multilayers'13

madrid

october 2013

Welcome to the Nanoscale Multilayers Madrid 2013!!!

An International Workshop on the Mechanical Behavior of Nanoscale Multilayers

October 1-4, 2013 • Madrid, Spain

About your Registration

Full Conference Registration and Student Registration include coffee breaks, conference luncheons, welcome reception and access to the technical sessions, the guided visit to Toledo, the conference banquet in Toledo and the guided visit to the Old Madrid.

Guests may purchase tickets for the welcoming reception and the conference luncheons at the registration desk. Tickets are $20 (Guided Visit to Old Madrid) and $85 (guided visit to Toledo and conference banquet).
Badges must be worn to gain access to the technical sessions and social functions.

Note to speakers

Presentations must be loaded in the central computer room 15 minutes before the start of the corresponding session.

Note to poster presenters

Posters must be placed on the available panels on Tuesday morning.

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CALENDAR OF EVENTS

Tuesday, October 1 *	Time	Location
Registration	8:00 to 8:45 am	Main Hall
Conference Opening	8:45 to 9:00 am	Auditorium
Session1: Nanoscale Multilayers I	9:00 to 11:00 am	Auditorium
Poster Session & Coffee Break	11:00 to 11:30 am	Main Hall
Session 2: Nanoscale Multilayers II	11:30 to 1:10 pm	Auditorium
Lunch	1:10 to 2:00 pm	Main Hall
Session 3: Radiation resistant materials I	2:00 to 3:40 pm	Auditorium
Poster Session & Coffee Break	3:40 to 4:10 pm	Main Hall
Session 4A: Characterization of multilayers I	4:10 to 5:10 pm	Auditorium
Session 4B: Radiation resistant materials II	4:10 to 5:10 pm	Seminar Room
Welcome reception visit to IMDEA Materials Institute	5:10 to 6:30 pm	

Wednesday, October 2 *	Time	Location
Session 5: Nanoscale multilayers III	9:00 to 11:00 am	Auditorium
Coffee Break	11:00 to 11:30 am	Main Hall
Session 6: Stresses in thin-fims and multilayers I	11:30 to 1:10 pm	Auditorium
Lunch	1:10 to 2:00 pm	Main Hall
Session 7: Nanomechanical testing	2:00 to 3:20 pm	Auditorium
Coffee Break	3:20 to 3:50 pm	Main Hall
Session 8A: Radiation resistant materials III	3:50 to 5:05 pm	Auditorium
Session 8B: Characterization of multilayers II	3:50 to 5:05 pm	Seminar Room
Guided visit to the Old Madrid	7:00 to 8.30 pm	
"Tapas" Dinner on Your Own		

Thursday, October 3	Time	Location
Session 9: Stresses in thin-fims and multilayers II	9:00 to 11:00 am	Auditorium
Coffee Break	11:00 to 11:30 am	Main Hall
Session 10: Nanoscale multilayers IV	11:30 to 1:30pm	Auditorium
Lunch	1:30 to 2:30 pm	Main Hall
Guided visit to Toledo	2:30 to 7:30 pm	
Conference Banquet in Toledo	8:00 to 10:00 pm	

Friday, October 4	Time	Location
Session 11: Nanoscale Multilayers V	9:00 to 11:00 am	Auditorium
Coffee Break	11:00 to 11:30 am	Main Hall
Session 12a: Characterization of multilayers III	11:30 to 12:45 pm	Auditorium
Session 12b: Processing I	11:30 to 12:45 pm	Seminar Room
Lunch	12:45 to 1:45 pm	Main Hall
Session 13a: Nanoscale Multilayers VI	1:45 to 3:00 pm	Auditorium
Session 13b: Nanoscale Multilayers VII	1:45 to 3:00 pm	Seminar Room
Conference Closure	3:00 to 3:15 pm	Main Hall

***On Tuesday and Wednesday afternoon, the possibility exists to carry out demonstrations on Hysitron scientific equipment. Talk to Hysitron representatives for details.**

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***On Tuesday and Wednesday afternoon, the possibility exists to carry out demonstrations on Hysitron scientific equipment. Talk to Hysitron representatives for details.**

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BASEMENT


TOILETS
ELEVATORS
TOILETS
STAIRS
ELEVATORS
MAIN HALL
SEMINAR ROOM
HALL
AUDITORIUM
ELEVATORS
STAIRS
TOILETS

About the Conference Location

Computer/Network Facilities

IMDEA Materials Institute has guest wireless connection available in the whole building.
Wifi: IMDEA-GUEST
Password: ImdeaInternet

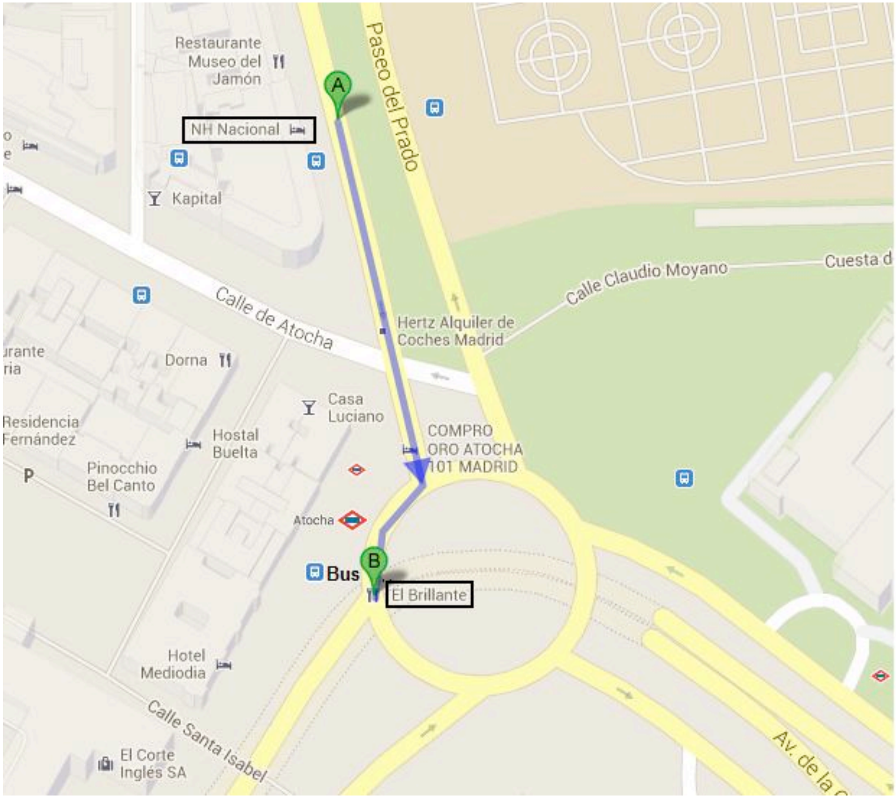
How to arrive to IMDEA Materials Institute

IMDEA Materials Institute is located at the Scientific and Technological Park of the Polytechnic University of Madrid in Tecnogetafe.
Address:
Calle Eric Kandel, 2
28906 Getafe

The conference provides **shuttle** services to and from the hotels to IMDEA Materials Institute. Please see below the shuttle schedule corresponding to each day of the workshop. The traveling time is approximately 30-40 mins. The bus stop near NH Nacional is shown in the map below.
It is highly recommended that all participants use this shuttle service, as it is the most convenient way to get to the venue.

Alternative ways to arrive to IMDEA Materials Institute include taxi (approximately 25-35 euro, travelling time from Atocha: 30 min, see detailed directions and map below) and public transport (traveling time from Atocha: 1h30min, see directions below).

If you have any question about how to reach IMDEA Materials please contact Mariana Huerta at +34 915493422.



Map showing the location of the bus to be taken near NH Nacional. For the other hotels the bus will be located in front of the hotel´s entrance.

SHUTTLE SCHEDULE FROM/TO THE HOTELS TO/FROM THE CONFERENCE VENUE

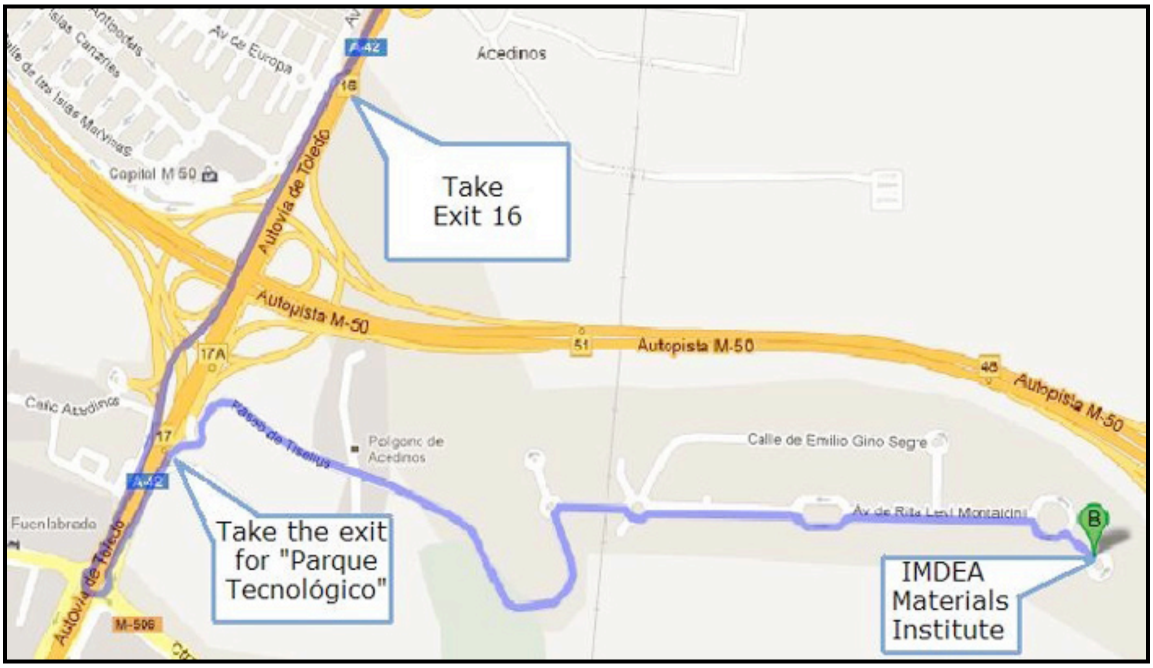
Hotel to IMDEA Materials Institute	IMDEA Materials Institute to Hotels, Old Madrid & Toledo
Tuesday , Oct 1st 7:45 am near NH Nacional* 7:45 am in front of NH Sur 8:10 am in front of NH Hesperia 12:15 pm near NH Nacional* 12:40 pm in front of NH Hesperia	**Tuesday , Oct 1st** 2:00 pm to NH Hesperia & NH Nacional 6:40 pm to NH Hesperia & NH Sur 6:40 pm to NH Nacional
Wednesday, Oct 2nd 8:15 am near NH Nacional* 8:15 am in front of NH Sur 8:40 am in front of NH Hesperia 12:15 pm near NH Nacional* 12:40 pm in front of NH Hesperia	**Wednesday, Oct 2nd** 2:00 pm to NH Hesperia & NH Nacional 5:15 pm to NH Hesperia and NH Sur 5:15 pm to NH Nacional**
Thursday, Oct 3rd 8:15 am near NH Nacional* 8:15 am in front of NH Sur 8:40 am in front of NH Hesperia 1:30 pm near NH Nacional*** 2:00 pm in front of NH Hesperia***	**Thursday, Oct 3rd** 2:30 pm to the guided visit to Toledo
Friday, Oct 4th 8:15 am near NH Nacional* 8:15 am in front of NH Sur 8:40 am in front of NH Hesperia	**Friday, Oct 4th** 3:30 pm to NH Hesperia & NH Sur 3:30 pm to NH Nacional

(*) The bus stop of the NH Nacional will be in front of the cafeteria “El brillante” (see map in the previous page)

(**) A bus will leave near NH Nacional (in front of the cafeteria “El brillante” - see map in the previous page) at 6:30 pm to go to the Plaza de la Villa, where the visit to the old Madrid will start.

(***) A bus will leave near NH Nacional & NH Hesperia in order to take accompanying persons who want to come to the visit to Toledo to IMDEA Materials Institute, where the tour will start.
Near NH Nacional 1:30 pm (The bus stop will be in front of the cafeteria “El brillante” - see map in the previous page)
From NH Hesperia 2:00 pm

Map (to be printed and shown to the driver if taking a taxi)



By taxi

From Atocha Train Station (near NH Sur and NH Nacional)

- Head southeast toward Paseo de la Infanta Isabel (130 m)
- Exit the roundabout onto Paseo de la Infanta Isabel (42 m)
- Take the ramp onto Paseo de la Infanta Isabel (260m)
- At the roundabout, take the 3rd exit onto Plaza del Emperador Carlos V (280 m)
- Continue onto Paseo de Sta María de la Cabeza (1.9 km)
- Continue onto A-42 (signs for Toledo/R-5/Badajoz/Plaza de Fernández Ladreda) (13.2 km)
- Take exit 16 toward M-506/Pinto/Fuenlabrada (1.1 km)
- Merge onto Autovía de Toledo (200 m)
- At the roundabout, take the 4th exit and stay on Autovía de Toledo heading to M-50/Madrid (400 m)
- Keep right at the fork, follow signs for A-42/Getafe/Madrid (68 m)
- After 68 meters turn right immediately following the sign "Parque Científico Tecnológico *TECNOGETAFE*".
- Continue onto Paseo de Tiselius, leaving Los Angeles school on your left until you reach a roundabout (entrance to Tecnogetafe) (1.2 Km)
- Take the first exit right and continue straight along the main avenue of the Technology Park (Avenida Rita Levi Montaicini) until the end of the avenue where you will reach a roundabout (1.0 Km)
- In the last roundabout take the first exit right onto Calle Eric Kandel, where IMDEA Materials Institute is located.

From Barajas Airport (Terminals T1, T2 y T3):

- Join the M-14 via the ramp on the left towards Madrid /Av.América/A-2/M-40/A-3/A-4/A-5.
- Take the left exit towards M-40/M-40 R-3/A-3/R-4/A-4/A-42/R-5 direction until you reach Exit 25 to take the A-42 towards Toledo. Keep driving along A42 highway (13.2 Km)
- Take exit 16 toward M-506/Pinto/Fuenlabrada (1.1 km)
- Merge onto Autovía de Toledo (200 m)
- At the roundabout, take the 4th exit and stay on Autovía de Toledo heading to M-50/Madrid (400 m)
- Keep right at the fork, follow signs for A-42/Getafe/Madrid (68 m)
- After 68 meters turn right immediately following the sign "Parque Científico Tecnológico *TECNOGETAFE*".
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- In the last roundabout take the first exit right onto Calle Eric Kandel, where IMDEA Materials Institute is located.

