Yoshihisa Harada¹; David C. Dunand¹; ¹Northwestern University, Dept. of Matls. Sci. and Eng., MLSB, Evanston, IL 60208 USA

Scandium trialuminide (Al₃Sc) precipitates confer high strength to Al-Sc alloys. Al₃Sc is also an interesting bulk structural intermetallic due to its low density, high melting point, good oxidation resistance and L1₂ crystal structure. However, very little is known on the alloying behavior of this intermetallic, whereby Sc can be replaced substitutionally by other elements to improve mechanical properties and decrease cost. The microstructure of arc-melted Al₃Sc with and without ternary alloying elements (Ti, V, Y, Zr, Nb, Hf, Ta) was examined by different techniques as a function of alloying element concentration. Optical and scanning electron microscopy were used to ascertain phases present at room temperature. The composition of each phase was determined by energy-dispersive x-ray microanalysis, while their lattice constants were measured by x-ray diffraction. Also, microhardness measurements allowed to rank the strengthening efficiency of each alloying element.

11:10 AM

The Chemical Composition and Microstructure of a Binary Eutectic Cr-Ta Alloy: Y. H. He¹; P. K. Liaw¹; C. T. Liu²; L. Heatherly²; E. P. George²; ¹University of Tennessee, Dept. of Matls. Sci. and Eng., Knoxville, TN 37996-2200 USA; ²Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831 USA

The effect of chemical composition on the solidification microstructures of binary Cr-Ta alloys was investigated employing both an arc-melt furnace and a high-temperature optical floating zone furnace. Five different Ta levels were investigated: 9.0, 9.2, 9.4, 9.6, 9.8, and 13.0% [all compositions in atomic percentage (at.%)]. The metastable and equilibrium microstructures of these solidified alloys were examined using optical and scanning electron microscopes. Because of Cr evaporation during directional solidification, the alloy with the best eutectic structure (Cr-9.4Ta) had a somewhat higher Cr concentration than that reported previously in the literature (Cr-9.8Ta). Alloys containing less than 9.4% Ta consisted of primary dendrite grains plus a Cr solid-solution phase. In alloys containing more than 9.4% Ta, regular polygonal grains of the primary Cr₂Ta Laves phase were observed. The Cr-13Ta alloy was found to have a hypereutectic microstructure consisting of a primary Laves phase plus eutectic. In the hypereutectic alloys, one of the plates of the Cr₂Ta phase in an eutectic colony was always connected to the primary Cr₂Ta Laves phase. The eutectic colony size decreased with increasing Ta concentration. In addition, the hardness of the arc-melted alloys was influenced by their chemical composition, with the eutectic alloy having the lowest hardness. An aligned microstructure of the binary eutectic Cr-9.4Ta alloy was obtained using directional solidification in a high-temperature optical floating zone furnace. This research is sponsored by the Fossil Energy Advanced Research and Technology Development (AR & TD) Materials Program under subcontract 11X-SP173V to the University of Tennessee with Dr. R. R. Judkins as the contact monitor, and the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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Microstructural Evolution of a Al67Mn8Ti24Nb1 Alloy during Mechanical Milling and Recovery Process: Jian Sun¹; Jing-xu Zhang¹; Geng-xiang Hu¹; ¹Shanghai Jiao-tong University, Sch. of Matl. Sci. and Eng., Huashan Rd. 1954, Shanghai 200030 PRC

Al67Mn8Ti25Nb1 is a Mn/Nb-modified multi-phase Al3Ti based alloy which is of the L12 matrix with dispersed DO22 precipitates. This alloy has been demonstrated to be of higher strength and toughness than the ternary Al67Mn8Ti25 alloy. The present paper is emphasized on the microstructural evolution of this alloy during mechanical milling and

recovery process by X-Ray Diffraction, Differential Thermal Analysis and Transmission Electron Microscopy. In particular, in-situ TEM observations were performed to study the reordering and precipitating during heating process. The results showed that the DO22 precipitates dissolved into the L12 matrix in the early stage of milling and the alloy structure transformed to FCC supersaturated solid solution after 10 hours and finally to the amorphous after 60 hours of milling. The opposite order of transformation occurred during the heating process in this alloy. Finally, the microstructural controlling and toughening of the Al67Mn8Ti25Nb1 alloy were discussed.

