DEVELOPMENT OF Ni-Co BASE ALLOYS FOR HIGH-TEMPERATURE DISK APPLICATIONS

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Abstract

The development of novel industrial cast and wrought (C&W) disk alloys with required combination of strength, creep and fatigue resistances is always desired and a challenge to material researchers. Recently, in the High Temperature Materials 21 Project (HTM21) led by the Japanese National Institute for Materials Science (NIMS), we proposed an innovative concept to design disk alloys. This concept is based on combining the characters of two kinds of γ–γ′ two-phase alloys (Ni-base and Co-base alloys) and leads up to some promising candidate alloys that can be processed by normal C&W processing route and shows at least a 30°C increase in temperature capability over the current C&W U720Li disk alloy. This paper shows the design idea, workability and mechanical properties of some Ni-Co-base alloys. Furthermore, full-scale-disk forgings were processed successfully for the evaluations of the processing ability and microstructure of new Ni-Co-base disk alloys, which demonstrated the advantages and possibility of Ni-Co-base disk alloys at the component level.

Introduction

The latest design of high-efficiency engines has high requirements for the mechanical properties and temperature capability of the key components (disk and blade) of engines, especially the first stage of disks and blades where the stress and temperature are the highest [1]. The current alloys intended for use as first-stage disks or blades either in aircraft engines or land-based gas turbine engines are γ/γ′ two-phase Ni-base superalloys because of their excellent properties, which include high-temperature strength and resistance to creep and fatigue [2-7]. By increased the volume fraction of γ′ phase and added heavily solid-solution-hardening elements, the disk alloys such as Rene 95, RR1000 and ME3 are developed [8-10]. However, such an increases in the volume fraction, combined with increased levels of refractory elements, has led to difficulties in the control of grain structure during the forging process and in the obtainment of an optimum microstructure after forging because of segregation problems and the complicated interactions between the forging parameters and the large number of microstructure constituent elements during processing via normal cast & wrought (C&W) route. Therefore, all these disk alloys are produced via powder metallurgy (P/M) route. However, P/M processing route demands ultra-clean powder as source material as well as requires expensive thermo-mechanical processing steps to improve the fatigue life, which limit the P/M disk alloy to some special applications.

Recently, in the High Temperature Materials 21 Project (HTM21) at the National Institute for Materials Science (NIMS), we are focusing our research on developing a novel industrial disk alloy that can be processed by inexpensive C&W route and has 50 °C higher temperature capability (1000-hour creep life under 630 MPa) than current C&W U720Li disk alloy as shown in Figure 1.

To achieve this exciting target, we proposed an innovative concept to design new disk alloys [11-14]. This concept is based on combine the characters of two kinds of the superalloys with γ/γ′ two-phase structure (Ni-base and Co-base superalloys), where Ni-base alloy may offer basic mechanical properties required for disk applications and added Co-base alloy may benefit to increasing the C&W processing window of the mixed alloys (Figure 2). We named these Ni-Co-base alloys TMW alloys. In this paper, the phase constitute and thermal stability, workability and mechanical properties of some TMW alloys based on the mixtures of U720Li alloy (a two-phase Ni-base alloy) and Co-16.9 wt%Ti alloy (a two-phase Co-base alloy) were investigated. The results show that this design concept may lead up to some promising disk alloys that can be processed by normal C&W processing route and have at least 30 °C up in temperature capability compared with the best of the current list of C&W Ni-base disk alloy, U720Li alloy. Based on the research, a promising composition was suggested to a material maker and a pancake disk with $\phi 430mm \times 65mm$ in size was produced successfully via a process route of triple-melting, billet making and disk forging.
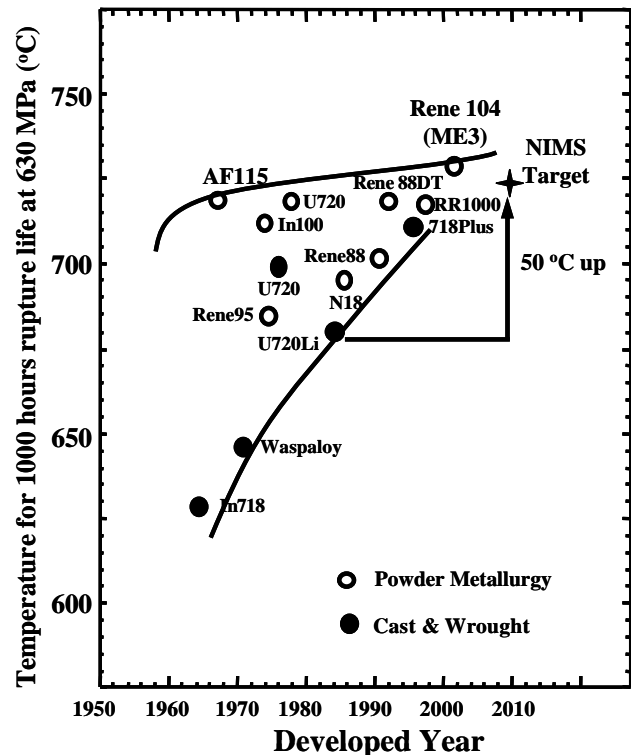

Figure 1. The target of new Ni-Co-based disk alloys compared with other commercial disk superalloys.