From Barajas Airport (Terminal T4):

- Go straight until you reach a fork where you must keep right to follow the signs to Madrid.
- Continue straight to take at a junction Eje-Aeropuerto/M-12 left towards Madrid/M-11/M-40 (In this highway will have to pay a toll).
- Take the M-40/M-40 Highway exit, continue in this sense and get to the fork, bear right and follow signs for M-40/E-90/A-2 / and merge onto highway Zaragoza/R-3/A-3/R-4/A-4/A-42/R-5 M-40/M-40.
- Take exit 25 to merge onto A-42 toward Toledo (10.5 Km)
- Take exit 16 toward M-506/Pinto/Fuenlabrada (1.1 km)
- Merge onto Autovía de Toledo (200 m)
- At the roundabout, take the 4th exit and stay on Autovía de Toledo heading to M-50/Madrid (400 m)
- Keep right at the fork, follow signs for A-42/Getafe/Madrid (68 m)
- After 68 meters turn right immediately following the sign "Parque Científico Tecnológico *TECNOGETAFE*".
- Continue onto Paseo de Tiselius, leaving Los Angeles school on your left until you reach a roundabout (entrance to Tecnogetafe) (1.2 Km)
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- In the last roundabout take the first exit right onto Calle Eric Kandel, where IMDEA Materials Institute is located.

Public Transport (not recommended)

You should take “Cercanias” line C-4 (in Sol or Atocha) heading to Parla and stop at the Metro/Cercanías station of *Getafe Central* (Estimated time from Atocha Station: 25 minutes, price 1.50€) and then catch the Tecnogetafe shuttle service (Estimated time 20 minutes, price free). The first stop of the bus service is located about 50 meters on the right/down as you exit the station (The bus stop is shown in the picture below). The shuttle service is run by the bus company *M. Forest* (in the top front of the bus you will see a sign of *Tecnogetafe*). The last stop of this shuttle is right in front of IMDEA Materials Institute.

Monday to Thursday

Getafe Central - IMDEA

Departure	Arrival
7:30 AM	7:50 AM
8:10 AM	8:30 AM
8:55 AM	9:20 AM
9:40 AM	10:00 AM
1:50 PM	2:10 PM
2:30 PM	2:50 PM
3:10 PM	3:30 PM
5:35 PM	5:55 PM
6:15 PM	6:35 PM
6:55 PM	7:15 PM
7:35 PM	7:55 PM

IMDEA - Getafe Central

Departure	Arrival
7:50 AM	8:10 AM
8:30 AM	8:55 AM
9:20 AM	9:40 AM
1:30 PM	1:50 PM
2:10 PM	2:30 PM
2:50 PM	3:10 PM
3:30 PM	3:50 PM
5:15 PM	5:35 PM
5:55 PM	6:15 PM
6:35 PM	6:55 PM
7:15 PM	7:35 PM
7:55 PM	8:15 PM

Friday

Getafe Central - IMDEA

Departure	Arrival
7:30 AM	7:50 AM
8:10 AM	8:30 AM
8:55 AM	9:20 AM
9:40 AM	10:00 AM
1:50 PM	2:10 PM
2:30 PM	2:50 PM
3:10 PM	3:30 PM
3:50 PM	4:10 PM

IMDEA - Getafe Central

Departure	Arrival
7:50 AM	8:10 AM
8:30 AM	8:55 AM
9:20 AM	9:40 AM
1:30 PM	1:50 PM
2:10 PM	2:30 PM
2:50 PM	3:10 PM
3:30 PM	3:50 PM
6:00 PM	6:20 PM

Programming Notes

Technical Sessions

The Technical program will begin Tuesday, October 1st and conclude Friday, October 4th

The conference will be organized to include plenary sessions on the different topics of the conference. Each session will start with a keynote lecture given by an internationally-recognized expert on a predetermined topic and will be followed by oral presentations on the same topic. Poster sessions will be scheduled between oral presentation sessions so graduate students, postdoctoral fellows, and young researchers can present their work. Time will be included in the program for ad hoc meetings, informal discussions, and/or outings to local cultural attractions. This format is designed to promote dialogue and enhance the exchange of ideas among the participants.

All participants are expected both to attend the entire conference and to contribute actively to the discussions. The conference will take place in an informal atmosphere.

Keynote Speakers:

- **D. Bahr**, *Washington State University, USA*
- **S. Bull**, *Newcastle University, UK*
- **R. Dauskardt**, *Stanford University, USA*
- **M. Demkovicz**, *Massachusetts Institute of Technology, USA*
- **P. Ho**, *University of Texas in Austin, USA*
- **A. Misra**, *Los Alamos National Laboratory, USA*
- **D. Pantuso**, *Intel Corporation, USA*
- **R. Schwaiger**, *Karlsruhe Insitute of Technology, Germany*

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Photography Notice

By registering for the conference, all attendees acknowledge that they may be photographed by IMDEA Materials Institute personnel while at events and that those photos may be used for promotional purposes.

Organizing Committee

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- **J.M. Molina-Aldareguia**, IMDEA Materials Institute
- **I. Martin-Bragado**, IMDEA Materials Institute
- **J. LLorca**, IMDEA Materials Institute & Polytechnic University of Madrid

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- **E. Zschech**, Fraunhofer Institute for Non Destructive Testing, Germany

GUIDED VISIT TO THE OLD MADRID <u>**Wednesday, October 2**</u>

The area known as *Austria's Madrid*, or the Madrid of the Habsburg, after the Austrian royal dynasty who reigned in Spain until 1700, is the oldest section of the city, and it is full of historical attractions. You will feel the charm of old medieval renaissance Madrid in the narrow quiet streets around Plaza de la Villa, the Convent of las Carboneras, la Basilica de San Miguel and, of course, the Plaza Mayor where you can find the Cava Baja street, which is very lively all nights of the week. Again, at the calle Mayor, there is the Mercado de San Miguel, a cute 1913 iron market. We will stop at the Cathedral of la Almudena, and will finish our journey at the Plaza de Oriente, in the vicinity of the Royal Palace.

- A bus will leave at 5:15 pm from IMDEA Materials to the NH Hesperia. It will wait there for 10 minutes for those who want to leave personal things. Afterwards the bus will go near NH Nacional.

- There will be a **bus** in front of the cafeteria "El brillante" (near **NH Nacional -** see map in the **page VIII**) at 6:30 pm to go to the **Plaza de la Villa**, where the visit to the old Madrid will start

GUIDED VISIT TO TOLEDO <u>Thursday, October 3</u>

TOLEDO, museum - city of great artistic beauty, was the capital of the country and one of the oldest cities in Europe. Toledo has wonderful examples of architecture from different cultures, Muslim, Jewish and Catholic. It was declared World Heritage by UNESCO. We will walk around the city to admire its streets and monuments, will visit the cathedral (classic gothic building built in white stone, with a magnificent choir, vestry, chapter houses and cloister), the church of Santo Tomas ("Burial of Lord of Orgaz", masterpiece of El Greco), the synagogue of Santa Maria La Blanca (a mudejar construction of the XII century) and the church of San Juan de los Reyes (built in commemoration of the Battle of Toro in the XV century with a unique gothic – flemish style).
After the tour, the **conference banquet** will take place in the Parador of Toledo.

- A bus will leave from NH Nacional & NH Hesperia to IMDEA Materials in order to take accompanying persons who want to come to the visit to Toledo, where the tour will start.
- Near NH Nacional 1:30 pm (The bus stop will be in front of the cafeteria "El brillante" - see map in page VIII)
  From NH Hesperia 2:00 pm
- The bus will leave IMDEA Materials to Toledo at 2:30 pm
- After the Conference Banquet the bus will return to the NH Nacional, NH Sur & NH Hesperia

Registration

Tuesday AM — Room: Main Hall
October 1, 2013 — Location: IMDEA Materials Institute

8:00 - 8:45 AM Conference Registration

Conference Opening

Tuesday AM — Room: Auditorium
October 1, 2013 — Location: IMDEA Materials Institute

8:45 - 9:00 AM Conference Opening
Session Chairs: J. Llorca; I. Martin-Bragado; J. Molina-Aldareguia

Session 1: Nanoscale Multilayers I

Tuesday AM — Room: Auditorium
October 1, 2013 — Location: IMDEA Materials Institute

Session Chair: Javier Llorca, Polytechnic University of Madrid/IMDEA Materials Institute

9:00 AM Keynote
Nanoscale Multilayers in Nanoscience and Bioscience Technologies: *Reinhold Dauskardt*[1]; [1]Stanford University

Reliability integrating new multi-functional films in emerging nanoscience, energy and bio technologies requires a new understanding of their adhesion and mechanical behavior. We describe our research by selecting several examples involving nanoscale multilayers with application in a number of such emerging technologies. Specifically, we discuss thin-film micromechanical testing metrologies and the relationship between composition, molecular design, and film structure on resulting mechanical and fracture behavior. We consider the effects of environmentally assisted fracture in moist and chemically active environments together with the behavior of multilayers in photovoltaic devices operating in solar UV environments. Finally, we consider the multilayer structure of the top stratum corneum layer of human skin and show how its biomechanical function can be influenced by exposures and treatments to reduce skin damage.

9:40 AM Invited
Length-Scale-Dependent Deformation and Fracture Behavior of Cu/X (X = Nb, Zr) Multilayers: *Gang Liu*[1]; Jin-Yu Zhang[1]; Lin Gao[1]; Xiang-Dong Ding[1]; Jun Sun[1]; [1]Xi'an Jiaotong University

The plastic deformation and fracture behavior of two different types of Cu/X (X = Nb, Zr) nanostructured multilayered films (NMFs) were systematically investigated over wide ranges of modulation period (MP) and modulation ratio (MR). It was found that both the ductility and fracture mode of the NMFs were predominantly related to the constraint effect of ductile Cu layers on microcrack-initiating X layers, which showed a significant length-scale dependence on MP and MR. Experimental observations and theoretical analyses also revealed a transition in strengthening mechanism, from single dislocation slip in confined layers to a load-bearing effect, when the Cu layer thickness was reduced to below about 15 nm by either decreasing MP or increasing MR. The fracture toughness of NMFs is similarly sensitive to both MP and MR. A fracture mechanism-based micromechanical model is developed to quantitatively assess the length-scale-dependent fracture toughness.

10:00 AM Invited
Mechanical Response and Internal Strains Evolution in Metallic Multilayered Composites: Laurent Capolungo[1]; Mohammed Cherkaoui[1]; *Cameron Sobie*[1]; [1]Georgia Institute of Technology

Nanolayer metallic composites materials have been the subject of several theoretical and experimental studies revealing their exceptional mechanical response, characterized by both a very high and a non-negligible ductility. Further comprehensive studies have revealed that a transition from the development of usual bulk dislocation microstructures to more architectured ones occurs when the individual layer thickness is decreased to about a few hundred nanometers. The work presented here aims at understanding the generation of these microstructures under confined layer slip and to provide a link with the development of internal strains as measured by high energy X Rays. With this, the roles of Koehler hardening to the mechanical response as well as that of interfacial dislocations to internal strains are investigated. The work is based on a discrete dislocation dynamics method coupled with a finite element method.

10:20 AM Invited
Process Design for Control of Texture Evolution and Interface Boundary Character in Bulk Cu-Nb Multilayer Nanocomposites: John Carpenter[1]; Rodney McCabe[1]; Josef Cobb[2]; Judy Schneider[2]; *Nathan Mara*[1]; Irene Beyerlein[1]; [1]Los Alamos National Laboratory; [2]Mississippi State University

Research on bulk nanolamellar Cu-Nb bimetallic composites fabricated via accumulative roll bonding has revealed several desirable properties including high strength, good ductility, and high thermal stability tied to both the interfacial density and the interfacial structure. In this talk the effects of processing parameters both within accumulative roll bonding and with friction stir processing are investigated with regards to their accompanying effects on texture, grain morphology, and strength. Neutron diffraction and electron microscopy are utilized and comparisons are made with the literature regarding single phase Cu, single phase Nb, and Cu-Nb composites. It is shown that by careful manipulation of the processing parameters, grain morphology, texture, and heterophase interfacial structure can be controlled during a severe plastic deformation synthesis pathway.

10:40 AM Invited

Nanoscale Colour Control: Protective Decorative Coatings: *Tomas Polcar*[1]; [1]Czech Technical University in Prague

In this talk we present a new design of decorative tungsten oxide coatings. The coatings were deposited with a graded refractive index by magnetron sputtering with pulsing the reactive gas. The controlled injection of the reactive gas can produced a concentration profile gradient from pure tungsten to tungsten trioxide, determining the final apparent colour of the coating. A dynamic sputtering model was built to simulate the growth of the coating during the reactive gas pulsing which was validated by direct measurement of the oxygen gradient. Finally, these results were used for an optical model allowing the optical properties of the deposited tungsten oxide layer to be described. This procedure allows the deposition of coatings with the desired colour by using the models to select the optimal oxygen pulse parameters. Proposed method can be easily applied to almost any metal/metal-oxide system to produce selected gradients of oxides or nitrides at nanoscale.

11:00 AM Break

Session 2: Nanoscale Multilayers II

Tuesday AM — Room: Auditorium
October 1, 2013 — Location: IMDEA Materials Institute

Session Chair: Reinhold Dauskardt, Stanford University

11:30 AM Keynote

Size Effects and Deformation Mechanisms in Metallic Multilayers: *Ruth Schwaiger*[1]; [1]Karlsruhe Institute of Technology (KIT)

The understanding of deformation of metallic multilayers has seen significant progress in the past decade. While nanoscale multilayers show great potential for high-strength applications, size-dependent deformation and predominant mechanisms are still under debate. Plastic deformation is governed by the constraint on dislocation motion but also strongly dependent on the interface structure between layers, which directly controls barrier strength and stability. Hindered dislocation motion inside small grains may make interfaces more active leading to interface-related deformation behaviors, e.g. grain rotation or grain boundary sliding typical of ultrafine grain sizes, possibly resulting in shear band formation. In this presentation, deformation behavior and size effects will be illustrated by means of two metallic multilayer systems with different combinations of microstructure length scale and interface structure (Cu/Au, Cu/Cr). The multilayers were investigated by indentation at different temperatures and insitu microcompression, both extremely useful methods for understanding mechanical behavior and deformation mechanisms of fine-grained structures.

12:10 PM Invited

Anisotropy of the Mechanical Response of Al/SiC Multilayers: *Javier Llorca*[1]; Jon Molina-Aldareguia[1]; Saeid Lotfian[1]; Carl Mayer[2]; Nikhilesh Chawla[2]; Amit Misra[3]; [1]IMDEA Materials Institute; [2]Arizona State University; [3]Los Alamos National Laboratory

Nanoscale multilayers are potential candidates for applications that require materials operating in extreme environments, like in thermo-solar and/or nuclear energy technologies. In the case of metal-ceramic multilayers, the combination of metallic and ceramic layers at the nanoscale has demonstrated a good combination of strength and toughness. However, these materials are inherently anisotropic due to the layered structure; however, the anisotropic response is not well understood because the strength has been traditionally measured by nanoindentation techniques. In this talk, we address this issue by comparing the mechanical behavior of Al/SiC multilayers as a function of loading direction using two different techniques: nanoindentation and micropillar compression. The former can be carried out without intensive sample preparation but analysis of the results is difficult due to the complex stress state imposed by the indenter. The latter requires the use of complex micromachining techniques but the results are easier to interpret.