Physical and Metallurgical Properties II

Wednesday PM Room: Salon 1
July 19, 2000 Location: The Westin Bayshore Hotel

Session Chair: M. G. Mendiratta; Takayuki Takasugi

1:30 PM

The Effects of Environment, Vacancies, Hydrogen Charging and Strain Rate on the Mechanical Behavior of FeAl: *Ian Baker*¹; D. Wu¹; M. Wittman¹; Y. Yang²; S. O. Kruijver³; E. P. George³; ¹Thayer School of Engineering, Dartmouth College, 8000 Cummings Hall, Hanover, NH 03755 USA; ²Hypertherm, Inc., 21 Great Hollow Rd., Hanover, NH 03755 USA; ³Oak Ridge National Laboratory, Mats. and Cers. Div., Oak Ridge, TN 03781-6093 USA

The interactions of quenched-in vacancies, strain rate, cathodic hydrogen-charging and specimen orientation on the fracture strain, fracture strength and yield strength have been studied in FeAl single crystals. It has been shown that, independent of strain rate, large fracture strains are possible in single crystals tested in vacuum. In contrast, in air, the ductility increases with increasing strain rate, from only a few percent at 1×10^{-6} s⁻¹ to ~26% at 1 s⁻¹. Interestingly, in air, the yield strength appears to increase with increasing strain rate up to 1×10^{-2} s⁻¹. Quenched-in vacancies increase the yield strength and concomitantly reduce the ductility (but not the fracture strength) in both air and vacuum. Cathodic charging has been clearly shown to reduce the yield strength both in single crystals and, to a lesser extent, in polycrystals. The mechanisms associated with each of these effects will be discussed. Research supported at Dartmouth College by the NSF grants DMR-9973977 and DMR-9812211, by DOE contract DE-FG02-87ER45311, and by the Oak Ridge Associated Universities ShaRE program contract DE-AC05-76OR00033; and at ORNL by the Division of Materials Sciences, U.S. DOE under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

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Hot Corrosion Property of TiAl: *Toshiharu Noda*¹; ¹Daido Steel Company Limited, R&D Div./Special Steel Rsch. Dept., 2-30 Daido-cho Minami-ku, Nagoya, Aichi 457-8545 Japan

Light-weight intermetallic compound TiAl was put to practical use as a turbine wheel of an automotive turbocharger in last year in the world for the first time. It is expected that the use of TiAl turbocharger to a diesel engine with much demand of the turbocharger will be expanded in future. As light oil and heavy oil used by diesel engines contain a small amount of sulfur and vanadium, alloys used in combustion gas undergo hot corrosion such as sulfur and vanadium attack by the deposits of combustion products accumulated on the alloy surface. To use TiAl in such environment, it is necessary to investigate the hot corro-

sion property of it. In this study, the sulfur and the vanadium attack properties of TiAl were studied in comparison with conventional heat-resistant alloys. Moreover, the effect of third alloying elements on hot corrosion was also investigated.

2:10 PM

The Corrosion Behavior of Titanium Aluminides in H₂/H₂S/H₂O Atmosphere at 800-1000°C: *W. Kai*¹; M. T. Chang¹; C. Y. Bai²; ¹National Taiwan Ocean University, Instit. of Matls. Eng., Keelung 20224 Taiwan; ²Chung-Cheng Institute of Technology, Dept. of Mech. Eng., Ta-Hsi, Tao-Yuan 33509 Taiwan

The corrosion behavior of three Ti-Al intermetallics containing 20, 30, and 40 wt.% Al was studied over the temperature range of 800-1000°C in a H₂/H₂S/H₂O gas mixture. Ti-20Al and Ti-40Al alloys were single-phase structure of Ti₃Al and TiAl, respectively, while Ti-30Al was two-phase structure of Ti₃Al + TiAl. The corrosion kinetics followed the parabolic rate law in all cases, regardless of temperature and alloy composition. The parabolic rate constants increased with increasing temperature but decreased with increasing Al content. The Ti-40Al alloy exhibited the best corrosion resistance among all alloys studied. The scales formed on Ti-Al intermetallics were heterophasic, duplex scales, consisting of the outer-scale layer of exclusive TiO₂ and the inner-scale layer of TiO₂ and some amounts of α -Al₂O₃ is responsible for the reduction of the corrosion rates as compared with those of pure Ti.