Alloy Design

New Ni-Co-base alloys were designed to be able to be processed by normal C&W route and have superior strengths by the control of the cobalt and titanium contents. The cobalt level is selected to control the gamma prime solvus temperature. Increasing amount of cobalt may decrease the gamma prime solvus temperature, which is desirable to obtain a large processing temperature range and reduce the stresses induced by controlled cooling and quenching of the alloy [15]. Titanium is a strong gamma prime former and readily substitutes for Al in the gamma prime phase [16].
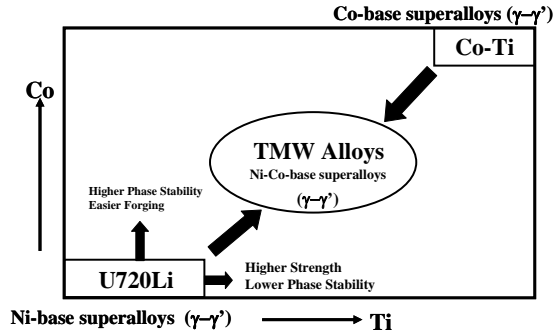

Figure 2. Design TMW disk alloys by an innovative concept.

Here we refer U720Li alloy as TMW 1 alloy as a comparative and got the compositions for other TMW alloys by mixing U720Li alloy with Co-16.9 wt%Ti alloy according to the following equation:

$$C_{TMW} = X C_{U720Li} + (1-X) C_{Co-16.9Ti} \qquad (1)$$

where C is composition and X may be changed from 0 to 1. By selecting X to be 0, 1, 0.15 and 0.2 respectively, we got the compositions for three TMW alloys and named as TMW-2, TMW-4A and TMW-5 (Figure 3). TMW-4A is just for calculation and is not investigated in the following tests. In Figure 3, the volume fraction of γ' phase at 760 °C was calculated by using of a Thermal-cal program with a Ni-RUMN database. Meanwhile, the compositions of the γ and γ' phases in each TMW alloys at 760 °C can also be calculated by the program. Basing on these calculated compositions, TMW-3 was designed as a tie-line alloy of TMW-2 and had 40% volume fraction of γ' phase, TMW-4 was designed as a tie-line alloy of TMW-4A and had
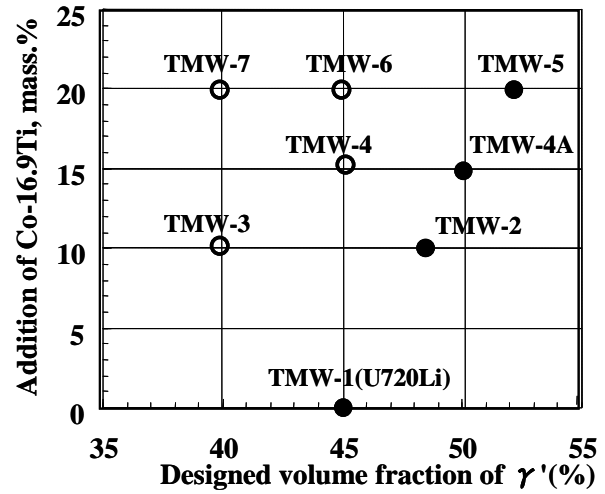

Figure 3. Schematic diagram of TMW disk alloys.

45% volume fraction of γ' phase, and TMW-6 and TMW-7 were designed as tie-line alloys of TMW-5 and had 45% and 40% volume fraction of γ'phase, respectively (Figure 3).

The nominal chemical compositions and the γ' phase volume fraction of TMW alloys are listed in Table 1. Like we know, commercial Ni-base disk alloys normally contain 10-20% Cr, up to 8% Al and Ti, 5-20% Co, and small amounts of B, Zr and C [17]. In TMW alloys, the contents of Co and Ti were higher than that of commercial disk alloys, and Co content was varied from about 22 to 31.1wt% and Ti content was varied from 5.4 to 7.4 wt%. No expensive element such like Ta and Nb contained in TMW alloys. The reported compositions of 718Plus, RR1000 and ME3 were also listed in Table I for a quick comparison.

Before further experiments, we used the Thermo-cal program to predict the existence of stable phases in TMW alloys at fabrication and operating temperatures. Besides γ and γ', σ phase was predicted to exist in all alloys. Meanwhile, η phase appeared in the alloys from TMW-3 to TMW-7 at the temperature above 500°C, and its content and solvus temperature increased with the increasing of Ti level. The solvus temperature of η phase is higher than that of γ' phase in the existed TMW alloy.