12:30 PM Invited

Effect of Scale on Structure and Strength of Layered Nanostructures in Deformed Metals: *Niels Hansen*[1]; Xiaodan Zhang[1]; Xiaoxu Huang[1]; [1]Technical University of Denmark

Nanoscale multilayers in metals and alloys can be produced by various physical and chemical deposition techniques but they can also form by plastic deformation to large strain. The strengthening mechanisms are analyzed for two typical structures: (i) bulk structures with a layer thickness in the range 50 – 500 nm and (ii) surface structures with a layer thickness in the range 5 – 100 nm. The structural morphology and structural parameters are analyzed by various electron microscopy techniques. This microstructural analysis forms the basis for a discussion of strengthening mechanisms and strength-structure relationships at different length scale. Finally strength estimates are validated for bulk structures by tensile testing and for surface structures by hardness testing on the micro/nanometer scale.

12:50 PM Invited

Mechancial Behavior of Preferred Interfaces in Bulk Multilayer Nanocomposites Produced via Accumulative Roll Bonding: *Nathan Mara*[1]; John Carpenter[1]; William Mook[1]; Weizhong Han[1]; Shijian Zheng[1]; Thomas Nizolek[2]; Jian Wang[1]; Thomas Wynn[1]; Irene Beyerlein[1]; [1]Los Alamos National Laboratory; [2]University of California, Santa Barbara

In this presentation, we report on the plastic deformation mechanisms in Cu-Nb lamellar nanocomposites processed via Severe Plastic Deformation as a function of decreasing layer thickness. We utilize Accumulative Roll-Bonding (ARB) to process bulk Cu-Nb nanolamellar composites from 1 mm thick high-purity polycrystalline sheet down to layer thicknesses of 10 nm. This processing technique has the advantage of producing bulk quantities of nanocomposite material, and also exposes the interface and bulk constituents to large strains (1000's of percent). These extreme strains result in rolling textures, interfacial defect structures, and deformation mechanisms very different from those seen in nanolamellar composites grown via Physical Vapor Deposition methods. For instance, deformation twinning is observed in Cu in ARB material as opposed to PVD material. Mechanical properties and behavior will be discussed in terms of the effects of interfacial content on deformation processes at diminishing length scales, and defect/interface interactions at the atomic scale.

1:10 PM Lunch (Room: Main Hall)

Session 3: Radiation Resistant Materials I

Tuesday PM Room: Auditorium
October 1, 2013 Location: IMDEA Materials Institute

Session Chair: Ruth Schwaiger, Karlsruhe Institute of Technology (KIT)

This session has been organised with the support of the European FP7 project RADINTERFACES (Project number: 263273)

2:00 PM Keynote
Design of He-resistant Materials: from Mechanisms to Applications: *Michael Demkowicz*[1]; [1]Massachusetts Institute of Technology

Helium (He) implanted into metals causes considerable damage and may be life-limiting in future fusion reactor materials. However, it may be possible to avert He damage if materials containing stable routes for continuous He outgassing can be engineered. I will describe an effort to achieve this goal using design of interface structure in nano-layered composites. The key to this design strategy is recognizing the relationship between heterophase interface structure and the mechanisms He precipitation at interfaces. This relationship has been elucidated using closely coupled multiscale experiments and modeling and has led to the design of interfaces specifically tailored to allow for continuous He outgassing while maintaining desirable mechanical properties. Applications of this design strategy to the prevention of other forms of damage in materials operating under extreme environments will be discussed.

2:40 PM Invited
Ab initio Simulations of the Structure and Energetics of Radiation-induced Point Defects in Metallic Multilayer Interfaces: Darío Fernández-Pello[1]; César González[1]; Artur Tamm[2]; Erki Metsanurk[3]; Ekhi Arroyo[4]; María Ángeles Cerdeira[1]; Sergio Luis Palacios[1]; Santiago Cuesta-López[4]; Alvo Aabloo[2]; Mattias Klintenberg[3]; *Roberto Iglesias*[1]; [1]Universidad de Oviedo; [2]University of Tartu; [3]Uppsala University; [4]Universidad de Burgos

Recently, interfaces between incoherent transition metal nanolayers showing semicoherent heterophase structures and lattice parameter mismatch have been proposed as perfect sinks for absorption of radiation-induced defects at intersections between misfit dislocations [1]. An accurate description from first principles of the diverse types of point defects in irradiated metals appears as an essential preliminary step towards the design of efficient multilayered materials capable of self-healing from radiation damage. We have performed a systematic energetic and structural study of the different point defects and small clusters of vacancies and He atoms in the bulk and surfaces of several transition metals that have been identified as possible candidates for building these interfaces. Supercell sizes, cut-off energies and reciprocal lattice k-mesh discretizations larger than the typically used in the literature are needed to avoid unphysical, size-related effects. [1] M. J. Demkowicz, A. Misra, and A. Caro, Curr. Opin. Solid St. M. 16 (2012) 101

3:00 PM Invited
Defect Distributions and Transport In nanocomposites: A Theoretical Perspective: *Enrique Martinez Saez*[1]; Blas Uberuaga[1]; Amit Misra[1]; Alfredo Caro[1]; [1]LANL

Nanomaterials hold great promise for a number of technologically important applications, from solar energy conversion to fast ion conductors. This is particularly true for radiation tolerant materials. Here, motivated by a series of experimental studies on oxide composites, we examine the dual phase nature of the material without the interfaces acting as defect sink. We solve a reaction-diffusion model of defect evolution of simple composites under irradiation which depends on defect properties within each phase with no special behavior accounted for at the interface. We identify three regimes of steady-state defect behavior that depend on the relative thermodynamics and kinetics of the defects in the phases comprising the composite. We conclude that the evolution of irradiation-induced defects in one phase of the composite is strongly controlled by the defect properties of the other phase, offering a route to controlling defect evolution in these materials.

3:20 PM Invited
Nanoscale Structural Properties of CuW and CuNb Metallic Multilayers: From Interface Structure to Improved Radiation Damage Resistance Response: *Santiago Cuesta-Lopez*[1]; [1]University of Burgos

Nanoscale multi-layered metallic composites (NMMCs) are potential candidates to be used as the base of a wide range of components for next generations of nuclear fission and nuclear fusion technology thanks to their greatest radiation resistance and excellent mechanical properties. We describe a multiscale predictive modelling framework able to identify structural peculiarities at the interface of CuNb and CuW multilayer's. Such multiscale framework combines density functional theory (DFT) and Molecular Dynamics (MD) to characterize interface structural states at the nanoscale level. We tested the radiation damage resistance for medium energies (PKAs > 10 KeV) and the evolution of defects at the interface. Final goal will be to find appropriate strategies to optimize the properties of NMMCs in response to new challenges presented by high performance and exigent engineering.

3:40 PM Break

Session 4A: Characterization of Multilayers I

Tuesday PM October 1, 2013 — Room: Auditorium, Location: IMDEA Materials Institute

Session Chair: Gerhard Dehm, Max-Planck-Institut für Eisenforschung

4:10 PM

Deformation and Damage Behavior of ZrN/ZrxAl(1-x) N Nanolaminate Thin Films: *Kumar Yalamanchili*[1]; Emilio Jimenez Pique[2]; Magnus Odén[1]; Naureen Ghafoor[1]; [1]Linköping Univeristy; [2]Universitat Politècnica de Catalunya

Superlattices and multilayers of transition metal nitrides are known to be hard and tough, while fundamental understanding of the improved properties in relation to deformation and damage behavior of the individual layers and the role of interfaces is incomplete. We have grown multilayers of ZrN/Zr0.7Al0.3N and ZrN/Zr0.5Al0.5N by reactive magnetron sputtering on MgO(001) substrates. The thickness of ZrN layer is maintained constant at 15-nm and the second layer thickness is systematically varied from 2-nm to 30-nm. Nanoindentation results show a layer thickness dependent hardness with a maximum value of 35 GPa at a wavelength of approximately 20-nm. Fracture study results show that the multilayers are significantly tougher than single layer ZrN and ZrAlN. Toughness is observed to increase with decreasing wavelength of the multilayers. X-TEM studies of indents are used to discuss the deformation and damage modes of ZrN/ZrxAl(1-x)N multilayers and to address structure-property relations of nanoscaled multilayers.

4:25 PM

Contact Damage and Fracture Mechanisms of Multilayered TiN/CrN Coatings at Micro- and Nano-length Scales: *Joan Josep Roa*[1]; R. Martínez[2]; Emilio Jiménez Piqué[1]; R. Rodriguez[2]; L. Llanes[1]; [1]Universitat Politècnica de Catalunya. Centre de Recerca en Nanoenginyeria (CRNE); [2]Departamento de Investigación y Desarrollo, Centro de Ingeniería Avanzada de Superfície, Asociación de la Industria Navarra

In recent years TiN/CrN multilayered PVD coatings are well known for their unique properties like high hardness and good wear resistance. Large number of studies has been reported, focused on tribology characterization as well as detailed TEM analysis of interfaces. However, information on the contact damage and fracture is rather scarce. A deeper knowledge is crucial to improve the performance of these materials and to enhance the lifetime of coated systems. In this study, a systematic nanomechanical study has been conducted in two multilayer TiN/CrN systems with different period bilayer. The investigation included the use of different indenter tip geometries to induce different stress fields and damage scenarios. Special attention has been paid to analyse main damage and fracture mechanisms at the micro- and nanometric scales using AFM, FE-SEM, and FIB. It is found that the multilayer approach is quite effective on promoting crack growth resistance mechanisms; thus, damage tolerance.

4:40 PM

Nano-multilayer V/ZnO Transparent Conduction Thin Films: *Yu Shan Wei*[1]; Yen Shuo Liu[1]; Cheng Yi Liu[1]; [1]National Central University

Nano-multilayer V/ZnO transparent conductive layers were prepared by sandwiching nano V and two ZnO layers by sputtering processes. Total thickness of the V/ZnO nano-multilayer is fixed at 200nm. The electrical properties (resistivity, mobility, carrier concentration) of the V/ZnO nano-multilayer were investigated by Hall measurement. With XPS and HR-TEM analysis, we conclude that the high mobility and carrier concentration of the V/ZnO nano-multilayer attribute to the interface layer forms between the V and ZnO layers: the Zn and O atoms diffused into immobile V layer and the nano-$ZnVO_3$ layers were formed. We produced several nano-multilayer by splitting V layer into the ZnO layer, which are 1×2, 3×4, 4×5, 6×7, 9×10 (V metallic layer × ZnO layer). The 3x4 structure shows the best electrical conduction among all multi-layer samples. We will explain how the nano interfacial layers between V and ZnO contribute the carrier concentration and serve as the carrier mobility path.

4:55 PM

Mechanical Characterization of Ultra-thin Coatings: *T. Chudoba*[1]; K. Mayekar[1]; [1]ASMEC GmbH

Ultra-thin coatings below 300nm thickness are used in many industrial applications, especially in optical and electronic components. Although it is known that they often don't exhibit the same properties like bulk materials or thicker coatings; a quantitative mechanical characterization of such thin coatings is mostly neglected. In regular nanoindentation practice, the indentation depth should always be restrained to 10% of the film thickness to avoid substrate effects on the measurement. Experimentally, to achieve this 10% limit in case of ultra-thin films is difficult. The determination of hardness, modulus or yield strength of such films requires ultra-high resolution in load and displacement measurement and an extremely precise determination of the shape of the tip used for the measurements. This apparently resulted in lack of quantitative analysis in this field. Hence it is necessary to develop a method to characterize such thin films accurately irrespective of the effect from substrate material. In this paper limits for the measurement of hardness and modulus are derived and examples for the measurement of ultra-thin coatings are given. The accurate determination of the tip geometry below 50nm is illustrated. Some alternative test methods are shown which allow the measurement of ultra-thin coatings where the indentation depth is equal or even larger than the film thickness. One of the methods is based on fully elastic measurements with spherical tips and the comparison with calculated load-displacement curves for coated and uncoated substrates. The other method is using the effective indenter model to describe the stress and deformation fields during an elasto-plastic indentation with a Berkovich tip. This model uses fast analytical calculations to separate the substrate influence and to derive the pure film hardness and modulus.

Session 4B: Radiation Resistant Materials II

Tuesday PM Room: Seminar Room
October 1, 2013 Location: IMDEA Materials Institute

Session Chair: Ignacio Martin-Bragado, IMDEA Materials Institute

This session has been organised with the support of the European FP7 project RADINTERFACES (Project number: 263273)

4:10 PM
Tuning Nanostructured W Deposition Procedure to Decrease the State Stress and to Improve the Adhesion: *Nuria Gordillo*[1]; Miguel Panizo-Laiz[1]; Iván Fernandez-Martin[2]; Elena Tejado[3]; Antonio Rivera[1]; Fernando Briones[4]; José Ygnacio Pastor[3]; José Manuel Perlado[1]; Raquel Gonzalez-Arrabal[1]; [1]Instituto de Fusión Nuclear, ETSII de Industriales, Universidad Politécnica de Madrid; [2]Instituto de Energía Solar (IES), Universidad Politécnica de Madrid. Instituto de Microelectrónica de Madrid, IMM-CNM-CSIC; [3]Dept. Ciencia Mat. CISDEM, ETSI de Caminos, Universidad Politécnica de Madrid; [4]Instituto de Microelectrónica de Madrid, IMM-CNM-CSIC

W has been proposed to be one of the best candidates for PFM for both laser (IC) and magnetic (MC) confinement fusion approaches. However, works carried out up to now have identified some limitations for W which have to be defeated in order to fulfil specifications. Nowadays, the capabilities of ultra-fine grain and nanostructured materials for nuclear fusion reactor applications are being investigated. We report on the growth of nanostructured tungsten coatings with a thickness in the micro-meter range by using DC magnetron sputtering and high impulse power magnetron sputtering (HIPIMS). The aim of this work is to study the influence of deposition parameters by two different methods (continuous mode and pulse mode) on the W coatings morphology, stress state and mechanical properties.

4:25 PM
Radiation Damage Evolution in Multilayered Metallic Composites: *Aaron Dunn*[1]; Laurent Capolungo[1]; Enrique Martinez[2]; Mohammed Cherkaoui[1]; [1]Georgia Institute of Technology; [2]Los Alamos National Laboratory

Owing to the specific properties of the hetero-interfaces they contain, metallic multilayered composites materials were shown to remain particularly stable under He implantation. The proposed work presents a model for the prediction of microstructure evolution and stability of CuNb multilayered system under various irradiation conditions. The approach relies on a recently developed spatially resolved cluster dynamics method. Such development will be discussed first and will be followed by the study of a couple demonstration cases; namely frenkel pair implantation in Cu, displacement cascades in thin Fe foils. Comparison and consistency between the proposed approach and both rate theory and object oriented kinetic Monte Carlo models will be shown. Amongst other, it will be shown that the approach proposed allows for prediction of microstructure evolution over broader time scales and irradiation conditions than both rate theory and OKMC. Finally, the approach is to be applied to the case of CuNb multilayers.