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The High-Temperature Corrosion of Fe-28Al Base Alloys Containing Cr and Y Additions in H₂/H₂S/H₂O Mixed Gases: *W. Kai*¹; M. T. Chang¹; D. L. Chiang¹; J. P. Chu¹; ¹National Taiwan Ocean University, Instit. of Matls. Eng., Keelung 20224 Taiwan

The high-temperature corrosion behavior of three Fe-28Al-xCr (where x=2, 5, and 10 at.%) ternary and Fe-28Al-5Cr-0.5Y quaternary alloys was studied over the temperature range of 700-900°C in H₂/H₂S/H₂O mixed gases. The results showed that the corrosion kinetics followed the parabolic rate law in all cases. The parabolic rate constants decreased with decreasing temperature, but fluctuated with increasing Cr content. It was found that the Fe-28Al-5Cr alloy revealed the best performance to improve the corrosion resistance compared to Fe-28Al in the ternary alloys studied. The Fe-28Al-10Cr alloy showed poor corrosion resistance, and whose corrosion rates were only slightly lower than those of Fe-28Al and much higher than those of low-Cr alloys. In addition, the addition of Y did not provide any significant reduction in the corrosion rates as compared to Fe-28Al-5Cr. The scales formed on Fe-28Al-xCr were also strongly dependent on temperature and Cr content, while the scales formed on the Y-containing alloy were nearly identical to those of Fe-28Al-5Cr.

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Environment-Assisted Cracking of Gamma Titanium Aluminide in Aqueous Sulfate Solutions: *Takumi Haruna*¹; Taku Iwata¹; Thiyagarajan Sundararajan¹; Toshio Shibata¹; ¹Osaka University, Dept. of Matls. Sci. and Process., 2-1 Yamada-oka, Suita, Osaka 565-0871 Japan

We have investigated the environment-assisted cracking (EAC) of gamma TiAl in aqueous sulfate solutions with various pH. A tensile test at a slow strain rate of 3.3×10^{-6} s⁻¹ was employed to evaluate the EAC susceptibility. During the test, the electrochemical potential of the specimen was kept at a given value. In the pH region beyond 7, high maximum stress with plastic deformation was found in the potential region beyond -1000mVH, and the maximum stress decreased as the potential was lowered from -1000mVH. Whereas, in the lower pH region, the minimum value in the maximum stress was found at around -1000mVH. The EAC behavior was compared with the potential-pH diagrams of the

systems of Al/H₂O and Ti/H₂O. Only the diagram of Ti/H₂O system explained the EAC behavior: The high, the middle, and the low maximum stress region corresponded with the thermodynamical stable regions of titanium oxide, titanium hydride, and titanium ion, respectively.

3:10 PM

Improvement in Wear Resistance of TiNi-Matrix Composites by Hot Isostatic Pressing: *Haizhi Ye*¹; D. Y. Li¹; R. Eadie¹; ¹University of Alberta, Dept. of Chem. and Matls. Eng., Edmonton, Alberta T6G 2G6 Canada

It has been recognized that matrixes of many industrial tribo-composites are not strong enough and rapid wear of the matrixes significantly decreases the integrated wear resistance of the composites. However, it is not always effective by using a harder matrix, since it may not act effectively as a binder to retain the reinforcing particles. TiNi alloy exhibits excellent wear resistance and flexibility due to its pseudoelasticity, which may render this alloy an ideal matrix material for tribo-composites. The pseudoelasticity provides the flexibility to retain the reinforcing hard particles, to accommodate large deformation and to absorb impact energy with less damage. Recent work indicates that TiNi-matrix composites do possess high wear resistance and their resistance could be further enhanced if high-density pores/voids in the composites can be decreased. Attempts were made to improve the composites using a hot isostatic pressing process (HIP). This paper reports effects of the HIP process on the reduction of pores/voids of TiN/TiNi and TiC/TiNi composites and resultant improvement in wear resistance of the composites. The mechanism responsible for the improvement is discussed, based on corresponding changes in the density of pores or voids, mechanical properties and microstructure that were determined using SEM, XRD and triboscope, respectively.