Table I. The Nominal Chemical Compositions of the TMW Alloys and Some Disk Alloys

Alloy	Ni	Co	Cr	Mo	W	Nb	Ta	Al	Ti	C	B	Zr	other	T_m	$T_{\gamma'}$	$\gamma'*_{vf}$
TMW1 (U720Li)	Bal.	14.8	16.3	3.1	1.3	-	-	2.4	4.8	0.03	0.02	0.03	-	1347	1142	45.0
TMW2	Bal.	21.8	14.4	2.8	1.3	-	-	2.3	5.9	0.03	0.02	0.03	-	1339	1182	48.7
TMW3	Bal.	23.1	16.8	3.1	1.3	-	-	1.9	5.4	0.03	0.02	0.02	-	1342	1135	40.0
TMW4A	Bal.	25.2	13.6	2.6	1.1	-	-	2.1	6.8	0.01	0.02	0.03	-	-	-	-
TMW4	Bal.	26.3	14.9	2.8	1.2	-	-	1.9	6.0	0.02	0.02	0.02	-	1340	1132	45.0
TMW5	Bal.	28.6	12.8	2.4	1.0	-	-	2.0	7.4	0.03	0.01	0.02	-	1330	1130	52.2
TMW6	Bal.	30.0	14.6	2.7	1.2	-	-	1.8	6.4	0.03	0.02	0.02	-	1338	1100	45.0
TMW7	Bal.	31.1	15.6	3.0	1.2	-	-	1.6	5.7	0.03	0.02	0.02	-	1347	1058	40.0
718Plus	Bal.	9.0	18.0	2.8	1.0	5.5	-	1.5	0.7	0.02	0.004	-	10Fe	-	-	-
RR1000	Bal.	18.5	15.0	5.0	-	-	2.0	3.0	3.6	0.03	0.02	0.06	0.75Hf	-	-	-
ME3	Bal.	20.6	13.0	3.8	2.1	0.9	2.4	3.5	3.7	0.04	0.03	0.05	-	-	-	-

*   At 760 °C

Experimental Procedures

For all tested seven TMW alloys, 50-kg ingots were made using vacuum induction melting (VIM) followed by casting and then hot extruded and hot rolled in the Special Metals Corporation (Huntington, WV). The ingots were hot extruded successfully into ϕ22 mm billets with extrusion ratio of 12:1 at about 1170°C. Before hot rolled, the ingots were soaked at 1149 °C for 12 hours. The final hot-rolling reduction of 75% (from 50.8 mm to 12.7 mm) was reached by rolling two passes at about 1100°C with inter-heat at 1121°C for 30 minutes. The hot rolling was conducted smoothly and the rolled plates have good surface condition. All follow-testing samples were cut from these rolled plates. Since only short cracks were observed at the surfaces of the extruded billets and hot-rolled plates, the workability of TMW alloys was great and acceptable.

Small samples with 10 mm × 10 mm × 5 mm were subjected to a standard heat treatment (1100°C/4hours followed by air cooling (AC), and then aging at 650°C/24hours/AC + 760°C/16hours/AC) reported for U720Li alloy. After the standard heat treatment, some samples were thermal exposed at 750°C up to 5000 hours. The phase constitutes and

the microstructure evolution caused by heat treatment and thermal exposure was observed by the analysis techniques of optical microscope, X-ray diffraction, three-dimensional atom probe (3DAP), scanning electron microscope (SEM) and transmission electron microscope (TEM). The specimens were prepared by metallographic polishing followed by etched in a solution of Marble reagent (10 g $CuSO_4$ + 50 ml HCl + 50 ml H2O). TEM samples were electrochemically polished with a twin jet polisher using a solution of 83% ethanol, 7% glycerol and 10% perchloric acid at -25°C.

Tensile specimens with a 22 mm × 4 mm diameter cylindrical gauge section were cut from the heat-treated plates. Tensile tests were carried out at temperatures ranging from room temperature to 1100°C. Constant load creep rupture tests were conducted at temperatures ranging from 650 °C to 760 °C and at initial applied stresses of 480 to 830 MPa.