4:40 PM
The Role of Grain Boundaries in the Light Species Behavior for Irradiated Nanostructured Tungsten: *Raquel Gonzalez-Arabal*[1]; Miguel Panizo-Laiz[1]; Nuria Gordillo[1]; Antonio Rivera[1]; Frans Munnik[1]; Elena Tejado[1]; Jose Ygnacio Pastor[1]; Ivan Fernandez-Martinez[1]; Jose Manuel Perlado[1]; [1]Universidad Politecninca de Madrid

One of the challenges in the design of future nuclear power plant is to develop materials capable to resist the hostile environment of a fusion reactor. Nowadays, W is assumed to be the best candidate as plasma facing material (PFM) in these reactors. However, some limitations have to be defeated i.e. the light species retention (H, He,…). In this work we study the H behavior in nanostructured (NW) W coatings as compared to coarse grain (CGW) counterpart. For this purpose resonant nuclear reaction (RNRA) experiments are carried out in NW and CGW samples implanted with (i) H at an energy of 170 keV, (ii) sequentially implanted with C (665 keV) and H (170 keV) and co-implanted with C (665 keV) and H (170 keV). Implantations were carried out at two different temperatures RT and 400ºC. The role of microstructure in radiation-induced damage and in the H behavior is discussed.

4:55 PM Invited
Electrochemical Deposition of Cu and Nb in Pyrrolidinium based Ionic Liquid for Multilayer Preparation: *Michele Mascia*[1]; Annalisa Vacca[1]; Simonetta Palmas[1]; Laura Mais[1]; Simone Rizzardini[1]; Francesco Delogu[1]; [1]Università degli Studi di Cagliari

A study on the electrochemical deposition of Cu/Nb multilayers is presented in this work. The electrodeposition tests were performed using 1-butyl-1-methylpyrrolidinium bis (trifuoromethylsulphonyl) imide as solvent. The electrochemical behavior of copper and niobium ions has been studied by cyclic voltammetry and chronoamperometry. Boron doped diamond was used as working electrode. The influence of such parameters as the temperature and supporting electrolyte has been also examined. The experimental tests were carried out under inert atmosphere, in order to avoid the presence of water. Cyclic voltammetries were performed at different temperatures and scan rates. According to the electrochemistry of the metals considered and based on the experimental results, the possible reaction path for the oxidation/reduction was proposed. Deposition tests were carried out at different potentials and the related samples were analyzed by SEM, EDX and XRD. Acknowledgments: This activity is supported by the EU-FP7, RADINTERFACES, grant agreement n. 263273.

Session 5: Nanoscale Multilayers III

Wednesday AM — Room: Auditorium
October 2, 2013 — Location: IMDEA Materials Institute

Session Chair: Michael Demkowicz, Massachusetts Institute of Technology

9:00 AM Keynote
Designing Metallic Nanolayered Composites for High Strength and Damage Tolerance: *Amit Misra*[1]; [1]Los Alamos National Laboratory

Nanolayered composites such as Cu-Nb are used as model systems to explore the interaction of interphase boundaries with defects introduced via plastic deformation or ion irradiation. The results of these experimental studies are integrated with atomistic modeling and dislocation theory to provide insight into the unprecedented combination of properties achieved in certain nanolayered composites such as ultra-high flow strengths, high plastic flow stability, high fatigue strength, high thermal stability, high sink strength for radiation-induced point defects and trapping of helium in the form of stable clusters at interfaces. The results on "bottom-up" synthesized model systems are compared with "top down" accumulative roll bonding (ARB) processed bulk Cu-Nb nanolayered composites. A quantification of the defect-interface interactions as well as the processing-interface structure relationship allows the development of materials design concepts with controlled interface structures in nanocomposites to achieve tailored response in engineering applications.

9:40 AM Invited
Elastic Modulus Mapping in Natural Multilayered Bio-Composites: *Igor Zlotnikov*[1]; Emil Zolotoyabko[2]; Peter Fratzl[1]; [1]Max Planck Institute of Colloids and Interfaces; [2]Technion

One-dimensional multilayered assembly is among the strategies employed by nature to obtain functional biomaterials resistant to catastrophic failure. Examples of multilayered architectures can be found in a variety of bio-composites: periodic arrangements of soft ultrathin organic and stiff (much thicker) mineral layers, such as biosilica in marine sponges and aragonite in the nacreous layer of mollusk shells; or purely organic plywood structures, in which the periodicity is maintained by the modulation of spatial orientation of stiff organic fibers, such as chitin or collagen. Comprehensive understanding of the mechanical behavior of such structures presents a major challenge due to the small dimensions of the constituents. In the present research, modulus mapping technique, based on nanometric Dynamical Mechanical Analysis (nanoDMA), was successfully adapted to map elastic modulus across the basal spicule of the marine sponge Monorhaphis chuni, where 35 nm thick organic layers are positioned within 50 times stiffer biosilica.

10:00 AM Invited
In-situ TEM Mechanical Testing of Nanotwinned Cu: *Daniel Kiener*[1]; Matthias Funk[2]; Zaoli Zhang[3]; Yue Liu[4]; Xinghang Zhang[4]; Chris Eberl[2]; [1]University of Leoben; [2]Karlsruhe Institute of Technology; [3]Austrian Academy of Science; [4]Texas A&M University

Nanotwinned materials show promising material properties by combining high strength with significant ductility. However, upon fatigue loading the nanotwinned structure has been observed to locally transform to an ultra-fine grained microstructure, which limits the fatigue strength. To elucidate the effects of cyclic loading on nanotwins, tensile and cyclic in-situ TEM tests were performed on sputtered nanotwinned Cu lamellas to investigate the mechanism underlying the de-twinning process. A FIB-based method was developed to mount samples on MEMS based push-to-pull devices such that the twin lamellas are viewed edge-on. Subsequently, samples were loaded in-situ in a TEM in a static or cyclic manner. We measure mechanical properties like modulus, yield strength, and fracture toughness comparable to bulk properties. At the same time, we observed the movement of groups of partial dislocations leading to a selective growth sets of twins. It is argued that this process is the mechanism underlying the de-twinning phenomena.

10:20 AM Invited
Plastic Strain Recovery in Nanocrystalline Thin Films: Yuesong Xie[1]; *Marisol Koslowski*[1]; [1]Purdue University

Materials with engineered microstructures can achieve exceptional functionality and performance not possible with bulk materials. Nanocrystalline materials exhibit high yield and fracture strengths, superior wear and radiation damage resistance. The reduction of grain size to nanometer scales leads to these enhanced properties but is also responsible for deformation mechanisms not present in coarse grained crystalline materials. One of these mechanisms is plastic strain recovery. Even though, plastic deformation is not recoverable in coarse grained materials, recent experiments in nanocrystalline materials show that plastic strain recovers after unloading. This surprising effect remains unexplained but is believed to be the result of the interaction between processes driven by thermally activated motion defects with different characteristic time and length scales. I will show an approach that incorporates thermally activated mechanisms, into large-scale numerical simulations of deformation by coupling dislocation dynamics simulations to a kinetic Monte Carlo algorithm to predict plastic strain recovery.

10:40 AM Invited
Influence of Interfacial Strength on Mechanical Properties of Lamellar TiAl Alloys: *Alexander Hartmaier*[1]; Mansour Kanani[1]; Rebecca Jansich[1]; [1]Ruhr-Universität Bochum

Titanium aluminides are promising materials for high-temperature applications. A microscopic understanding of deformation and fracture mechanisms of lamellar TiAl alloys is the basis for a systematic improvement of mechanical properties. The typical lamella spacing in TiAl alloys is between 50 to 70 nm, such that it is expected that interfacial properties play a dominant role in the mechanical behavior. Hence, we apply *ab inito* methods within the framework of density functional theory to characterize the mechanical properties of the twin grain boundaries in the TiAl phase and the phase boundaries between TiAl and Ti_3Al phases. In the next step molecular dynamics simulations with semi-empirical embedded atom method potentials are applied to study the fundamental processes occurring during plastic deformation in the lamellar structure. The results of this work allow us to draw some conclusions on the mechanical properties of lamellar structures in general.

11:00 AM Break

Session 6: Stresses in Thin-films and Multilayers I

Wednesday AM Room: Auditorium
October 2, 2013 Location: IMDEA Materials Institute

Session Chair: Daniel Pantuso, Intel Corporation

11:30 AM Keynote
Buckling of Silicon Nanostructures: Effects of Scaling and Contact Friction: *Paul Ho*[1]; Bin Li[2]; Zhiquan Luo[3]; Rui Huang[1]; [1]The University of Texas at Austin; [2]Philips Research Center; [3]Intel

As the dimension of material approaches nanoscale, the material properties will become stronger, tougher and more difficult to break. This was investigated from the buckling behavior of silicon nanostructures. Electron beam lithography was combined with anisotropic etching to fabricate silicon nanostructures with width as small as 20 nanometers. Silicon nanostructures were fabricated with width from 200 nm to 40 nm and vertical sidewalls almost atomically smooth. Nanoindentation tests were performed in AFM to observe the buckling behavior of the silicon nanolines. Under indentation, the silicon nanolines showed extraordinary toughness as they deformed elastically without plastic deformation to about 8%, approaching the theoretical limit for silicon. Buckling instability was observed at a critical load, with a displacement burst up to □1/3 of line height, which was fully recoverable upon unloading. The effects of width scaling and contact friction on the buckling behavior have been investigated and the results will be presented.

12:10 PM Invited
On the Atomic Interface Structure of a Hard Nitride Coating on MgO: *Gerhard Dehm*[1]; Zaoli Zhang[2]; [1]Max-Planck-Institut für Eisenforschung; [2]Erich Schmid Institut für Materialwissenschaft

Superhard coatings with hardness values exceeding 40 GPa require a sophisticated microstructure design in order to minimize plastic deformation while simultaneously preventing crack formation. This can be achieved by using two immiscible phases, where one phase forms a nanocrystalline network embedded into a nanometer thin second phase. In such a scenario the interface structure finally determines the resulting global mechanical behavior. This was recently predicted in literature by density functional theory for the system TiN-SiN-TiN where oscillations in the atomic structure were identified as possible weak points. With the recent progress in experimental characterization of materials by aberration corrected transmission electron microscopy interfaces can be studied at atomic resolution with picometer precision. In the present talk we report a quantitative comparison of the interface structure of VN/MgO using ab initio density functional theory, aberration-corrected high-resolution transmission electron microscopy, and electron energy-loss spectroscopy.

12:30 PM Invited
Auto-organizing ZrAlN/TiN Multilayers: *Lina Rogström*[1]; Naureen Ghafoor[1]; Mats Ahlgren[2]; Magnus Odén[1]; [1]Linköping University; [2]Sandvik Coromant

Multilayer structured coatings often exhibit higher hardness than coatings of the corresponding single phases while the temperature stability can be low due to intermixing of the sublayers at high temperatures. In multilayers where one of the sublayers consists of a metastable phase, the presence of interfaces will influence the phase separation. Here, we show that in multilayers consisting of ZrAlN layered with ZrN or TiN, phase separation of ZrAlN results in a layered structure within the ZrAlN sublayers during annealing. When TiN is used as the second sublayer, intermixing at the sublayer interfaces results in a three phase structure with smaller sublayer thicknesses compared to the as deposited coating. The enhanced layering with annealing temperature improves the mechanical properties of the coating after annealing.

12:50 PM Invited
Characterization and Processing of Multilayers using SPM with Diamond Tip: *Oleg Lysenko*[1]; Vladimir Grushko[1]; Eugene Mitskevich[1]; Volodymyr Ivashchenko[2]; Nikolay Novikov[1]; [1]Institute for Superhard Materials; [2]Frantsevich Institute for Problems of Materials Science

We present the combined technique to characterize the multilayers on the base of scanning probe microscopy with conductive diamond tip. The technique includes scanning in tunneling mode, indentation/scratching and tunneling spectroscopy of a surface. Diamond tip can mechanically remove layer by layer from a surface with scratching so the main feature of our approach is ability to characterize the separated films. All procedures are carried out with the same diamond tip. Purposely synthesized boron-doped single-crystal diamonds were used as a tip material. The probe with built-in diamond tip is directly mounted on the controlling piezo-element. Such a mechanism in contrast to the flexible cantilever is the best suitable for multifunctional utilization due to the axis of the diamond's tip does not deflect while the tip penetrates the surface. SPM includes the electromagnetic loading measurement system. Results of the nanolayered TiN/BCN coatings characterization with proposed techniques are discussed.

1:10 PM Lunch (Room: Main Hall)

Session 7: Nanomechanical Testing

Wednesday PM Room: Auditorium
October 2, 2013 Location: IMDEA Materials Institute

Session Chair: George Pharr, University of Tennessee and Oak Ridge National Laboratory

2:00 PM Invited

In situ Micro-thermomechanical Testing: Part I – Experimental Considerations: Jeffrey Wheeler[1]; Rejin Raghavan[1]; Ivo Utke[1]; *Johann Michler*[1]; [1]EMPA - Materials Science & Technology

Multilayer thin films are of significant technological interest, since they offer significant benefits over their single layer counterparts in both functionality and mechanical properties. To investigate the thermally activated mechanisms which control these benefits, nano-thermomechanical testing is required. Some issues with this sort of testing have been have been resolved in recent years using indenter and sample heating in vacuum. However, the precise determination of the temperature at the contact is still a concern in many systems. This is especially true for multi-layer coatings on dissimilar substrates. Here, this concern is addressed using thermally-instrumented indenters with calibrated apex temperatures. By utilizing this technique in situ in the SEM, a unique ability of both observation and measurement of micro-thermomechanical deformation has been achieved. In Part II of this talk, results on the behaviour of ceramic-ceramic and metal-ceramic multilayers using nanoindentation and in situ micro-thermomechanical testing using this system will be presented.

2:20 PM Invited

In-situ Micro-thermomechanical Testing: Part II - Plasticity of Multilayer Thin Films: Rejin Raghavan[1]; Jeffrey Wheeler[1]; *Johann Michler*[1]; [1]EMPA - Materials Science & Technology

Using a combined approach, comprehensive studies of the micro-thermomechanical behaviour of ceramic/ceramic and metal/ceramic multilayers synthesized by both atomic layer deposition (ALD) and magnetron sputtering will be presented. Using nanoindentation, it will be shown that ALD is a unique technique for studying the inverse Hall-Petch softening mechanism observed in nanomaterials with sub-50 nm grain size. The mechanical behavior of sputtered Cu/TiN multilayers at ambient and elevated temperatures was studied by micropillar compression. At elevated temperatures, the Cu layers with sub-500 nm thicknesses diffused to form beads on the cylindrical surfaces of the micropillars before compression. Assisted by mechanical stresses, the ‘beaded’ grains flow to form micro-whiskers or micro-crystals on quasi-static compression of the micropillars at 200 and 400C. These observations will be discussed in terms of plausible melting point depression, interfacial diffusion and super-plastic flow of the confined metallic layer.