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Rotary Friction Welding of an Fe₃Al Based ODS Alloy: Peter D. Sketchley¹; *Philip L. Threadgill*¹; Ian G. Wright²; ¹TWI, Granta Park, Great Abington, Cambridge CB1 6AL Great Britain; ²Lockheed Martin Energy Research Corporation, Oak Ridge, TN 37381 USA

An Fe₃Al based oxide dispersion strengthened (ODS) alloy is under consideration for possible use as tubes in advanced heat exchangers. As part of the validation process, it is necessary to investigate methods of joining the alloy to itself, and to Haynes 230 alloy. Previous experience on iron aluminides has shown them to be weldable by several processes, but it is known that fusion processes invariably lead to a loss of the Y₂O₃ oxide dispersion which is an essential feature of ODS alloys. Therefore, solid state processes offer a potential advantage, and in this work continuous drive rotary friction welding has been investigated as a method to join the Fe₃Al ODS alloy in both the recrystallised and unrecrystallised condition. Trials were also undertaken to join both recrystallised and unrecrystallised material to Haynes 230 alloy. All welds were made in 15mm diameter material, using a conventional continuous drive rotary friction welding machine. Welds were evaluated initially using bend tests, and detailed metallographic observations of the grain size, and the nature of the solid state interface between the materials. It was found possible to make high quality welds containing no flaws for a variety of welding conditions. The microstructural condition of the Fe₃Al ODS alloy had no apparent influence on weldability, and no difficulty was encountered in making the dissimilar metal joints. The results obtained are discussed in terms of the microstructures obtained, and are compared with other studies on joining iron aluminides and ODS alloys.

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The Effect of Texture on the Weld Structure and Properties of Gamma-TiAl Sheet Material: *Viola L. Acoff*¹; Shenavia Wilkerson¹; ¹The University of Alabama, Dept. of Metall. and Matls. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

The microstructure and mechanical properties of a welded gamma-TiAl sheet material was investigated. The alloy was received in the primary annealed condition which consisted of a microstructure containing equiaxed gamma grains with alpha-2 particles located at grain boundaries and triple points. Gas tungsten arc welding was performed both along and transverse to the direction of rolling and the microhardness, tensile properties and microstructure of the welds were determined. Preliminary results showed that the specimens welded transverse to the rolling direction had cracks that were parallel to the welding direction (normal to the rolling direction) and the cracking was much more severe than those for the specimens welded in the longitudinal direction. The fusion zone hardness of the transverse specimens was slightly lower than that for the longitudinal samples. This corresponded to a coarser fusion zone microstructure for the transverse samples. The weld structure-property relationship was established as a function of texture.

4:10 PM +

Comparative Study on the Resistance of Aluminide Intermetallic Compounds and Platinum Against Electrical Discharge: *Flavio Andrés Soldera*¹; Nenad Milivoje Ilic¹; Frank Mücklich¹; ¹University of Saarland, Instit. for Funct. Matls., P.O. Box 15 11 50, Gebäude 22, 7. Etage, Saarbrücken, Saarland D-66123 Germany

Electric arcs produce extreme thermal load on the surface of materials, which leads to a strong erosion in the material surface. An approach was made in order to compare the resistance of aluminide intermetallic compounds and platinum, against this attack. Volumes of individual craters produced by the discharge under defined physical conditions and different atmospheres were measured. The precise quantification of the crater topography could be achieved by the application of white light interferometry. The material loss showed comparable results in both materials, nevertheless, the microstructure of the aluminides seems to have a big influence on the erosion characteristics. Special attention was given to the characterization of the eroded surface after long time arcing.

4:30 PM

Effects of TiN Nano-Powder on Wear Behavior of TiC/TiNi Composite: *Y. C. Luo*¹; D. Y. Li¹; ¹University of Alberta, Dept. of Chem. and Matls. Eng., Edmonton, Alberta T6G 2G6 Canada

Pseudoelastic TiNi alloy has been found to exhibit high wear resistance, which greatly benefits from its pseudoelasticity. The wear resistance of this alloy can be considerably enhanced when hard particles such as TiC are embedded in the alloy. Recent work by the authors demonstrated that such a composite could be further improved by adding nano-TiN powder to the TiNi matrix of the composite. In this work, integrated wear performance of TiC/TiNi composites containing various fractions of nano-TiN particles were evaluated using a pin-on-disk tribometer. Effects of the nano-TiN particles on mechanical and tribological properties of the TiNi matrix were investigated using a triboscope—a combination of nano-mechanical probe and atomic force microscope. It was demonstrated that the balance between the pseudoelasticity and the hardness determined the wear resistance of the TiNi matrix. A small fraction of nano-TiN particles increased the hardness of the TiNi matrix without losing much pseudoelasticity. As a result, the integrated wear resistance of the composite was greatly enhanced. A wear model for the synergic effect of hardness and pseudoelasticity on wear resistance was developed to elaborate the experimental observation.