Results

Microstructure and Thermal stability

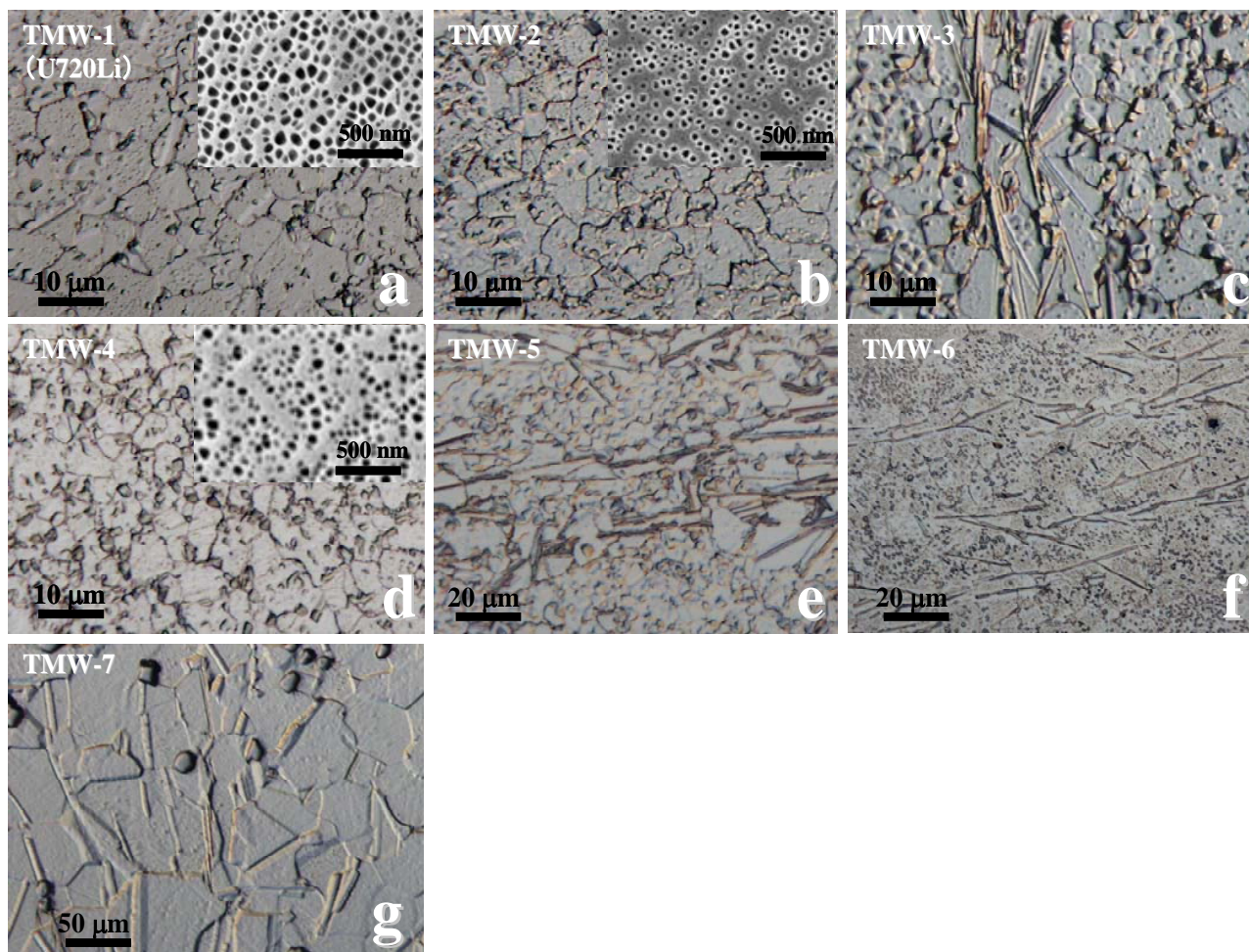Figure 4 shows the heat treated microstructures of TMW alloys. All

Figure 4. Typical microstructures of the TMW alloys examined in this study.

Table II. The Compositions of the Constitute Phases Analyzed by AP-FIM (3D)

	γ phase				γ′ phase			
	Ni	Co	Al	Ti	Ni	Co	Al	Ti
TMW-1(U720Li)	50.1	24.4	1.6	0.5	74.2	3.1	8.0	13.1
TMW-2	42.7	26.2	1.9	0.5	70.1	6.2	7.1	14.3
TMW-4	30.5	34.5	2.2	1.0	65.6	8.9	7.8	13.7

observed TMW alloys had a homogeneous microstructure which implies that the solution heat treatment was performed fully for both U720Li (TMW-1) and new Ni-Co-base alloys. The grain sizes are about 10 μm for alloys from TMW-1 to TMW-6 and over 50 μm for alloy TMW-7. For alloy TMW-1 (U720Li), TMW-2 and TMW-4, only γ/γ' two-phase structure consisting of large primary γ' precipitates, with sizes varying between 0.5 and 1.7 μm (Figures 4a, 4b and 4c), interspersed with fine γ' precipitates (typically less than 0.1 μm and formed upon aging or cooling from the aging treatment) was identified, and the morphology and size of γ' phase were almost the same (Figures 4a, 4b and 4d, enlarged view can be found at the right corner of the figure respectively). The compositions of the phases in TMW-1, TMW-2 and TMW-4 alloys were measured from selected volume analysis by the 3DAP as shown in Table II, which indicates that the γ' phase existed as $(Ni,Co)_3(Ti,Al)$ in these three TMW alloys, added cobalt was preferentially partial to the γ and titanium was partial to the γ' phase. Meanwhile, increasing cobalt and titanium in the TMW-2 and TMW-4 compared to TMW-1 resulted in an increase of the Co and Ti contents in the major γ' phases. For the other observed TMW alloys (Figures 4c, 4e, 4f and 4g), some plate-shape phase was observed. Chemical analysis indicated that the plate-shape phase was rich in Ni and Ti, and was confirmed to be η phase (Ni_3Ti) by X-ray diffraction analysis.

Microstructure investigations of TMW alloys subjected to thermal exposure at 750 °C for 2500 hours showed that there was no η phase

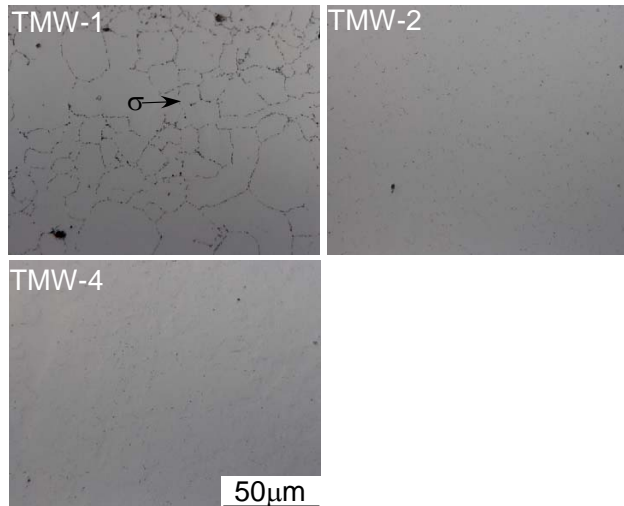existed in TMW-1, TMW-2 and TMW-4 alloys, but sigma (σ) phase was found in all tested alloys (Figure 5). The volume fraction of σ phase was greatly lower in TMW-2 and TMW-4 alloys than that in TMW-1 alloy (U720Li), suggesting that new Ni-Co alloys have better thermal stability compared with U720Li at temperature up to 750 °C. This investigation also implies that increase Co-Ti content and decrease Cr content in U720Li alloy had a great effect on suppressing the formation of topological close packed (TCP) phase, i.e. σ phase, in the added alloys.