2:40 PM Invited

Ductile Film Delamination from Compliant Substrates using Hard Overlayers: *Megan Cordill*[1]; Vera Marx[2]; Christoph Kirchlechner[2]; [1]Erich Schmid Institute of Materials Science; [2]Max Plank Instiut für Eisenforschung GmbH

Flexible electronic devices call for copper and gold metal films to adhere well to polymer substrates. Measuring the interfacial adhesion of these material systems requires the formulation of new techniques. Presented will be a new strategy to induce well defined areas of delamination measure the adhesion of Cu and Au films on polyimide substrates. The technique utilizes a hard overlayer and tensile straining to cause buckle formation. FIB cross-sectioning of the buckles reveals that the interface fails without any deformation of the polyimide substrate. Further investigation of the interface microstructure and the measurement of the film stresses present during delamination aid the development of the technique and the role the hard overlayer plays constraining the plastic deformation of the ductile layer. The new method will also allow one to examine the effects of thin adhesion layers such as Cr and Ti used to improve the adhesion of flexible systems.

3:00 PM Invited

In Situ Deformation of Metallic Interlayers: *Finn Giuliani*[1]; [1]Imperial College London

There are many interesting mechanical properties that can be achieved with metal/ceramic multilayers especially if they are defined more broadly to include structures such as MAX phases. However, the deformation mechanisms, such as hysteresis, can be complex. To simplify the problem, in this work we have concentrated on the effect of a single metal interlayer within a ceramic bi-crystal. The samples were produced by sputtering niobium on to sapphire substrates and then diffusion bonding the two coated crystals together. This allowed the production of sapphire micropillars with containing a ~50-200 nm niobium layer. These were then loaded in situ within a microbeam laue set up. By varying the layer thickness the measured strength of the pillar could be varied along with the magnitude of the hysteresis. This hysteresis was tracked by the load-displacement trace along with the movement and elongation of diffraction spots associated with the interlayer.

3:20 PM Break

Session 8A: Radiation Resistant Materials III

Wednesday PM Room: Auditorium
October 2, 2013 Location: IMDEA Materials Institute

Session Chair: Mohammed Cherkaoui, Georgia Institute of Technology

This session has been organised with the support of the European FP7 project RADINTERFACES (Project number: 263273)

3:50 PM

Density Functional Theory Study of CuNb Metal-metal Interface: *Artur Tamm*[1]; Erki Metsanurk[2]; Santiago Cuesta-López[3]; Roberto Iglesias[4]; Mattias Klintenberg[2]; Alvo Aabloo[1]; [1]University of Tartu; [2]Uppsala University; [3]Universidad de Burgos; [4]Universidad de Oviedo

Modern nuclear reactors require materials that can withstand large doses of radiation for a long period of time. Nanoscale metallic multilayer composites (NMMC), such as Cu/Nb, have shown an improved radiation damage tolerance. Experimental study of NMMC materials is timeconsuming and difficult, therefore multiscale simulation methods have to be applied. Recently many atomistic simulations based on empirical potentials have been performed to understand the influence of interface structure energetics on radiation damage tolerance of CuNb composites. Since the potentials used are fitted to only a limited set of properties, thus transferability might interfere with the predictions arising from such calculations. In this study we show the CuNb lowest energy interface as predicted by current potentials and DFT calculations to assess whether the MD predicted interfaces are energetically favorable. Thise CuNb interface structure can be used in future atomistic simulations such as irradiation studies or mechanical response simulations CuNb NMMC material.

4:05 PM

DFT Study of Helium Mobility Inside a Metallic System: *César González*[1]; Angeles Cerdeira[1]; Darío Fernández-Pello[1]; Sergio Palacios[1]; Roberto Iglesias[1]; [1]Universidad de Oviedo

Transmutation reactions are expected to produce Helium nuclei inside a fusion reactor. These particles can be incorporated degrading the first layers of the wall. Multilayers of Cu/Nb(W) have been proposed as a protective coating of the plasma-facing structural materials. For that reason, a lot of work has been done in the understanding of He trapping inside the bulk and in the interfaces using: from density functional theory (DFT) to finite elements, concluding that: He atoms forms bubbles in the metallic vacancies and/or interfaces. We present, a complete study of He-bubbles formation and mobility inside a metallic Cu/Nb/W bulk using the DFT formalism. An energetic analysis shows the preferential paths chosen by the moving He atom, between interstitial positions and (n)-vacancies. Due to the restrictions regarding system sizes of DFT techniques, small clusters of only four or five helium atoms can be studied inside up to a tetra-vacancy.

4:20 PM

A Coupled Diffusion-phase Field-crystal Plasticity Framework to Study Creep Response of Irradiated Nano Materials: *Aurelien Villani*[1]; Esteban Busso[1]; Samuel Forest[1]; Benoît Appolaire[2]; [1]Mines Paristech; [2]ONERA

Some of the most deleterious degradation mechanisms in high temperature polycrystalline materials are those caused by the diffusion of point defects through either the crystal lattice or the grain boundary regions, and by their interaction with other lattice defects such as dislocations and grain boundaries. They can lead to cavitation, local swelling and creep due to the formation of point defect clusters, amongst others. In an irradiated environment, nano-materials are expected to show self-healing properties, by evacuating the irradiation produced defects, such as vacancies and helium atoms, through dislocation, grain boundary and interface diffusion. A coupled diffusion-crystal plasticity framework is linked to the phase field method to study creep in irradiated nano-layered materials. Simulation results involving the development of radiation damage (i.e. stable voids or cavities) as well as creep deformation during irradiation of a polycrystal are presented and discussed.

4:35 PM

Empirical Interatomic Potential for Studying Radiation Damage and He in Cu/W Multilayers: *Erki Metsanurk*[1]; Artur Tamm[2]; Roberto Iglesias[3]; Mattias Klintenberg[1]; Alvo Aabloo[2]; César González[3]; [1]Uppsala University; [2]University of Tartu; [3]Universidad de Oviedo

Modeling multilayered materials in multi-scale approach is a demanding task as the most accurate, quantum mechanics based methods, such as Density-Functional Theory (DFT), are limited to hundreds of atoms in length and femtoseconds in time scale which therefore cannot directly provide input to models at the top of the multi-scale.An indirect approach to acheive that is to fit the relevant data obtained from DFT calculations to a simpler model which allows to extend the length and time scales by several orders of magnitude while keeping acceptable precision. We present a semi-empirical, embedded atom method based interatomic potential for studying Cu/W/He system using molecular dynamics. Emphasis is put on correct representation of equilibrium thermodynamics, point defect formation and migration energies for various configurations and defect binding energies. This allows the potential to be used for analyzing collision cascades and subsequent defect migration and clustering in Cu/W multilayers.

4:50 PM

Study of Helium-bubbles Nucleation at Interfaces in Cu/Nb Multilayer Materials: *Laura Agudo-Merida*[1]; Ignacio Martin-Bragado[1]; [1]IMDEA Materials Institute

In this work, we focus on studying the atomistic mechanisms that affect helium mobility and solubility at Cu/Nb interfaces at different depths. Experiments show that near the He implantation surface, Cu/Nb multilayers do not form He-bubbles as easily as at some depth. To elucidate why apparently same He concentrations not always produce similar number of He-bubbles we use an Object Kinetic Monte Carlo simulator (MMonCa) with a previously validated parametrization. In our simulation box we introduce Helium ions (with $10^17/cm^2$), interstitials and vacancies, at room temperature during 3 hours. Then, we observe that near the surface, the higher vacancy concentration reduces the number of He reaching the interface below the critical value needed to begin the He-bubble nucleation. Consequently, our simulation results allow us to conclude that vacancy concentration is a basic parameter when trying to explain the evolution of defects in He-irradiated multilayer materials.

Session 8B: Characterization of Multilayers II

Wednesday PM — Room: Seminar Room
October 2, 2013 — Location: IMDEA Materials Institute

Session Chair: Steve Bull, Newcastle University

3:50 PM

The Effect of Vanadium Content and Temperature on Stick-Slip Phenomena Under Friction of CrV(x)N Coatings: *Alex Laikhtman*[1]; L. Rapoport[1]; V. Perfiliev[1]; A. Moshkovich[1]; I. Lapsker[1]; [1]Holon Institute of Technology (HIT)

The aim of this work is to investigate the stick-slip phenomenon under friction of CrV(x)N (x = 0 %, 12 %, 27 %, 35 %) coatings at room and high temperatures. This study is mostly concentrated to CrV(0)N and CrV(35)N coatings. The effects of vanadium (V) content and the temperature on the friction and stick-slip parameters for the contact pair: coating-ceramic ball were analysed. The amplitude (difference between the maximal and minimal values of the friction force), the static force (at the beginning of the friction) and the kinetic friction force (average value in the steady state) were chosen for the description of the stick-slip phenomenon. The surface morphology, the structure and the mechanical properties of coatings were analyzed by SEM, AFM, and XRD techniques.

4:05 PM

3D STEM XEDS Approach for the Chemical Analysis of Multilayered Structures: *Dominique Delille*[1]; Oleg Lourie[1]; Daniela Sudfeld[1]; Bert Freitag[1]; Arda Genç[2]; Huikai Cheng[2]; Jonathan Winterstein[2]; Lee Pullan[2]; [1]FEI Company; [2]FEI Company US

Complexity of the multilayered structures needed in the continuously shrinking 2D/3D nano-devices architecture has been driving the development of 3D STEM tomography techniques for some time in Materials Science meanwhile 3D chemical information was still missing. Non-monotonic dependence of EELS signal on TEM foil thickness and limited collection efficiency/geometry of single detector XEDS systems didn't make those good candidates. The FEI ChemiSTEM™ technology features a patented windowless four silicon drift detector (SDD) design (FEI Super-X) and a high brightness XFEG Schottky source, optimized for high X-ray collection efficiency, fast XEDS mapping and improved tilt response. Monotonic relationship between EDS signal and chemical element concentration in the TEM foil, together with unrivaled >300 kcps output, allows fast recording of STEM XEDS maps tilt series, thus interpretable like normal z-contrast images. Examples of 3D chemical mapping using XEDS are shown on III-V quantum wells, LEDs, turbine blades, metal-gate transistor, and catalytic nanoparticles.

4:20 PM

Al_2O_3-TiO_2 Nanolaminates by Atomic Layer Deposition: A Study on the Laminate Structure and Mechanical Property: *Xuwen Liu*[1]; Sakari Sintonen[1]; Eero Haimi[1]; Mikko Laitinen[2]; Saima Ali[1]; Jaako Julin[2]; Oili Ylivaara[3]; Harri Lipsanen[1]; Timo Sajavaara[2]; Riikka Puurunen[2]; Simo-Pekka Hannula[1]; [1]Aalto University; [2]University of Jyväskylä; [3]VTT Technical Research Centre of Finland

Three sets of 100 nm thick ALD Al_2O_3-TiO_2 nanolaminates were deposited on 150 mm p-type (001) silicon wafers. Bilayer thickness (0.1 to 50 nm), volume per cent of TiO_2 (0 to 100%) and ALD temperature (110-300°C) were the variables in each corresponding set. The laminate hardness and elastic modulus were measured by nanoindentation under displacement control. X-ray reflectivity (XRR) was used for the thickness of each alternating layer, the density of each phase as well as the surface roughness at the interfaces. Time-of-flight elastic recoil detection analysis (TOF-ERDA) was used for the through-laminate impurity. Incorporating TiO_2 into Al_2O_3 reduced the film hardness. The TiO_2 content had a complex effect to the elastic modulus of the film. Better understanding of the subtle influence of the laminate structure and composition to the film property is reached by combining the information on the physical and chemical state of each individual layer.

4:35 PM

Inhomogeneous Structural and Mechanical Properties of Multilayered Thin Films Revealed at Micro- and Nano-Scale: *Jozef Keckes*[1]; R. Daniel[1]; A. Riedl[2]; M. Stefenelli[2]; J. Zalesak[1]; C. Mitterer[1]; [1]Montanuniversität Leoben; [2]Materials Center Leoben Forschung GmbH

Nanostructured thin films exhibit inhomogeneous depth gradients of microstructure, strain and mechanical properties. There is a need to develop novel experimental techniques to which can provide thickness-dependent data with sub-micron resolution. In this contribution, results from scanning synchrotron X-ray nano-diffraction studies of microstructure and strain on multi-layered thin films will be presented. On the examples of CrN-, TiN- and TiAlN-based coatings, it will be demonstrated that the newly developed experimental technique [1] can be used to analyse thickness-dependent gradients of strain, crystallographic texture, phases and grain size with resolution below 100nm. Additionally, results from mechanical tests obtained from bending experiments on micro-cantilevers fabricated from graded and multi-layered thin films [2] will be used to illustrate variability and anisotropy of mechanical properties at sub-micron scale. 1] Keckes et al., Scripta Mat. 67 (2012) 748.[2] Riedl et al., Scripta Mat. 67 (2012) 708.

4:50 PM

Microcompression Study of Al-Nb Nanoscale Multilayers: *Seung Min Han*[1]; Youbin Kim[1]; Arief Budiman[2]; Arief Budiman[2]; J. Kevin Baldwin[2]; Nathan Mara[2]; Amit Misra[2]; [1]Korea Advanced Institute of Science and Technology; [2]Los Alamos National Laboratory

Nanoscale multilayers with incoherent interfaces have unique mechanical properties that are expected to be dependent on the interfacial shear strengths. In this study, the mechanical properties of incoherent Al-Nb multilayers with 5nm and 50nm repeat layer spacings were first studied using microcompression tests, where the average 5% ow stresses for the 5nm repeat layer spacing (2.1GPa) was reported to be higher than that of 50nm repeat layer spacings (1.4 GPa), as expected. The results from this Al-Nb study were compared with those of the previous report on Cu-Nb multilayers to study the effect of different interfacial shear strength on the overall mechanical properties of the multilayers. Al-Nb multilayers that have higher interfacial shear strengths compared to Cu-Nb were shown to have higher flow stresses for the same repeat layer spacing, indicating that the interfacial shear strength plays an important role in determining the strength of the incoherent multilayers.