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Aging Effect on Micro-Mechanical and Micro-Tribological Properties of Pseudoelastic TiNi Alloy: *Xin J. Ma*¹; D. Y. Li¹; ¹University of Alberta, Dept. of Chem. and Matl. Eng., Edmonton, Alberta T6G 2G6 Canada

Recent studies have shown that TiNi shape memory alloy could be an excellent tribo-alloy due to its special deformation behavior—pseudoelasticity. It was anticipated that the wear resistance of this alloy

would be further improved by heat treatment, such as aging that can introduce coherent $Ti_{11}Ni_{14}$ precipitates. The $Ti_{11}Ni_{14}$ precipitates may strengthen the alloy and influence its pseudoelasticity as well. In this work, cold-drawn Ti-51at%Ni alloy was aged at various temperatures in the range from 350°C to 600°C. The micro-wear behavior of differently treated specimens was investigated using a nano-mechanical probe. It was demonstrated that the specimens respectively aged at different temperatures showed remarkably different wear resistances. In order to understand the effect of aging on the wear behavior and provide the guidance for microstructural optimization, micro-mechanical properties of the specimens were investigated using the nano-mechanical probe and corresponding macro-wear behavior was also evaluated using a pin-on-disc tribometer. Martensitic transformation temperatures and microstructures of different specimens were determined using DSC and TEM, respectively. Efforts of aging on the wear resistance of the TiNi alloy are discussed.

Creep II and Superplastic Deformation

Wednesday PM Room: Salon 3
July 19, 2000 Location: The Westin Bayshore Hotel

Session Chairs: K. Maruyama; Soo-Woo Nam

1:30 PM

Creep Characteristics and Internal Stress in γ -TiAl: O. Takahashi¹; Y. Terada¹; M. Takeyama¹; T. Matsuo¹; ¹Tokyo Institute of Technology, Dept. of Metall. and Cer. Sci., Meguro-ku, Tokyo 152-8552 Japan

The creep deformation of γ -TiAl polycrystal alloys has been characterized in terms of the activation energy for creep Q_c , stress exponent n and internal stress σ_j . The stress exponent of the minimum creep rates n is 3 at stresses below 70 MPa, regardless of the temperatures from 1123 to 1223K, whereas it increases to 8 above 150 MPa. The activation energy for creep Q_c obtained at 68.6 MPa varies from 300 to 700 kJ/mol with increasing the temperature. The internal stress σ_j is measured by strain dip tests decreases with increasing the temperature. The decrease of internal stress would be attributed by the decrease in dislocation density with increasing the temperature. Based on these findings, it is elucidated that the activation energy Q_c^* obtained at a constant effective stress $\sigma_e (= \sigma_a - \sigma_j)$ becomes 245kJ/mol, independent of the temperature.

1:50 PM

Creep Behavior of Gamma-TiAl Sheet Material with Differently Spaced Fully Lamellar Microstructures: Anita Chatterjee¹; Eduard Arzt¹; Helmut Clemens²; ¹Max-Planck-Institut für Metallforschung, Seestr.92, Stuttgart 70174 Germany; ²Universität Stuttgart, Institut für Metallkunde, Seestr.71, Stuttgart 70174 Germany

Recent investigations have shown that the interface spacing in fully lamellar microstructures has a major influence on the creep behavior of gamma-TiAl based alloys. In order to study the dependence of lamellar spacing on creep, fully lamellar microstructures exhibiting different interface spacings but comparable colony size were adjusted in Ti-46.5at.%Al-4at.%(Cr,Nb,Ta,B) sheet material by appropriate heat-treatments. Creep tests were conducted in a temperature range of 700 to 800°C and stresses between 100 and 300 MPa. The results indicate that the minimum creep rate decreases with decreasing lamellar interface spacing. In addition, activation energies and stress exponents were determined in order to describe the creep controlling mechanism. A model assumption, which considers the limitation of the free dislocation path

by stored dislocations as well as by geometrical obstacles was applied to explain the role of the interface spacing during creep. In addition, microstructural changes during short term creep will be discussed.