It is noted that there was no η phase observed in TMW-4 alloy exposed at 750 °C for 2500 hours, where about 5% η phase is expected according to the program prediction. Two main reasons may cause the difference. First, the database we used in the thermodynamic calculations is a Ni-RUMN database, which is suitable for normal commercial Ni-base superalloys where Ni content is over 50 wt.% and Co content is lower 24 wt.%. As indicated in Table I, the compositions of present TMW alloys are extremely deviated from the commercial alloys. Second, the investigated samples were heat treated at the conditions that may be far from the equilibrium state.

Mechanical Property Testing

Since the purpose of this investigation was to examine the effect of new design idea on the mechanical properties of U720Li alloy, the following mechanical tests have been conducted only on TMW-1, TMW-2 and TMW-4 alloys, which have the same phase constitution and grain size. The optimum heat treatment condition for U720Li (1100°C/4hours followed by air cooling (AC), and then aging at 650°C/24hours/AC + 760°C/16hours/AC) was selected for treating all tested specimens.

Tensile Strength

Tensile tests have been performed at room temperature, 650°C, 750°C, 850°C and 1100°C, and average tensile results are presented in Figure 6. The tensile strength of the TMW alloys developed in this study was higher than that of U720Li alloy (TMW-1). TMW-4 alloy demonstrated an ultimate tensile strength up to 1700 MPa at room temperature and 1140 MPa at 750 °C, which is much better than that of U720Li alloy (Figure 6a). Moreover, even at 750 °C, the yield strengths of TMW-2 and TMW-4 alloys were still maintained at a level above 950 MPa, which is approximately 100 MPa higher than that of U720Li alloy (Figure 6b). The strength of TMW-2 and TMW-4 alloys were about 70°C up compared with the performance of TMW-1 (U720Li) alloy at 650 °C (Figure 6b). At 1100°C, the tensile strengths of all tested TMW alloys had almost the same value, which permits TMW alloys to be forged as U720Li alloy (TMW-1) (Figure

Figure 5. The microstructures of the TMW alloys subjected thermal exposure at 750 °C for 2500 hours.
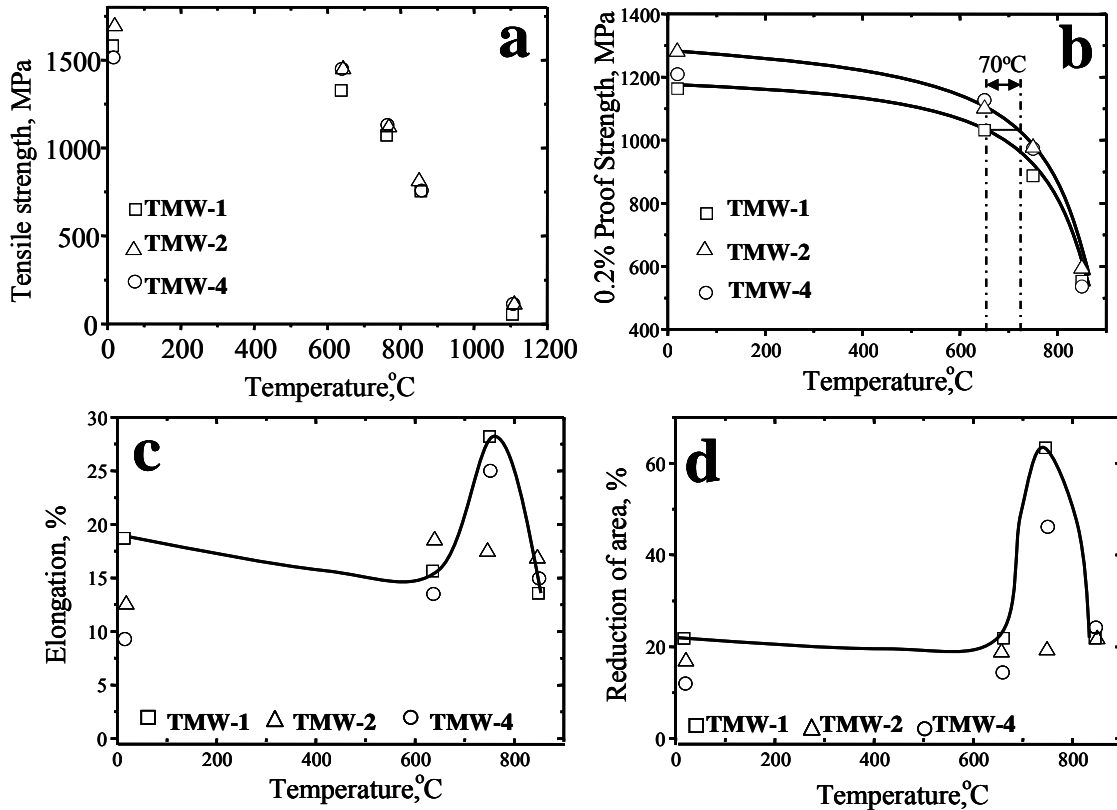
Figure 6. Results of the tensile tests for the tested TMW alloys.