Session 9: Stresses in Thin-films and Multilayers II

Thursday AM Room: Auditorium
October 3, 2013 Location: IMDEA Materials Institute

Session Chair: Paul Ho, The University of Texas at Austin

9:00 AM Keynote

Multi-scale Modeling of Thermo-mechanical Reliability in Si and Interconnects: *Daniel Pantuso*[1]; Michael O'Day[1]; Satish Radhakrishnan[1]; Baha Kilic[1]; Arkaprabha Sengupta[1]; Mercedes Hernandez[1]; [1]Intel Corporation

As the micro-electronics industry continues its relentless push of Moore's law, significant innovations in terms of process, new materials introduction and overall integration are required to enable scaling and meet performance goals [1]. As a result, several new challenges are imposed at every technology node that needs to be overcome. One of these is the thermo-mechanical reliability risks. In order to understand thermo-mechanical reliability issues, one needs to have a very good understanding of the driving forces and their variability leading to different failure modes in Si and interconnect structures [2-3]. In this sense, modeling plays a critical role to provide this fundamental understanding and quantify the level of stress or strain that is sustained by these structures at the nano-scale size. In this presentation, we review the different scales involved in the thermo-mechanical reliability analysis of silicon devices and interconnects. We focus on two major thermo-mechanical issues: die package interactions [4] and line wiggling [5]. The first impacts mainly the structural integrity of weak ILD materials integrated in the silicon back-end. We will show the mechanisms leading to this failure mode and the modeling methodologies used to quantify the driving forces across all different scales involved. The line wiggling is a phenomena mainly occurring at the scale of sub-20 nm features impacting compliant ultra low-k ILD materials. It is due to an instability (buckling) of highly compressive sacrificial materials (hardmasks) required to pattern small features. The weak ILD material cannot sustain the forces due to the buckling of the hard mask on top it resulting in significant undulation. Modeling of the mechanisms associated with this undulation depends on the accurate description of the mechanical properties of all the materials in the integrated stack at the nm scale and how the different processes (clean, etches) impact them. References:M. Bohr, ISSCC 2009, pp. 23-28. The new era of scaling in a SoC world.A. Yeoh, et.al, ECTC 2006, pp. 1611-1615. Copper Die Bumps and Low-K Dielectrics in 65nm High Volume Manufacturing. D. Ingerly, et.al, IITC 2012. Low-k Interconnect Stack with Metal-Insulator-Metal Capacitors for 22nm High Volume Manufacturing. S. Rangaraj, et.al, ECTC 2013. Low-K ILD Reliability Through Chip-Package Assembly: Engineering Appropriate Stress Tests And Process Certification Criteria. A. Myers, MRS 2013 Spring Meeting. Fabrication Challenges in the Patterning of Sub-20 nm BEOL Features.

9:40 AM Invited

Assessment of Fracture and Elastoplastic Properties of Thin and Very Thin Films: *Ibon Ocaña*[1]; M. Reyes Elizalde[1]; Maria Trueba[1]; Jose Martinez-Esnaola[1]; Mercedes Hernandez[2]; Daniel Pantuso[2]; Michael Haverty[2]; Guangai Xu[2]; [1]CEIT; [2]Intel

Microelectronic industry is driven by the continuous miniaturization process conducing to the introduction of new materials. These materials are subjected to stresses mainly due to thermal mismatch, microstructural changes or process integration which can be in the origin of mechanical reliability issues. This work aims at developing tests to assess fracture or elastoplastic behaviour of thin ceramic, metallic and polymeric films. Proposed tests are based on indentation combined with sample preparation using mostly FIB. Among other techniques, different test geometries for microbeams have been evaluated and quantitative data have been obtained combining experimental results with analytical or numerical models, depending on the material under study. For the particular case of metallic layers, a strong dependence of plastic behavior on the orientation of the grains close to the fixed end has been detected. EBSD and FEM modelling using an in-house crystal plasticity subroutine have been combined to account for this effect.

10:00 AM Invited

Twinning in Strained Ferroelastics Thin Films: Microstructure and Statistics: *Xiangdong Ding*[1]; E.K.H. Salje[2]; Turab Lookman[3]; Jun Sun[1]; [1]Xi'an Jiaotong University; [2]University of Cambridge; [3]Los Alamos National Laboratory

The generation of functional interfaces such as superconducting and ferroelectric twin bounda-ries requires new ways to nucleate as many interfaces as possible in bulk materials and thin films. In this talk we show that the nucleation and propagation of twin boundaries in ferroelastics and martensites thin films depend sensitively on temperature and system size. The statistics of detwinning process in Ferroelastics thin films is thermally activated at high temperatures, whereas it follows athermal behaviour at low temperatures (PRB 2011, 2013). We further found that the strained thin films of ferroelastics with small bulk moduli have higher junction densities, which even increase with diminishing sample size (Adv Mater 2012,2013, JOM 2013). The change of the complexity and the number density of twin boundaries in thin film represent an important step forward in the development of domain boundary engineering, where the functionality of the materials is directly linked to the domain pattern.

10:20 AM Invited

Wear Resistance of Carbon/Boron Nitride Nanometric Multilayers: *Ignacio Jimenez*[1]; Ricardo Torres[1]; Ignacio Caretti[1]; José Cascales[1]; [1]ICMM-CSIC

We have studied the tribological properties of multilayers composed of nanoscale hexagonal carbon (h-C) and hexagonal boron nitride (h-BN) layers. These two materials are soft and lubricant in bulk, with poor wear resistance under high loads. However, when a stacking of nanoscale layers is formed, the wear resistance improves significantly showing wear rates below 1x10-7 mm3/N•m.The h-C/h-BN multilayers were formed by sequential evaporation of (i) carbon and (ii) the simultaneous evaporation ofboron with a concurrent nitrogen ion beam. In this way, a series of multilayers composed of sublayers ranging from a minimum thickness of 1.5 nm to a maximum thickness of 80 nm are obtained. Details on film preparation and characterization by SEM and TEM microscopies are discussed. Finally, the friction and wear resistance are evaluated by pin-on-disk tests under different test conditions.

10:40 AM Invited

Micromechanisms of Contact Deformation and Fracture in Multilayer Transition Metal Nitride Based Hard Coatings: *Vikram Jayaram*[1]; [1]Indian Institute of Science

The indentation response of columnar PVD coatings of nitrides is dominated by interfaces: column boundaries and the film-substrate interface are ever present, while multilayer laminate structures introduce additional interfaces between like phases (e.g., TiN-AlTiN) or dissimilar phases (TiN-Ti or ZrN-Zr). This talk will address the following issues: (1)What are the different basic damage modes and how are they influenced by substrate hardness, film thickness and residual stress? (2)How do interfaces between iso-structural hard phases alleviate fracture? (3) In nitride-metal composites, how do the metal layer thickness and volume fraction influence deformation? In concluding, the talk will examine the possibility of creating new architectures that get away from the laminate geometries dictated by the vapour deposition process.

11:00 AM Break

Session 10: Nanoscale Multilayers IV

Thursday AM — Room: Auditorium
October 3, 2013 — Location: IMDEA Materials Institute

Session Chair: David Bahr, Purdue University

11:30 AM Keynote

Size Effects in Plasticity and Fracture in the Indentation Assessment of Multilayer Coatings on Glass: *Steve Bull*[1]; [1]Newcastle University

The mechanical response of very thin oxide coatings (<100nm) which are used as antireflection and barrier layers in low emissivity architectural glass have been studied by nanoindentation methods to determine the effect of coating architecture and thickness on elasticity, plasticity and fracture toughness. This presentation will review the methods for obtaining hardness, elastic modulus and fracture toughness data for very thin coatings and assesses the existence of size effects in the mechanical response of oxide coatings with different thickness and different multilayer architectures on a glass substrate. As expected there are no size effects in elasticity but there are size effects in plastic response. For oxide coatings in the thickness range 100 to 400nm no size effects in fracture toughness were observed. For thinner layers in a multilayer architecture it is the fracture properties of the surrounding layers which control any observed size effects.

12:10 PM Invited

A Simple Stochastic Model for Yielding in Specimens with a Limited Number of Dislocations: *George Pharr*[1]; P. Phani[2]; Kurt Johanns[3]; Easo George[1]; [1]University of Tennessee and Oak Ridge National Laboratory; [2]Nanomechanics Inc.; [3]University of Tennessee

A simple stochastic model based on a random distribution and orientation of dislocations is developed to explain recent experimental observations of enhanced strength in small specimens containing a limited number of dislocations. Two different types of randomness are introduced: randomness in the location of dislocations and randomness in the stress needed to activate them. For convenience, the randomness in the activation stress is modeled by assigning a random Schmid factor, while the randomness in location is treated by a simple probabilistic model and verified with Monte Carlo simulations. The model is tested by comparing its predictions to: (1) recent experimental observations of the yield strengths of sub-micron diameter Mo alloy fibers measured in micro-tension and compression tests, and (2) the dependence of nanoindentation pop-in loads on indenter radius. The model predicts not only the observed size dependence of the strength, but the size dependence of the scatter as well.

12:30 PM Invited

Strain Rate Sensitivity and Related Strengthening Mechanism Transition in Nanoscale Ag/W Multilayers: *Qing Zhou*[1]; Fei Wang[2]; Ping Huang[1]; Kewei Xu[1]; [1]Dep of Mater Sci & Eng, Xi'an Jiaotong University; [2]School of Aerospace, Xi'an Jiaotong University

Nanoscale Ag/W multilayered thin films with wide ranges of both modulation period and modulation ratio (the ratio of Ag layer thickness to W layer thickness) were deposited by d.c. sputtering deposition. The cross-sectional morphologies of the multilayers were characterized under transmission electron microscopy and the strain rate sensitivity was examined by nanoindentation testing. It is indicated that the strain rate sensitivity was modulation period and modulation ratio dependent. Both the as-deposited columnar structure and the constraining effect between the ductile Ag and stiff W layers were found to affect the strain rate sensitivity of the multilayers significantly. In addition, a strengthening mechanism transition, from single dislocation slip in confined layers to accommodating deformation between the two constitute layers was proposed while changing the characterizing length scale of modulation period and modulation ratio.

12:50 PM Invited

Length-Scale- and Strain-Rate- Dependent Shear Banding Deformation in Nanoscale Crystalline/Amorphous Multilayer: *Fan Xue*[1]; Fei Wang[2]; Ping Huang[1]; Kewei Xu[1]; [1]Dep of Mater Sci & Eng, Xi'an Jiaotong University; [2]School of Aerospace, Xi'an Jiaotong University

Nanoscale crystalline/amorphous (C/A) multilayers with individual layer thickness ranging from several nanometers to 100nm were prepared by d.c. magnetron sputtering technique. The shear banding deformation behavior of the C/A multilayers were characterized via nanoindentation testing and scanning electron microscopy observation. A critical range of individual layer thickness ranging from 20 to 50nm, below which no shear bands (SBs) were found around residual indentation while above which numerous SBs appeared, was identified. In addition, applied strain rates were found to play a crucial role in forming SBs in the C/A multilayers system, as more SBs were observed at higher applied strain rates. Possible mechanisms corresponding to the length-scale- and strain-rate-dependent shear banding deformation behavior were extended discussed in terms of the special interface structure between the two constitute layers, i.e., crystalline and amorphous layers, and the stress incompatibility.

1:10 PM Invited
High-temperature Mechanical Properties of Physical Vapour-deposited (PVD) and Accumulative Roll-bonded (ARB) Cu/Nb Nanoscale Metallic Multilayers: *Miguel Monclus*[1]; Irene Beyerlein[2]; Nathan Mara[2]; Shijian Zheng[2]; Tomas Polcar[3]; Javier Llorca[1]; Jon Molina[1]; [1]IMDEA Materials; [2]Los Alamos National Laboratory; [3]Czech Technical University

Nanoscale metallic multilayers (NMMs) with layer thickness (L) in the nm range exhibit extraordinary mechanical properties because of their high density of interfaces. Of all NMMs systems, Cu/Nb have received the most attention due to their remarkable strength, toughness, high stability and resistance to irradiation. In this paper we report on the room- and high-temperature deformation behaviour of Cu/Nb NMMs produced by physical vapour deposition (PVD) and accumulative roll bonding (ARB) processes, with L in the range 5-50 nm. Multilayers were characterized using nanoindentation and micropillar compression at temperatures up to 400°C. Similar trends in the evolution of strength with temperature were found for both types of layers with deformation dominated by a confined layer slip mechanism, except for a transition to a more thermally assisted dislocation transmission mechanism for the thinnest ARB NMM. The microstructure under the indents was examined by transmission electron microscopy (TEM).

1:30 PM Lunch (Room: Main Hall)

Session 11: Nanoscale Multilayers V

Friday AM — Room: Auditorium
October 4, 2013 — Location: IMDEA Materials Institute

Session Chair: Amit Misra, Los Alamos National Laboratory

9:00 AM Keynote
Additional Strengthening Mechanisms in Multicomponent Metallic Nanolayers: *David Bahr*[1]; [1]Purdue University

This paper describes the addition of second phase particles of BCC metals within an FCC/FCC/BCC trilayer film. Cu and Ni are used as the FCC layers, while Cr and Nb as the BCC layer. Sputtering was used to create films with layer thicknesses between 4 and 30 nm. Properties were evaluated using nanoindentation and tensile testing at room temperature. Elevated temperature indentation testing showed the tri-layer films show less temperature sensitivity to softening as the film thickness decreases. The experimental results are compared to MD and DD simulations to explain the origin of the strengthening effects observed experimentally.

9:40 AM Invited
High Temperature Mechanical Behaviour of Al/SiC Multilayers: *Jon Molina-Aldareguia*[1]; Saeid Lotfian[1]; H.Y Xie[2]; Carl Mayer[2]; Nikhilesh Chawla[2]; Javier Llorca[1]; Amit Misra[3]; [1]IMDEA Materials Institute; [2]Arizona State University; [3]Los Alamos National Laboratory

Nanoscale multilayers exhibit extremely high strength and toughness. However, little is known about their high temperature behavior due to the difficulty on testing thin-films at high temperature. In this paper we discuss the high temperature mechanical behavior of metallic-ceramic (Al/SiC) nanoscale multilayers. The high temperature mechanical properties were characterized using two different techniques: nanoindentation and micropillar compression at temperatures up to 300°C. The former can be carried out without intensive sample preparation but analysis of the results is difficult due to the complex stress state imposed by the indenter. The latter requires the use of complex micromachining techniques but the results are easier to interpret. In view of this, the results obtained using both techniques will be compared and discussed. Finally, post-deformation microstructural analysis was carried by FIB and atomic force microscopy (AFM) to provide insight into the deformation mechanisms.

10:00 AM Invited
Strong and Ductile Amorphous-Crystalline Nanocomposites: Inga Knorr[1]; *Cynthia Volkert*[1]; [1]University of Göttingen

We have investigated the micromechanical behavior of nanoscale multilayers composed of amorphous and nanocrystalline materials with the aim of achieving high composite strengths and ductilities. Three sets of samples (PdSi/Cu, ZrO_2/Ti, and polycarbonate/Cu) are studied with layer thicknesses between 10 to 120 nm. Nanoindentation and microcompression are used to determine strengths, strains to failure and the deformation process in the multilayer films. The composite strengths can be used to determine the layer thickness dependent strengths of the individual layers, which are found to be limited by local constraint from the surrounding layers below a certain thickness. The high strains to failure and the complex deformation morphologies of the multilayer samples can be described in terms of three phenomena: co-deformation, strain softening and interlayer shear band formation. Simple models for all three are proposed which provide some guidance for optimal selection of composite materials and geometries.