2:10 PM

Creep of the Intermetallic NiAl: Y. L. Wang¹; I. P. Jones¹; Raymond Edward Smallman¹; ¹The University of Birmingham, Sch. of Metall. and Matls., Edgbaston, Birmingham B15 2TT UK

The Intermetallic NiAl in the stoichiometric and alloyed (Ni40 Al50 Fe10%) condition was prepared by plasma arc melting from 99.999% pure Al and 99.99% pure Ni. Compression creep specimens solution treated at 1200°C were tested at 600°C, 750°C and 900°C at stresses of 118-181 MPa at low temperatures, and 31-75 MPa at the highest temperature, before quenching into iced water to retain the high temperature structure. Crept specimens were sectioned at 45° to the compression axis, thinned and examined in a CM20 electron microscope. At all temperatures and stresses used the specimen exhibited a relatively short primary creep regime, followed by extensive steady-state creep obeying the classical creep equation with the stress exponent $n \sim 3$. The creep rate depends sensitively on composition and the iron addition reduces the rate by an order of magnitude at 900°C and 31.25 MPa. Creep is mainly due to the glide and climb of $\langle 100 \rangle$ dislocations leading to subgrain boundary formation. Further detailed microscopy will be presented.

2:30 PM

Inverse Creep of Intermetallics: R. E. Smallman¹; Tie Sheng Rong¹; S. C.D. Lee¹; I. P. Jones¹; ¹The University of Birmingham, Schl. of Metall. and Matls., Edgbaston, Birmingham B15 2TT UK

Conventional primary and steady state creep have been observed in intermetallics, but under certain conditions primary creep is sometimes followed by a region in which creep-rate increases with strain. Observations have been made of inverse creep in both polycrystalline and single crystal Ni_3Al and TiAl. The stability of microstructure reflected in steady-state creep is destroyed in inverse creep by microstructural instability. In both polycrystal and single crystal Ni_3Al , the operation of cube cross-slip, below the peak temperature, can increase dislocation activity and lead to inverse creep. Some stress-temperature conditions do exist, however, for cube cross-slip to operate without leading to inverse creep. Cube slip is considered a necessary requirement but is not the only condition for inverse creep. In TiAl, inverse creep is observed at temperatures above $0.6T_m$ for both polycrystalline samples (Ti-52Al, Ti-56Al) and for single crystals (Ti-56Al). In Ti-52Al, $\langle 110 \rangle$ ordinary dislocations predominate but $\langle 101 \rangle$ and $\langle 112 \rangle$ superdislocations also operate. In Ti-56Al for both single crystal and polycrystal samples $\langle 101 \rangle$ superdislocations predominate with much fewer $\langle 110 \rangle$ dislocations. There was no evidence for increased dislocation activity of either $\langle 110 \rangle$ or $\langle 101 \rangle$ at the onset of inverse creep. More commonly there is evidence of increased climb resulting in dislocation networks and low-angle boundaries. Inverse creep at these temperatures is considered to be dependent on recovery and the mobility of sub-grain boundaries.

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Creep Behavior in an Extruded β Solid Solution Nb-Ti Base Alloy: E. A. Loria¹; R. W. Hayes²; S. M. Allameh³; W. O. Soboyejo³; ¹Reference Metals Company, Inc., 1000 Old Pond Rd., Bridgeville, PA 15017-0217 USA; ²Metals Technology, Inc., Northridge, CA 91324 USA; ³Princeton University, Princeton, NJ 08558 USA

Extrusion, as a processing route, is the basis for this study of the β solid solution 44 Nb-35Ti-6Al-5Cr-8v-1W-0.3Mo-0.5Hf (at %) alloy. Heat treatments on longitudinal and transversely oriented specimens from the commercially produced 8 cm diameter extrusion revealed that 1 hr at 1200°C produced a recrystallized β structure that was stable between 25 and 704°C, with remarkable tensile strength and ductility within this range. Tension creep testing with the same specimens em-

phasizes an analysis of the creep curve for 704°C and 104 MPa, which apply to service conditions. A short primary creep regime to a steady state and an extended territory creep regime are observed, with an inverted creep behavior indicative of Class I creep. A range of dislocation configurations was observed which include dislocation looping around TiC particles; Srolovitz-type dislocation grazing of TiC particles; kinked and bowed particle dislocations in a few regions.