6a). The tensile elongation of TMW-2 and TMW-4 alloys were lower than that of U720Li but was around 10% at room temperature and was close to that of U720Li (TMW-1) alloy at 850 °C (Figure 6c). The reduction of area of TMW-2 and TMW-4 alloys had the close values except at 750°C (Figure 6d).

Creep-Rupture Properties

Tensile creep tests have been performed in air and under various temperatures ranging from 650 °C to 750 °C, and loads ranging
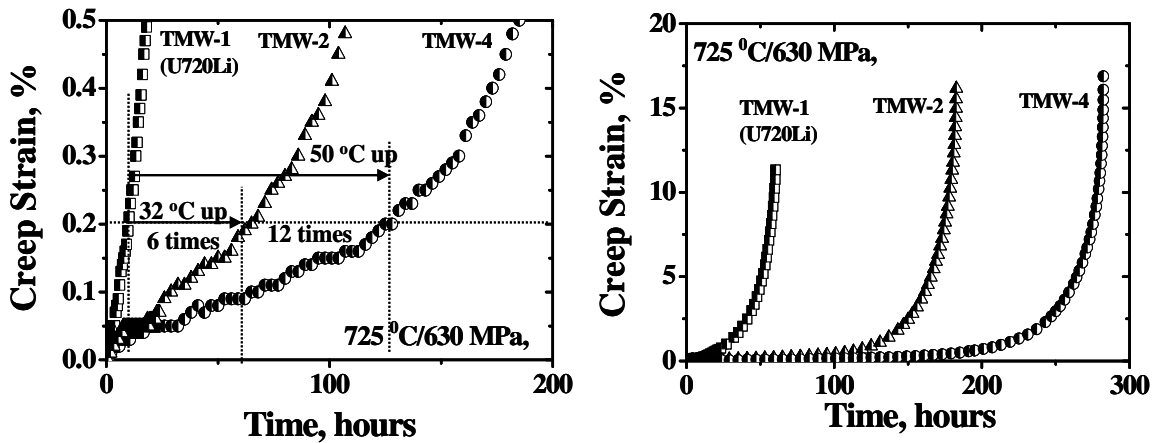

Figure 7. Tensile creep strain vs time for TMW-1, TMW-2 and TMW-4 alloys tested at our targeted temperature and stress.
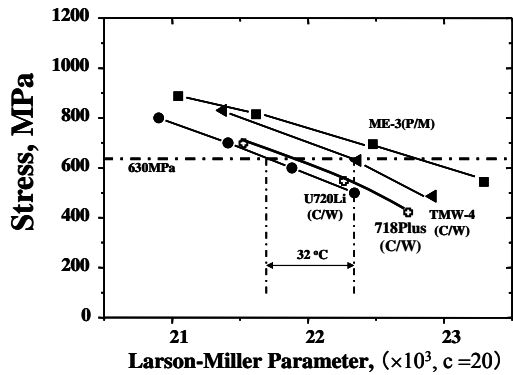
Figure 8. Larson-Miller plot of stress rupture life of TMW-4 alloy in comparison with reported 718Plus, U720Li and ME3 alloys.

from 480 MPa to 830 MPa. Time to 0.2% creep strain at 725 °C/630 MPa was about 6 times longer for TMW-2 and was about 12 times longer for TMW-4 alloy compared with TMW-1 (U720Li), which was about 32°C and 50 °C higher respectively compared with the current commercial U720Li alloy by Larsen-Miller Parameter (Figure 7a). The rupture life of TMW-4 at 725 °C/630MPa is about 6 times compared with that of TMW-1 (U720Li). Creep-rupture comparisons of TMW-4 alloy with fine grain size (10μm) to reported C&W 718Plus [18], C&W U720Li alloy [19], and the latest P/M disk alloy, ME3(Rene 104) [20], by a Larsen-Miller Parameter plot are shown in Figure 8. Temperature gains of about 32 °C and 29°C by TMW-4 alloy compared with U720Li alloy and 718Plus respectively could be expected. Meanwhile, at low temperature and high stress, the creep resistance of TMW-4 alloys was comparable with that of ME3 (Rene 104) alloy.

