

SYMPOSIUM B

Structure and Mechanical Properties of Nanophase Materials— Theory and Computer Simulations vs Experiment

November 28 – 30, 2000

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Symposium Support

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Proceedings published as Volume 634
of the Materials Research Society
Symposium Proceedings Series.

* Invited paper

SESSION B1: MECHANICAL PROPERTIES AND DEFORMATION BEHAVIOR I

Chair: Helena Van Swygenhoven
Tuesday Morning, November 28, 2000
Hampton (Sheraton)

8:30 AM *B1.1

AN OVERVIEW OF DEFORMATION AND FRACTURE OF EMBEDDED PHASES IN FINE SCALE METALLIC COMPOSITES. J.D. Embury and C. Sinclair, Department of Materials Science and Engineering, McMaster University, Hamilton, Ontario, CANADA.

The deformation of nanoscale materials, metallic multilayers, and drawn in-situ composites all raise important issues as to the mechanism of deformation of the embedded hard phases and events at the interface interfaces. This presentation will examine the detailed mechanisms by which fine scale composites make the transition from elastic to plastic deformation. This includes the use of neutron diffraction methods to examine the elastic strains sustained by the embedded phase prior to plastic flow. Also detailed TEM studies will be presented. Consideration will also be given to large strain deformation of in-situ composites and the importance of the structure of the interfaces in plasticity and fracture processes.

9:00 AM B1.2

TEM OBSERVATION OF NANOCRYSTALLINE COPPER DURING DEFORMATION. Richard C. Hugo, Harriet Kung, Los Alamos National Laboratory, MST-8, Los Alamos, NM; Carl J. Youngdahl, Julia Weertman, Northwestern University, Department of Materials Science and Engineering, Evanston, IL.

Numerous theoretical calculations and simulations suggest that at the small end of the grain size spectrum crystalline materials deform primarily by a mechanism other than dislocation plasticity. Much of the experimental data on this subject is indirect, consisting of correlations between some measure of strength and grain size. TEM observation of a sample during straining can provide direct information on deformation behavior in nanocrystalline material. Nanocrystalline samples of copper were prepared using inert gas condensation and an optimized sequence of powder outgassing and compaction. TEM specimens were cut, electropolished, and mounted in a straining stage. Several structural changes were captured on video during deformation, including numerous sudden shifts in contrast of grains and parts of grains, occasional directly observed dislocation motion, opening and propagation of cracks around the electropolished hole, and occasional gradual changes in contrast. Larger grains appear to account for much of the deformation. However smaller grains also exhibit contrast shifts, suggesting that they too are participating in the deformation. Relationships between internal structure and deformation will be discussed, including the influence of grain size on dislocation activity and contrast shifts.

9:15 AM B1.3

SUPERPLASTICITY IN NANOCRYSTALLINE Ni₃Al AND Ti ALLOYS. S.X. McFadden, A.V. Sergueeva, A.K. Mukherjee.

The advent of nanocrystalline materials has provided new opportunities to explore grain size dependent phenomenon. Superplasticity is a grain size dependent phenomenon defined by the ability to attain tensile elongation of 200% or more. Superplasticity in microcrystalline materials has been well characterized. The constitutive equations that describe microcrystalline superplasticity predict enhanced properties for nanocrystalline materials. Enhanced properties include lower superplastic temperature, higher superplastic strain rate, and lower flow stresses. Investigations with nanocrystalline Ni₃Al and ultra-fine grained Ti alloys have shown a reduction in the superplastic temperature. However, the flow stresses in these materials are significantly higher than expected. The high flow stresses are accompanied by strong strain hardening. Transmission electron microscopy in situ straining of nanocrystalline Ni₃Al has shown that grain boundary sliding and grain rotation occurred during straining. The sliding and rotation decreased with strain. Dislocation activity was observed but was not extensive. There was no observable dislocation storage. The parameters of the generalized constitutive equation for superplasticity for nanocrystalline Ni₃Al and Ti alloys are in reasonable agreement with the parameters for microcrystalline material. The constitutive parameters suggest that nanocrystalline superplasticity shares common features with microcrystalline superplasticity. In contrast, the observed flow stresses and strong strain hardening indicate that nanocrystalline superplasticity is not a simple extension of microcrystalline behavior scaled to finer grain size. The authors gratefully acknowledge support from the National Science Foundation under grant NSF-DMR-9903321.

9:30 AM B1.4

THE ROLE PLAYED BY TWO PARALLEL FREE SURFACES IN THE DEFORMATION MECHANISM OF NANOCRYSTALLINE

METALS: A MOLECULAR DYNAMICS SIMULATION. Peter Derlet and H. Van Swygenhoven Paul Scherrer Institute, Villigen PSI, SWITZERLAND.