3:10 PM Break

3:30 PM Invited

High Temperature Deformation Behavior and Superplasticity in Iron Aluminides: *Dongliang Lin*¹; Aidang Shan¹; ¹Shanghai Jiao Tong University, Schl. of Matls. Sci. and Eng., Shanghai 200030 PRC

High temperature deformation behavior in a variety of iron aluminides with compositions range of 25-40 at% aluminum content and Ti or Cr alloy element additions was examined by tensile test, optical microscope (OM), orientation image microscope (OIM) and transmission electron microscope (TEM). Superplasticity was demonstrated in all these alloys with an initial grain size of more than 60 micrometer in a temperature range of 650-1100°C. Ti addition was found to have beneficial effect than Cr on the superplasticity. The large-grained iron aluminides exhibit all deformation characteristics of conventionally fine-grained superplastic alloys, without the prerequisites of fine grain size and grain boundary sliding. At the earlier stage, extensive grain boundary migration during deformation was revealed by OM while an equiaxed subgrain structure was found under TEM. After superplastic deformation initial large grains were found to be broken into finer grains with grain size of 10-30µm which varies with temperature and strain rate. The observed superplasticity behavior is explained by continuous recovery and recrystallization. During Superplastic deformation, an unstable subgrain network forms and the subgrain boundaries transforms into low and high angle grain boundaries by absorbing the gliding dislocations. A dislocation sliding and climbing process accommodated by subgrain boundary sliding, migration and rotation, maintains the superplastic flow to proceed.

4:00 PM

Grain Refinement of Intermetallics by Severe Plastic Deformation: *Shankar M.L. Sastry*¹; Rabindra Nath Mahapatra²; Shailendra K. Varma³; ¹Washington University, Mech. Eng. Dept., Campus Box 1185, One Brookings Dr., St. Louis, MO 63130 USA; ²Naval Air Systems Command, Matls. Lab., Code 6.3.4.2, Patuxent River, MD 20670 USA; ³University of Texas, Metall. Eng., El Paso, TX 79968 USA

Intermetallics literature indicates a trend of the beneficial effect of grain refinement on ductility, toughness, and yield strength, particularly when the grain size is reduced to < 1 mm. However, conventional thermomechanical processing of bulk intermetallics has not resulted in sub-micrometer sized grains. Nanograined intermetallics produced by particulate processing routes such as mechanical alloying and solution phase synthesis invariably have excessive contamination and porosity and improvements in ductility and toughness have not been realized using nanoparticle processing approach. Severe plastic deformation (SPD) has been found to be a cost effective and viable method for producing ultra fine grain structures in bulk materials without the problems associated with particulate processing routes. In the present investigation, severe plastic deformation by equal channel angular extrusion (ECAE) was used to produce ultra fine grain microstructures in titanium aluminides and nickel aluminides. The ECAE process consists of extruding a well-lubricated billet through two intersecting channels of identical cross-section. Deformation is achieved by simple shear in a thin layer at the crossing plane of the channels. A large and uniform strain intensity per pass can be reached in material under low pressure and load without a reduction of the initial billet cross section. The process can be repeated a number of times in the same tool because the

billet cross-section remains constant. Gamma titanium aluminides can be deformed to large strains using equal channel angular extrusion at temperatures below the eutectoid isotherm. Such a method provides flexibility in producing greater microstructural control than hitherto has been possible. For example, lamellar $\gamma + \alpha_2$ phases can be deformed to large strains at as low temperature as 1000°C and recrystallized to fine grained equiaxed microstructure. Ni₃Al and NiAl have been processed by ECAE to produce fine grain microstructures. Preliminary results show that severe plastic deformation (SPD) with concurrent or post deformation recrystallization holds promise for improving the room temperature ductility and toughness and high temperature superplasticity of intermetallics.