10:20 AM Invited
Ultrahigh Deformability of Crystalline Cu/amorphous Cu-Zr Nanolaminates: Deformation-induced Devitrification: *Jinyu Zhang*[1]; Gang Liu[1]; Lin Gao[1]; Jun Sun[1]; [1]State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University

Metallic glasses are lucrative engineering materials owing to their outstanding mechanical properties such as superior strength and great elastic strain. However, the Achilles' heel of metallic amorphous materials — low plasticity caused by instantaneous catastrophic shear banding, significantly undercut their structural applications. Here, the nanolayered crystalline Cu/amorphous Cu-Zr micropillars with equal layer thickness spanning from 20 to 100 nm are uniaxially compressed and it is found that the Cu/Cu-Zr micropillars exhibit ultrahigh homogeneous deformation (= 30% strain) rather than localized shear banding at room temperature. This extraordinary plasticity is assisted by the deformation-induced devitrification (DID) via absorption/annihilation of abundant dislocations, triggering the cooperative shearing of shear transformation zones in glassy layers, which simultaneously renders the work-softening. The synthesis of such heterogeneous nanolayered structure not only hampers shear band generation but also provides a viable route to enhance the controllability of plastic deformation in metallic glassy composites via DID mechanism.

10:40 AM Invited
Multiscale Simulations of Elasticity in Nanostructured TiAlN Hard Coatings: *Ferenc Tasnadi*[1]; Igor Abrikosov[1]; Magnus Odén[1]; [1]IFM Linköping University

In TiAlN the anisotropic microstructure has a decisive impact on the hardness through an isostructural spinodal decomposition. Computational simulations of anisotropic or tensorial materials properties, such as elasticity, of substitutional alloys is among the most challenging and required issue in materials science. In this study we present an ab-intio framework [1] to predict and analyze the elasticty of TiAlN. We predict a large elastic anisotropy and compare the theoretical results with experiments [2]. We further underline the significance of our result through presenting the microstructure evolution in TiAlN on the dependence of the elastic anisotropy [3]. [1] F. Tasnádi et al., Phys. Rev. B 85, 144112 (2012). [2] F. Tanádi et al., Appl. Phys. Lett. 97, 231902 (2010). [3] J. Ullbrand, Phase field modeling of Spinodal decomposition in TiAlN, Licentiate thesis 2012, IFM Linköping University

11:00 AM Break

Session 12A: Characterization of Multilayers III

Friday AM — Room: Auditorium
October 4, 2013 — Location: IMDEA Materials Institute

Session Chair: Johann Michler, EMPA, Swiss Federal Laboratories for Materials Testing and Research

11:30 AM
An Object Kinetic Monte Carlo Model for Segregation in Multilayered Alloys: *Ignacio Dopico*[1]; Jose Luis Gomez-Selles[1]; Pedro Castrillo[2]; Ignacio Martin-Bragado[1]; [1]IMDEA Materials; [2]Universidad de Valladolid

A model for defect segregation in multilayered alloys has been developed and implemented into the Object Kinetic Monte Carlo simulator MMonCa. The model takes into account the point defect generation and diffusion, dopant diffusion and dopant segregation for each local composition material, as well as their charge properties and different states of defects in order to be able to reproduce the experimental results through a comprehensive computation of the phenomena involved in multilayered materials. Finally, results comparing simulations with actual experiments will be provided and discussed to validate and benchmark the robustness of the model and its implementation with examples of simulations on SiGe/Si layers compared with previous simulations, SIMS measurements and TCAD.

11:45 AM
An Object Kinetic MonteCarlo Approach to Helium Interaction with Grain Boundaries in Tungsten: Antonio Rivera[1]; *Gonzalo Valles*[1]; Raquel Gonzalez-Arrabal[1]; Jose Manuel Perlado[1]; Ignacio Martín-Bragado[1]; [1]Universidad Politecnica Madrid

Tungsten has been proposed as an armor material for nuclear fusion reactors. However, the major concern is related to the accumulation of helium in vacancy-like defects and subsequent development of bubbles eventually leading to detrimental exfolliation of the material. A nanostructured tungsten layer deposited on a steel substrate is being studied as an improved armor with a significantly formation of vacancy clusters due to the efficient annihilation of point defects at grain boundaries and interfaces. In this work we present object kinetic MonteCarlo simulations of the point defect kinetics as well as the helium interaction at interfaces and compare with recent experimental results. The simulations indeed show that at operation temperatures the grain boundaries contribute to reduce the undesired pressurized vacancy clusters.

12:00 PM Invited
The Size Effect: Micropillars and the Hall-Petch Effect: *David Dunstan*[1]; A. Bushby[1]; [1]Queen Mary University of London

Structure size effects have become apparent in a wide range of micromechanical testing configurations, from nanoindentation to micropillar compression. We have presented strong evidence that these are all manifestations of a single underlying size effect, fundamentally due to the relationship between dislocation curvature and stress, resulting in a higher stress required to drive dislocation extension or multiplication events in small volumes [Dunstan and Bushby, 2013, Int. J. Plasticity 40, 152]. Experiment and theory clearly indicate a dependence of strength on the inverse of size. We critically review the classic experimental evidence for the Hall-Petch inverse-square-root law. We conclude that the Hall-Petch effect is not an inverse-square root dependence on grain size and is the same size effect as seen in micromechanical testing and described by critical thickness theory. This has implications for models of dislocation – grain boundary and dislocation – interlayer boundary interaction.

12:20 PM
CrN/AlN Multilayer Hard Coatings Studied by Atom Probe Tomography: *Darius Tytko*[1]; [1]Max-Planck Institut für Eisenforschung

Nanoscale CrN/AlN multilayers are highly promising as

FRIDAY AM

hard coating materials for cutting tools due to their outstanding hardness, wear and oxidation resistance. By controlling the interface sharpness and the bilayer period of the multilayer system, the material can reach hardness values as high as 40 GPa. However, during operation at elevated temperatures such hard coatings undergo significant microstructural changes, which can cause a decrease in hardness. The aim of this study is to elucidate the nanostructural changes of CrN/AlN multilayers upon various thermal treatments and correlate them to the mechanical properties. Using RF – magnetron sputtering, CrN/AlN multilayer superlattices with different bilayer periods were produced. The basic characterization of as-deposited and annealed multilayers was conducted by Atom Probe Tomography in conjunction with X-Ray Diffraction and Transmission Electron Microscopy.

12:35 PM

Thermal Expansion of Cu Constrained in a TSV-Structure: *Ude Hangen*[1]; Jeremiah Vieregge[1]; Jaroslav Lukes[1]; David Vodnick[1]; [1]Hysitron, INC.

Silicon and Copper are materials used in semiconductor structures that do exhibit a large mismatch in the thermal expansion. Thermal cycling during the device's function or high temperature of around 400°C during production can induce stresses that potentially cause delamination in the device. Large stresses are built up in through silicon via structures because of their dimensions. A TSV is typically 5 to 25µm diameter hole in a Si wafer filled with copper that electrically connects front and back of the wafer. In-situ SPM imaging at elevated temperatures was used to examine plastic flow of copper caused by different thermal expansion coefficients of Si and Cu. In order to predict TSV's performance and lifetime, it is important to understand the relationship between time, temperature and mechanical properties. Combining reference frequency creep testing with a temperature stage control allows measurement of material behavior over a wide range of times and temperatures.

Session 12B: Processing

Friday AM — Room: Seminar Room
October 4, 2013 — Location: IMDEA Materials Institute

Session Chair: Nathan Mara, Los Alamos National Laboratory

11:30 AM

Formation of a Multilayer Structure with Electrical Contact Explosion: *Evgeny Grigoryev*[1]; Eugene Olevsky[2]; [1]MEPHI; [2]San Diego State University

Electric explosion of a tungsten carbide – cobalt material around high-speed steel forms a hardening coating on it surface. Refractory WC-Co composite material was sprayed and it has formed a WC/W coating on high speed steel without the use of additives or sintering agents to suppress chemical decomposition and enhance bonding of coating with substrate. The essential structure properties of formed coating are determined by parameters of the pulse current duration less than 100 microseconds and amplitude of more than 1 mega ampere per square cm and the properties of exploding electrode. The hardening layers of tungsten carbide and pure nanocrystalline tungsten have been formed upon the surface of high-speed steel as a result of electric exploding. Crystalline grains of tungsten have an almost spherical form and characteristic size less than 400 nanometers. Micro hardness of the coating layers and high-speed steel structures was measured.

11:45 AM

Ni/Al Nanolayers Produced by Sputtering and Cold Rolling: *Sónia Simões*[1]; Ana Sofia Ramos[2]; Filomena Viana[1]; Maria Teresa Vieira[2]; Jorge A. Ferreira[3]; Omid Emadinia[1]; Manuel Fernando Vieira[1]; [1]CEMUC, Department of Metallurgical and Materials Engineering, University of Porto; [2]CEMUC, Department of Mechanical Engineering, University of Coimbra; [3]LNEG, Laboratório Nacional de Energia e Geologia

Ni/Al nanolayers is an interesting system once it is known to transform into NiAl in a rapid, exothermic and self-sustained reaction. These nanolayers have significant potential for use as a controllable and localised heat source for joining temperature-sensitive materials, like microelectronic components. The heat released and the phase formation resulting from the reaction of Ni and Al multilayer depend on the processing, composition, interdiffusion or intermixing prior to phase transformation, bilayer thickness and annealing conditions. The influence of these variables on the reaction of commercial nanolayers and others, produced by sputtering and cold rolling, is exploited in this research. The structural evolution of the Ni/Al multilayer with temperature was studied by differential scanning calorimetry, X-ray diffraction and scanning and transmission electron microscopies. The nanolayers formed reveal differences in structure, heat release and phase formation, thereby affecting the potential to improve the process of diffusion bonding of several alloys.

12:00 PM

Fine-scale Multilayered Structures Produced by Angular Accumulative Drawing: *Janusz Majta*[1]; Krzysztof Muszka[1]; Marcin Kwiecien[1]; Paulina Graca[1]; [1]AGH University of Science and Technology

The highly refined structure was obtained by continuous Accumulative Angular Drawing (AAD). The recently developed AAD process is characterized by a complex strain path history, being an effect of wire diameter reduction, bending, tension and torsion that directly affects the microstructure changes in the final product. The process allows for obtaining severely deformed and high inhomogeneous microstructure. It has been shown that control of the microstructure development leads to nanoscale multilayered structures characterized by further improvement of strength, ductility and fatigue. The texture analysis was also consequently performed. The non-linear combined hardening model was applied to capture the strain path effects with special emphasis on the role of the inhomogeneity of the accumulated energy. Two methods were employed to determine the density of dislocations and dislocation interaction. Finally, it is shown that both the complex strain path history and the multilayered structure evolution can be quantitatively characterized numerically.

12:20 PM

Extrinsic and Intrinsic Size Effects of Amorphous CuZr/nanocrystalline Cu Nanolaminates at Micron & Submicron Scale: *Wei Guo*[1]; Jiahao Yao[1]; Korte Sandra[1]; Dierk Raabe[1]; Eric Jägle[1]; [1]Max-Planck-Institut für Eisenforschung GmbH

We synthesized nanolaminates with alternating layers of CuZr metallic glass and nanocrystalline Cu nanolaminates and employed both microcompression method and indentation method to study the effect of Cu layer thickness on the co-deformation behavior. While the Cu layer thickness increases from 10 nm to 100 nm, there is a deformation-mode transition from highly localized shear banding to preferential deformation of Cu layer through an intermediate stage of co-plastic deformation of both amorphous layer and nanocrystalline layer. Furthermore, a 40% strain without fracture was achieved accompanied with multiplication of shear bands. This present work suggests that the introduction of soft nanocrystalline phase into metallic glass thin films may prevent shear bands from ultrafast runaway proceeding and enhance the multiplication of shear bands, which increases the deformability and fracture toughness of the material. Finally, extrinsic and intrinsic size effects on yield stress will be discussed.

12:30 PM

Low Cost Solution based Synthesis of SLG/Mo/CISe/CdS/Al:ZnO Heterojunction Thin Film Solar Cells: *Muhammad Aftab Akram*[1]; Mohammad Islam[2]; Mohammad Mujahid[1]; [1]National Uiversity of Sciences and Technology Pakistan; [2]Center of Excellence for Research in Engineering Materials, Advanced Manufacturing Institute, King Saud University, Saudi Arabia

We present results from primarily solution based synthesis of thin film solar cells based on copper-indium-diselenide (CISe) absorber layer. Over soda-lime glass (SLG) substrates, molybdenum thin films with biaxial stress configuration were deposited via dc-plasma magnetron sputtering. Using salt precursors of copper, indium and selenium in a long-chain alcohol, 10µm absorber layer was applied over Mo back contact layer via paste coating followed by annealing at ~550 C for 1 hour in inert atmosphere. Solution processing routes offer simple, cost-effective alternatives to vacuum processing of thin film solar cells. Using chemical bath deposition, a 50-nm thick CdS buffer layer was produced over p-type CISe and front contact Al:ZnO was obtained by spin casting of a precursor sol. Sodium diffusion from SLG into Mo film as well as MoSe2 formation at the Mo/CISe interface was also examined and contact resistivity was found to be mediated by the presence of $MoSe_2$.

12:45 PM Lunch (Room: Main Hall)

Session 13A: Nanoscale Multilayers VI

Friday PM	Room: Auditorium
October 4, 2013	Location: IMDEA Materials Institute

Session Chair: Jon Molina-Aldareguia, IMDEA Materials Institute

1:45 PM

Electrofabrication of Nanoscale Multilayer Coatings of Ni-Cd Alloy for Better Corrosion Protection: *Ampar Hegde*[1]; Vaishak Rao[1]; [1]NITK, Surathkal

Nanoscale Multilayer Alloy (NMA) coatings of Ni-Cd were performed galvanostatically on mild steel using square current pulses by alternatively changing the cathode current densities (CCCD's). NMA coatings were made up by alternating layers of alloys of two composition while keeping each individual layer thickness below ~100 nm. The NMA coatings were developed under different conditions of CCCD's and number of layers; and their corrosion behavior were evaluated in 5% NaCl by electrochemical DC and AC methods. Under optimal condition, the corrosion resistance of NMA Ni-Cd coating was found to be few tens of times higher than monolayer Ni-Cd coating deposited from same bath for same time. The experimental results of NMA coatings are presented in Tables and Figures while explaining the reasons for enhanced corrosion protection in the light of structure-property relationship of nanomaterials through XRD, SEM and EDAX analysis.

2:00 PM

Fabrication and Mechanical Behavior of Multilayer Laue Lenses: *Sven Niese*[1]; Adam Kubec[2]; Peter Krueger[1]; Stefan Braun[2]; Andreas Leson[2]; Ehrenfried Zschech[1]; [1]Fraunhofer IZFP Dresden; [2]Fraunhofer IWS Dresden

Multilayer Laue lenses are currently explored as X-ray lenses for nanometer focusing and imaging in X-ray microscopy and related methods. Compared to Fresnel zone plates, they allow tremendously larger aspect ratios of the single zones, showing a path for high-efficiency high-resolution optics for hard X-rays. Layer thicknesses follow the zone plate law, yielding in dozens of microns height multilayer stacks with minimum layer thicknesses in the single nanometer range. To ensure the geometrical precision of the lens, layer deposition done by magnetron sputter deposition has to be performed with deviations of less than 0.1 %. We show results for a Si/WSi_2 system as well as strategies to avoid undesired elastic deformation at subsequent processing of single lenses and to exploit useful deformations for enhanced lens performance.