Former molecular dynamics computer simulations of polycrystalline Ni and Cu metals with mean grain sizes ranging between 3 and 20 nm demonstrated a change in deformation mechanism as function of grain size: at the smallest grain sizes all deformation is accommodated in the grain boundaries (Van Swygenhoven et al. Acta Materialia, 47 (19) 1999). In this paper we report on the influence of the presence of two free surfaces on the deformation behaviour. The purpose of these simulations is to study which phenomena observed in in-situ tensile experiments performed in the electron microscope can be expected to be intrinsic properties of the deformation process and which phenomena are due to the presence of two free surfaces separated by only a small distance. The simulated nanocrystalline samples, formerly relaxed with continuous boundary conditions, are now relaxed with two parallel free surfaces: grain boundary relaxation and grain reorientation are observed. In addition surface steps emerge in particular grains. Such structural relaxations are studied as a function of grain size and their influence on the deformation mechanism is reported.

10:15 AM *B1.5

MECHANICAL SPECTROSCOPY OF NANOCRYSTALLINE METALS. Ennio Bonetti, Dept. of Physics and INFN, Bologna, ITALY.

The mechanical behaviour of nanocrystalline Nickel and Iron prepared by inert gas condensation and by mechanical attrition was studied by mechanical spectroscopy techniques in the quasi static and dynamic low-frequency stress-strain regimes. Different types of measurements were carried out allowing a study of the relationships between dynamic and static response functions, corresponding to specific types (static-dynamic) of the conjugate variables stress and strain. A major item addressed was the variation of the mechanical response after controlled thermal annealing so as to induce microstructural modifications affecting the interfaces and/or grains. Other structural features affecting the mechanical behaviour such as the grain size, interfaces disorder degree, porosity and density have been taken into account. The analysis of the anelastic spectra by a combination of quasi-static and dynamic measures allowed to trace a draft phenomenological modeling of the elasto-plastic behaviour of nanocrystalline metals. In particular, creep and stress relaxation experiments at low temperatures ($T < 0.25 T_m$) show a large time dependent recoverable strain with a thermally activated character. The activation energy is comparable with that for grain boundary diffusion. Some links with the mechanical behaviour predictions for nanocrystalline metals derived from recent molecular dynamics simulations will be discussed.

10:45 AM B1.6

INTERFACE STRESS IN NANOCRYSTALLINE MATERIALS. R. Birringer, M. Hoffmann, University of the Saarland, Dept of Physics, Saarbruecken, GERMANY.

Nanocrystalline materials are characterized by a high density of internal interfaces (grain or phase boundaries). This enables probing the interface stress by measuring the pressure exerted on the nanocrystals by the network of interfaces comprising the interfacial component of the nanocrystalline sample. For materials with fcc symmetry, we are able to discriminate residual stress and hence obtain the absolute value of the interface stress. Combining this result with measurements of the overall elastic constants of nanocrystalline materials, we can estimate lower and upper bounds for the interface elastic constants.

11:00 AM B1.7

GRAIN SIZE DISTRIBUTION AND DISLOCATION CHARACTER IN NANOCRYSTALLINE COPPER. T. Ungár, G. Tichy, Eötvös University Budapest, Budapest, HUNGARY; P.G. Sanders, R. Mitra, J.R. Weertman, Northwestern University, Evanston, IL.

The grain size, grain-size distribution and the dislocation structure have been investigated by X-ray diffraction profile analysis in nanocrystalline copper produced by inert gas condensation and compaction. The FWHM, the integral breadths and the Fourier coefficients of the diffraction profiles are analyzed by the *modified* Williamson-Hall and Warren-Averbach procedures in terms of apparent size parameters and the dislocation model of the mean square strain, $\langle \epsilon^2 \rangle$. The three apparent size parameters are converted into the log-normal size distribution of grains by a standard least square method. The dislocation structure is characterized by the dislocation density and the *hkl*-dependent average contrast factors of dislocations, $\bar{\tau}$. Analysis of the experimentally determined $\bar{\tau}$ values has shown the presence of the usual $\langle 110 \rangle$ dislocations. However as the grain size decreases, and especially below about 50 nm, it is

revealed that dislocations with cubic Burgers vectors $\langle 100 \rangle$ become more and more frequent. It is assumed that these sessile Lomer-type dislocations reside mainly in the grain boundary regions.

11:15 AM **B1.8**

STRUCTURAL EVALUATION OF FCC MATERIALS MECHANICALLY MILLED. Jianhong He, Enrigue J. Lavernia, Univ. of California Irvine, Dept. of Chemical & Biochemical Engineering & Materials Science, Irvine, CA.