Microstructures Associated with Creep

The microstructures of TMW-4 crept to rupture at 725 °C/ 630 MPa show that coarsening grains occurred in some areas whereas a mixture of inter-granular and trans-granular fracture existed (Figure 9a). The γ' precipitates were sheared by deformation bands as indicated by the arrows in Figure 8b.
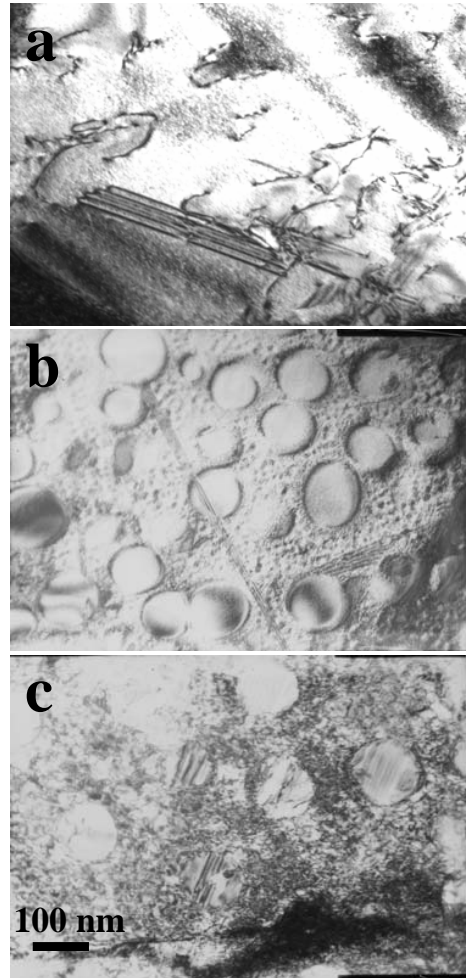

Figure 10. TEM micrographs showing the deformation substructures the samples crept to 2% at 725 °C/630 MPa in (a) TMW-1; (2) TMW-2 and (c) TMW-4 alloys.
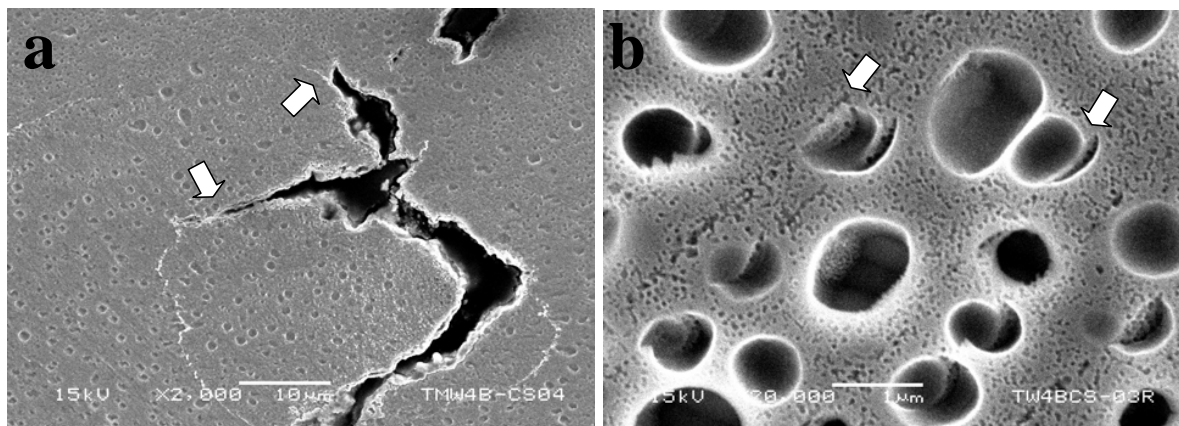

Figure 9. SEM images showing the microstructures of the TMW-4 alloys crept to rupture at 725 °C/630 MPa.

The dislocation structures corresponding to the specimens of TMW-1 and TMW-4 alloys deformed at 725 °C/630 MPa to a strain of 2 pct (about 40-hours creep time for TMW 1 and 240-hours creep for TMW-4) are shown in Figure 10. In TMW 1 alloy, a few of anti-phase boundary (APB)–coupled dislocation pairs and stacking faults (SFs) existed in both the γ and the γ' precipitates (Figure 10a). The γ' precipitates were sheared by the slip of APBs and SFs. Evidence of dislocation bypass through bowing and climbing has not been observed. In TMW-2 and TMW-4 alloys, no APB-coupled dislocation pairs were observed and the γ' precipitates were sheared by the slip of SFs (Figures 10b and 10c). Higher density of SFs in TMW 4 than in TMW 2 alloys was observed. Meanwhile, no coarsening of the γ' precipitates was investigated in all observed samples.

Discussion

A striking result of this investigation is the remarkable superior strength and higher creep resistance of TMW 4 alloy compared with U720Li (TMW-1) even both of these alloys have the same γ' volume fraction and grain size (Table 1 and Figure 4). As we known, the strength of this kind of two-phase alloys depends on a number of interrelated micro-structural parameters including the volume fraction, particle size, distribution, and chemical characteristics of the γ' precipitates that present in a disordered solid-solution face centered cubic γ matrix, along with the content of solid solution hardening elements and the grain size. It has been reported that cobalt content has little direct influence on the tensile strength of a Ni-base disc alloy Udimet 700 [15]. Inspection of Table I shows that the significant differences between the tested alloys are that TMW 2 and TMW 4 have more cobalt and titanium, but less Cr. Meanwhile, the contents of main solid solution hardening elements, like Mo and W, are lower in TMW-2 and TMW-4 alloys than that in U720Li (TMW-1) alloy (Table I). Therefore, the strength improvement may be due solely to the precipitate hardening. Comparisons of the tensile properties of the TMW alloys with the same volume fraction of γ' precipitate (TMW-1 and TMW-4) suggest that the precipitates, $(Ni,Co)_3(Al,Ti)$, in Ni-Co base alloys may supply more strength hardening effect with a increase of the Ti content in the precipitates [15, 21]. However, with the increased volume fraction of γ' precipitate, the creep resistance of TMW-2 alloy was lower than TMW-4 alloy at all tested conditions, which implies that the strength hardening effect of γ' precipitates in TMW alloys is influenced greatly by the composition. The deformed microstructure in the crept specimens (Figure 10) indicates that the SFs became more energetically favorable in TMW-2 and TMW-4 alloys. Empirically, the creep rate has been shown to vary as $(\Gamma/Gb)^3$ where Γ is the stacking fault energy, G the shear modulus and b the burgers vector [22]. Even the dependence of creep rate upon stacking fault energy has so far remained unexplained [23, 24], this empirical formula may hold in the case of TMW alloys, where TMW 4 alloys with lower SFs energy or higher SF density may have lower creep rate than that of TMW 2 alloys. A systematic and broader study is needed to fully understand the deformation mechanism and the effects of key elements, such like Co, Ti and Al, on the mechanical properties of the matrix and precipitate in these Ni-Co base superalloys.