4:20 PM

Observation of High-Strain-Rate Superplasticity in Coarse-Grained Iron Aluminides: *Jinn P. Chu*¹; S. H. Chen¹; W. Kai¹; K. Inoue²; ¹National Taiwan Ocean University, Instit. of Matls. Eng., 2 Pei-Ning Rd., Keelung, Taiwan 20224; ²University of Washington, Dept. of Matls. Sci. and Eng., Roberts Hall, FB-10, Seattle, WA 98195 USA

For metals and ceramics exhibiting superplasticity, the fine-grained structure (typically <10 µm) is generally known to be indispensable. Yet, as a result of dynamic recovery and recrystallization, Fe-Al-based alloys have been reported to show superplasticity when the grain size is much greater, >500 µm. To establish better understanding of superplastic properties, the present study is directed toward investigation of the strain rate effect on superplastic deformation of Fe-Al-based alloys. Tensile tests are conducted at temperatures of 700, 800 and 900° C in air at initial strain rates ranging from 5×10^{-1} to 1×10^{-4} sec⁻¹. It is found that even at a high strain-rate of 5×10^{-1} sec⁻¹ Fe-28Al-2Cr (in atomic percent) alloy exhibits more than 280% elongation, while Fe-28Al shows ~200% elongation in the same strain rate range. Microstructures are studied and results show that grain structures are altered after superplastic deformation. These microstructural alterations are resulted from the dynamic recovery and recrystallization, as evidenced by grain-boundary migration and grain refinement. In this paper, the high-strain-rate superplasticity will be discussed in light of microstructural evolution results.

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Low-Temperature Superplastic Behaviors of a Thermo-Mechanically Processed TiAl Based Alloy: *Jian Sun*¹; ¹Shanghai Jiao-tong University, Sch. of Matl. Sci. and Eng., Huashan Rd. 1954, Shanghai 200030 PRC

Low-temperature superplasticity of TiAl alloys becomes important because the superplastic deformation temperature is normally high (1000°C or above), which results in significant oxidation and cavitation of the alloys. This paper emphasized on the low-temperature superplastic behaviors of a TiAl-based alloy subjected to multi-step thermo-mechanical processing. The alloy was investigated systematically at 800~900°C and at strain rates from 2×10^{-5} to 2×10^{-4} 1/s. The flow stress and strain rate sensitive value as a function of strain rates were determined by the incremental strain rate tests. Evolution of microstructures of tested alloys was performed by SEM and TEM. The results showed tensile elongation of 234% and 533% at 800°C and at 2×10^{-4} 1/s and 2×10^{-5} 1/s, respectively. The activation energy has been measured to be 220 kJ/mol. These results will be compared with the superplastic behaviors of the same alloy tested at relatively higher temperatures (1000~1075°C) and possible low-temperature superplasticity mechanism of this TiAl based alloy will be discussed also.

5:00 PM

Superplastic Deformation Behavior in a Large-Grained TiAl Alloy: *Feng Sun*¹; Dongliang T.L. Lin¹; ¹Shanghai Jiao Tong University, Schl. of Matls. Sci. and Eng., Shanghai 200030 PRC

High temperature deformation behavior were investigated systematically in a large-grained Ti-47Al-2Mn-2Nb-B alloy having nearly equiaxed

γ phase, in which fine particles of α_2 distribute uniformly. Specimens coated with enamel were tested in air, at temperatures from 1025°C to 1100°C, and strain rates ranging from $4 \times 10^{-5} \text{s}^{-1}$ to $8 \times 10^{-4} \text{s}^{-1}$. Both flow behavior and microstructure evolution during deformation have been examined, then the strain rate sensitivity value m and the Zener-Holloman factor Z were evaluated. Similar to FeAl reported in previous work, the TiAl alloy also exhibits all deformation characteristics of conventionally fine-grained superplastic materials, without the prerequisites of fine grain size and grain boundary sliding. In most cases, an elongation over 200% was gained. A maximum elongation of 270% with a m value of 0.3 was obtained at 1100°C and a initial strain rate of $4 \times 10^{-5} \text{s}^{-1}$, under the condition of uniform deformation. It is shown that the specimens may fail prematurely due to surface oxidation, a greater elongation could be expected while testing in vacuum. The metallographic examination have shown that the average grain size of large-grained TiAl alloy decreased during superplastic deformation, after that, a much finer grain size could be obtained. Combined with the TEM observations of substructure, a dynamic recovery and recrystallization process is discussed, which is believed to be the control mechanism of superplastic behavior for the TiAl alloy studied in the paper.

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