2:15 PM

The Influence of Stacking Fault Energy on the Formation of Highly Nanotwinned Cu-Al Alloys: *Leonardo Velasco*[1]; Mikhail Polyakov[1]; Andrea Hodge[1]; [1]University Of Southern California

Thin films of Cu (99.999%) and Cu-Al alloys (Cu- 6 wt.% Al and Cu- 4 wt.% Al) were sputtered under identical conditions, in order to compare nanotwinned microstructures. Due to the wide range of stacking fault energies (SFE) in these materials (6-78 mJ/m2), the microstructure features were expected to change substantially. The temperature during processing was measured in order to explore and understand the different sputtering behavior for Cu and Cu-Al alloys and its influence on generating a nanotwinned structure. The samples produced were characterized by TEM and FIB. The Cu-Al samples showed highly columnar and nanotwinned structures, while the Cu samples presented few columnar grains, limited number of nanotwinned grains, and fine grain size. The reduced SFE of the Cu-Al alloys promoted a highly aligned nanotwinned columnar grain structure.

2:30 PM

Microstructural Variations in Cu/Nb and Al/Nb Nano Metallic Multilayers: *Mikhail Polyakov*[1]; Andrea Hodge[1]; Christian Kübel[2]; [1]University of Southern California; [2]Karlsruhe Institute of Technology

Nano metallic multilayers were fabricated by magnetron sputtering using a miscible (Al/Nb) and an immiscible (Cu/Nb) system. The samples were characterized by means of transmission electron microscopy techniques, among them automated crystallographic orientation mapping (ACOM). The ACOM technique indexes individual nano-electron diffraction patterns and allows for the resolution of crystal structures and orientations at the nanoscale. The microstructures of the Nb layers were shown to vary with Al and Cu layer thicknesses. The Al and Cu layers were found to consist of both amorphous and crystalline regions, which had different effects on the overall microstructures.

2:45 PM
Tensile Properties of Electroplated CoNi Nanoscale Multilayers with Bimodal Grain Size: Matthew Daly[1]; *Glenn Hibbard*[1]; Chandra Veer Singh[1]; [1]University of Toronto

With the surging industrial demand for high performance and ultra-light materials, a significant need has developed for elegant nanotechnology solutions to replace conventional engineered materials. Electroplated nanoscale multilayers have emerged as a promising candidate, because of their synergistic exploitation of dislocation-based plasticity and the Hall-Petch hardening phenomenon. The current work investigates the mechanical properties of electroplated CoNi nanoscale multilayers with a bimodal grain size distribution. Pulsed electroplating techniques are used to deposit CoNi in solid solution, with alternating layer grain sizes of ~10 and ~200 nm. Mechanical properties are characterized via uniaxial tensile testing combined with a digital image correlation system and electron microscopy is utilized to image the as-deposited CoNi multilayer structure. Using the experimentally measured elastic-plastic properties as material inputs, a finite element model is constructed in order to predict the mechanical response of the CoNi nanoscale multilayer.

Session 13B: Nanoscale Multilayers VII

Friday PM
October 4, 2013

Room: Seminar Room
Location: IMDEA Materials Institute

Session Chair: Daniel Kiener, University of Leoben

1:45 PM
Thermal Stability of Nanoscale Multilayers: *Ana Sofia Ramos*[1]; André João Cavaleiro[1]; Maria Teresa Vieira[1]; Jerzy Morgiel[2]; [1]CEMUC, University of Coimbra; [2]Polish Academy of Sciences

Metallic nanolayered thin films/foils, in particular Ni/Al multilayers, have been used to promote joining. The objective of this work is to evaluate the thermal stability of nanoscale metallic multilayers with potential for joining applications. Low (Ti/Al and Ni/Ti), medium (Ni/Al) and high (Pd/Al) energy multilayer thin films with nanometric modulation periods and near equiatomic overall chemical composition were prepared by dual cathode magnetron sputtering. Their thermal stability was studied by: i) differential scanning calorimetry combined with X-ray diffraction (XRD), and ii) in-situ XRD using cobalt and synchrotron radiation. It is possible to detect traces of intermetallic or amorphous phases in the as-deposited short period multilayers, except for the Ti/Al films where no reaction products that might be formed during deposition were identified. For short periods (below 20 nm) the equilibrium phases are directly achieved upon annealing, whereas for higher periods trialuminide phases are formed in Ti/Al, Ni/Al and Pd/Al multilayers.

2:00 PM
Mechanisms for Residual Stress Evolution in Vapor-deposited Ultra-thin Metal Layers: *Hang Yu*[1]; Carl Thompson[1]; [1]Massachusetts Institute of Technology

Control of the residual stress in ultra-thin (10-100nm) metal layers is critical for many applications, including flexible electronic devices, protective coatings, micro/nano beam based actuators, sensors, and accelerometers. However, intrinsic stresses on the order of 100MPa to 1GPa can be generated during vapor phase growth. Evolution of the nano-scale structure of the film is closely correlated with evolution of the residual stress. Because of the nanoscale grain size in these layers, evolution of the grain structure affects the residual stress even at homologous temperatures as low as 0.2-0.3. When deposition is stopped, the stress often continues to evolve, with a reversible fast component and an irreversible slow component. Based on in situ real-time stress measurements, structure characterization and analytical modeling, we conclude that the underlying mechanisms are associated with changes in the roughness of the film surface and the grain size, respectively.

2:15 PM
Multilayer ALD Coating of Light Water Reactor Zirconium Alloy Cladding Material: *Pellin Michael*[1]; Abdellatif Yacout[1]; Marshall Mendelsohn[1]; Di Yun[1]; Walid Mohamed[1]; [1]Argonne National Laboratory

The accident at Fukushima Daiichi nuclear power plant raised concerns about nuclear reactors safety. The plant experienced an accident in which the fuel elements zirconium (Zr) alloy cladding tubes reacted with high temperature steam and went through severe oxidation releasing hydrogen, ultimately leading to hydrogen explosion. Those events led to interest in coating cladding surfaces to slow or arrest high temperature steam-cladding reaction. Atomic layer deposition (ALD) technique is proposed here to coat Zr alloy cylindrical surfaces with multiple nano-scale layers of different materials to protect against oxidation. Key to coating performance is its stability in an irradiation environment as well as its mechanical performance under reactor operating conditions. This paper describes activities at Argonne national Laboratory in relation to the use of ALD technique to deposit multilayers of nano-scale coating material on Zr alloy cladding and investigation of coating stability under simulated high temperature steam and irradiation damage environment.

2:30 PM

Modelling of Oxide Failure in Highly Reactive Nanocristallised Multilayered Metal Structures during Thermomechanical Processing: *Michal Krzyzanowski*[1]; W. Mark Rainforth[2]; [1]AGH University of Science and Technology; [2]The University of Sheffield

The advanced physically based modelling approach developed earlier for oxide failure at the roll-stock interface has been extended to provide the basis for detailed investigations of the interfacial oxidation during duplex techniques combining nanocrystallisation processes with a subsequent co-rolling in order to produce multilayered bulk structures characterised by improved mechanical properties. The research encompassed modelling of the combined hot compression-tension test followed by hot rolling of the assembly of steel strips preliminary subjected to the surface nanocrystallisation. The possibility of a co-operative relationship between the formation of oxide scale related defects at the interfaces and formation of shear zones within the affected areas around the interfaces has been demonstrated numerically. The through-thickness shear zones within the material can link the scale related defects on both the upper and lower interfaces. Formation of the scale related shear zones within the strip volume takes place mainly during the subsequent rolling passes.

2:45 PM

Express Test-Evaluation and Application of a Novel Technique for Rapid Acquisition and Mapping of Accurate Mechanical Properties: *Holger Pfaff*[1]; [1]Agilent Technologies

Nanoindentation has become a well-established technique to quantify Young's modulus and hardness of microscopic volumes of materials and allows mapping out mechanical properties on surfaces with high spatial resolution. Mechanical maps are extremely useful to understand gradients in engineering materials due to processing or changes in operation. Up to now however, the acquisition of such maps was extremely time consuming, taking a single Pixel requiring several tens of seconds to even minutes. Recently a novel technique has been introduced for fully quantitative rapid mapping of Young's modulus, hardness and topography simultaneously. The new technique generates such maps about a hundred times faster without compromising the accuracy of the mechanical data. Acquiring the information of several hundred individual tests in less than 10 minutes, not only allows for accurate mapping of these properties, but also strongly boosts the statistic verification for properties of mechanically inhomogeneous specimens or rough sample surfaces. In addition the technique minimizes effects of thermal drift, a well-known issue for quantitative tests in the nano scale. In recent studies we have performed series of tests to investigate the full potential as well as the limits of this technique. The results will be summarized together with illustrating some recent applications.

Conference Closure

Friday PM	Room: Auditorium
October 4, 2013	Location: IMDEA Materials Institute

Session Chairs: J. Llorca; I. Martin-Bragado; J.Molina-Aldareguia

3:00 PM Conference Closure

Poster Session

Tuesday AM Room: Main Hall
October 1, 2013 Location: IMDEA Materials Institute

P-1: Influence of the IR-mirror Layer Composition in the Mechanical Properties of Mo:Si_3N_4 Cermet Based Solar Selective Multilayer Coatings: *Leopoldo Alvarez-Fraga*[1]; Miguel Monclus[2]; Jon Molina-Aldareguia[2]; Ramón Escobar-Galindo[1]; Carlos Prieto[1]; [1]ICMM/CSIC; [2]IMDEA Materials Institute

The design of solar selective coatings for concentrated solar power (CSP) applications requires a precise knowledge of the optical and mechanical behaviors of materials forming the coating. We have proposed the Mo:Si_3N_4 cermet as the component for absorber layers to prepare selective multilayer coatings with excellent selective properties. Coatings are formed by (i) an IR-mirror metal, (ii) a double cermet and (iii) an antireflective dielectric layers. Optical characteristics at the visible range are determined by thickness and composition of the cermet and antireflective layers and the behavior at the IR-range is mainly determined by the nature of the metal forming the IR-mirror, where silver is the preferred metal. In this work, we study the mechanical behavior of solar selective multilayer coatings prepared by magnetron sputtering in which the Ag IR-mirror is modified by Ti and TiN in order to improve its hardness.

P-2: Three-dimensional Crack Visualization in Patterned Layer Structures by In-situ X-ray Microscopy: *Sven Niese*[1]; Alex Hsing[2]; Jeff Gelb[3]; Kevin Fahey[3]; Reinhold Dauskardt[2]; Peter Krueger[1]; Ehrenfried Zschech[1]; [1]Fraunhofer IZFP Dresden; [2]Stanford University; [3]Xradia Inc.

In-house X-ray microscopy (XRM) fills the gap in-between transmission electron microscopy and microfocus X-ray imaging. Due to the negligible refraction inside the specimen, a three-dimensional reconstruction can be obtained from a series of single radiographs by computed tomography with sub-100 nm resolution. Additional features of XRM are long working distances in the centimeter range, enabling an easy integration of in-situ devices, and the possibility to enhance the contrast at phase boundaries like cracks by Zernike phase contrast. We implemented a miniaturized dual cantilever beam tester inside our microscope. The dimensions of the specimen are typically 50×50×500 μm^3. We present experimental results showing crack propagation in metallization layers of state-of the art microelectronic devices as a complementary method to the well known macroscopic tests in fracture mechanics.

P-3: Pattern Transfer by using Multilayer Graphene as a Sacrificial Layer: *Jeong Min Woo*[1]; Min-Sik Kim[1]; Dae-Seon Kim[1]; Jae-Hyung Jang[1]; [1]Gwangju Institute of Science and Technology

Multilayer graphene has been studied as nanoribbon, transparent electrode and electron channel layer for the application of electronics, but there have been not many studies on the mechanical behavior of the multilayer graphene. Graphene layer is consisted of sp2 bonding with carbon atoms that it has one free arm to have covalent bonding, and it forms π-π bonding in multilayer graphene. By breaking π-π bonding, we demonstrated transferrable metamaterial structure that is composed of periodic subwavelength metallic structure. The multilayer graphene was grown on nickel by chemical vapor deposition, and the conventional optical lithography was utilized to fabricate metamaterial on multilayer graphene. Then, PMMA was coated and cured on the sample. Finally, the PMMA film was detached carefully from the sample so that the metamaterial was successfully transferred from nickel to PMMA with no deformation and irregular array.

P-4: Mechanical and Microstrutural Properties of W/Cu and Zr/Nb Nanoscale Multilayers: *Miguel Monclus*[1]; Tomas Polcar[2]; Emilio Frutos[2]; Javier Llorca[1]; Jon Molina[1]; [1]IMDEA Materials; [2]Czech Technical University

Cu/W and Zr/Nb nanoscale metallic multilayers (NMMs) with individual layer thicknesses (L) in the range 5-30 nm were deposited by magnetron sputtering. The hardness and modulus of the deposited NMMS were investigated via nanoindentation. W/Cu NMMs exhibited large variation of hardness and modulus with penetration depth, with the hardness peaking at ≈ 4.5 GPa for L = 15 nm, while the thinnest layers (L = 5 nm) exhibited the lowest modulus and hardness values. The softer Cu layer appears to dominate multilayer behavior and the modulus does not follow rule of mixtures. Regarding the Zr/Nb NMMs, hardness is lowest for thinnest layer (L = 5 nm) at ≈ 6 GPa, compared with a value closer to 7 GPa for L = 15 nm and L = 30 nm. The microstructure of the NMMs was investigated by TEM and observations discussed.

P-5: Atomistic Simulations of Vacancy Energetics in Pure Transition Metals: Darío Fernández-Pello[1]; César González[1]; Artur Tamm[2]; Erki Metsanurk[3]; María Ángeles Cerdeira[1]; Sergio Luis Palacios[1]; Alvo Aabloo[2]; Mattias Klintenberg[3]; *Roberto Iglesias*[1]; [1]Universidad de Oviedo; [2]University of Tartu; [3]Uppsala University

Nanoscale multilayers composed of bcc/fcc transition metals have been shown to present superior radiation resistance [1]. Vacancies are the most common point defects appearing in irradiated materials at high temperatures. We have performed a systematic atomistic study, of the energetics of vacancies in pure transition metals, identified as possible candidates for building those multilayers. The atomic shell displacements from the unrelaxed structures reveal a complex behaviour related to the electronic valence band configuration. We conclude that quite large supercells are needed to be confident about the reliability of the results and get rid of unphysical, size-related effects. MD data are crosschecked against DFT simulations to test with the highest precision the validity of the commonly accepted potentials for these systems in the presence of the rapidly changing ionic environment of a vacancy. 1. M. J. Demkowicz, A. Misra, and A. Caro, Curr. Opin. Solid St. M., 16, 101 (2012).

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