Full-scale Pancake Manufacturing

As expected, new Ni-Co-base TMW alloys showed superior tensile strength and creep resistance at temperatures up to 750 °C than U720Li alloy and had good fabrication ability. For evaluated the process ability and the quality of TMW alloys at manufacturing level, some full-scale pancakes (400 mm in diameter and 65 mm in thickness) based on TMW-2+4Co alloy were manufactured by conventional C&W processing route including triple-melting, billet making, pancake forging and ultra-sonic inspection in Mitsubishi Materials Corporation.

Triple Melting

Two ingots were produced successfully through primary vacuum induction melting (VIM), electro-slag re-melting (ESL) and vacuum arc re-melting (VAR). Each of the VAR ingots was 500 mm in diameter and weighted about 1.7 ton. The surfaces of the VAR ingots were checked carefully and no evidences of instability during the re-melting process were found. No defect such as cracks, pores or heavy segregations was observed. Chemical analysis showed that most of the element compositions were well controlled to the nominal values, although Al and Ti contents slightly varied within the VAR ingots, which may be caused by ESR process.

Billet making

To select an optimum forging condition for the billet processing, we checked the tensile properties of TMW-2+4Co by Gleeble testing. The results showed that the high area reduction over 30% could be obtained in temperatures ranging from 1000 to 1150°C (below the γ' solvus temperature), while poor ductility was obtained in all testing conditions above the γ' solvus temperature. According to these results, the VAR ingots homogenized below the γ' solvus temperature were forged successfully and the final billets with 200 mm in diameter were obtained without heavy surface cracks. No defect such as cracks or segregations was investigated in the billets, and the grain structure was fine and homogeneous enough for further processing.

Pancake Forging

In this trial, three pancake disks were forged at the same deformation condition under 1100°C, 1120°C and 1140°C. All of
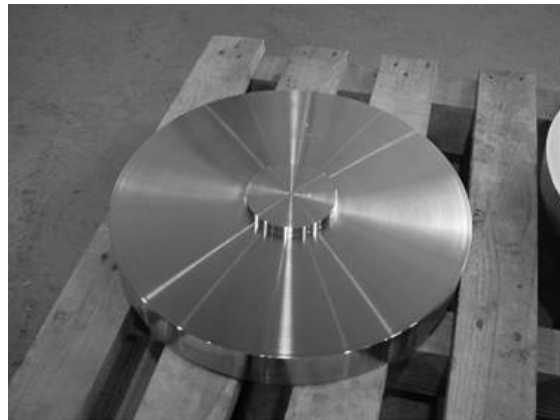

Figure 11. Pancake forged from a TMW alloy.

three pancakes were forged successfully without crack occurrences during process. Figure 11 shows the appearance of a forged pancake. The homogeneous grain structure with the average grain size of about 10μm was obtained in the pancake forged at 1100°C, while some coarsening grain was observed at the center of the pancakes forged at higher temperatures. This trial supplies very useful experience for processing other TMW alloys in manufacturing level.

Summary

In this paper, a series of Ni-Co-base disk alloys, named TMW alloy, were designed and evaluated based on their work abilities, phase constitutes and thermal stabilities, and mechanical properties. The results show that some new Ni-Co-base disk alloys are full of promise to be fabricated by a normal C&W process route and have superior phase stability, strength and creep resistance compared with U720Li (TMW-1) alloy. For tested TMW alloys with γ/γ' two-phase structure, TMW-4 alloy demonstrated an ultimate tensile strength up to 1700 MPa at room temperature, 1140 MPa at 750 °C and 0.2% proof strength at a level above 950 MPa at 750 °C. At our targeted temperature and stress (725°C/630MPa), time to 0.2% creep strain was about 12 times longer and creep-rupture life was about 6 times longer for TMW-4 alloy compared with that of U720Li alloy (TMW-1). Therefore, a temperature gain of about 32 °C by TMW-4 alloy compared with U720Li alloy could be expected. Based on a TMW alloy, a full-scale pancake (400 mm in diameter and 65 mm in thickness) with the average grain size of about 10μm were manufactured successfully by conventional C&W processing route including triple-melting, billet making and pancake forging.

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