The microstructures of mechanically milled Inconel 625, NiCr, and Al powders were investigated using transmission electron microscope and X-ray diffraction. A high dislocation density was observed in the as-received gas atomization powders. During the initial stage of milling, grain size decreased nonuniformly; i.e., some grains reached the nanoscale (less than 100 nm), while others remained in the micron-scale. However, at the final stage of milling, all grains decreased to the nanoscale regime, and the dimensions of individual grains was quite similar. A high density of deformed faults was observed throughout milling process; this is thought to be associated with the development of the final grain structure. On the basis of the present findings, a mechanical milling mechanism is proposed as follows. First, grains in a powder particle are elongated in deformation direction, similar to that of rolled materials. Second, grains are frequently elongated in random directions because milling causes deformation in random directions, and edge regions of the grains are heavily suffered from strain fatigue. Third, fracture occurs in the regions suffered from strain fatigue, in other words, strain fatigue leads to fracture edge regions of the grains into nano-sized grains, concurrently, these nano-sized grains rotate relative to the original grain. Finally, the whole grain is divided into nano-sized grains by strain fatigue fracturing.

11:30 AM **B1.9**

MOLECULAR DYNAMICS SIMULATION OF NANO-SIZED CRYSTALLIZATION DURING PLASTIC DEFORMATION IN AN AMORPHOUS METAL. Ryuichi Tarumi, Akio Ogura, Masayuki Shimojo, Kazuki Takashima, Yakichi Higo, Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, JAPAN.

Amorphous metals have high strength and isotropic mechanical properties resulted from the absence of long-range ordering. It is therefore expected to be used as a structural material. Amorphous phase is a non-equilibrium state so that structural changes may occur due to not only heat treatment but also plastic deformation. The precipitation of nano-sized crystalline phase has been observed after the plastic deformation of amorphous alloys, but the mechanism of crystallization has not been established yet. Computer simulation is one of the most valuable tools to investigate phenomena that are difficult to observe experimentally. In this study, an NTP ensemble (the Number of atoms, Temperature and Pressure in the model are constant) molecular dynamics simulation was performed to investigate the mechanism of nano-sized crystallization during plastic deformation in an amorphous metal. In molecular dynamics simulation, an amorphous model is constructed from 1372 Ni atoms interacting via a Morse type pairwise additive potential. The applied shear stress and the temperature of the model were controlled by Parrinello-Rahman and Nose methods, respectively. At shear stresses below 2.4 GPa, shear strain increased linearly with increasing shear stress. However, when shear stress reached 2.8 GPa, large shear deformation occurred until the shear strain reached 0.79. During this shear deformation, crystallization was observed in the model. The crystalline phase had a fcc structure which had an orientation relationship, i.e. the shear direction and a (111) plane of the crystalline phase were parallel. This relationship was consistent with TEM (Transmission Electron Microscopy) observation for a Ni-P amorphous alloy. Mechanisms of nano-sized crystallization during shear deformation were discussed.

11:45 AM **B1.10**

FORMATION OF NANO-SIZED CRYSTALS DURING PLASTIC DEFORMATION IN AMORPHOUS ALLOYS. Megumi Sato, Akio Ogura, Ryuichi Tarumi, Masayuki Shimojo, Kazuki Takashima, Yakichi Higo, Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, JAPAN.

Amorphous alloys, which have no long-range order, are not in a thermally equilibrium state. Therefore, amorphous alloys will transform to crystalline phase by not only heat treatment but also plastic deformation. The crystallization process during plastic deformation is considered to be different from that during heat treatment. However, the detail of the crystallization during plastic deformation has not yet been clarified. In this study, structural changes during plastic deformation have been investigated for two types of amorphous alloys that have different thermal stability, and the effects of thermal stability on the crystallization have been examined. The materials used were Ni-P and Fe-Si-B amorphous alloys. Micro-sized cantilever beam type specimens with dimensions of

$10 \times 8 \times 55 \mu\text{m}^3$ were prepared from the amorphous alloys by focused ion beam machining. Bending tests were performed using a mechanical testing machine for micro-sized specimens. After the bending tests, top and side surfaces of the specimens were observed by a scanning electron microscope. Shear bands were observed clearly on surfaces of both the Ni-P and Fe-Si-B specimens. Transmission electron microscopy observation revealed the formation of nano-sized crystals in the Ni-P specimen. Furthermore, the crystalline phase had an orientation relationship with shear direction. In contrast, no crystalline phase was observed in the Fe-Si-B specimen. Crystallization temperatures of Fe-Si-B and Ni-P specimens were approximately 820 K and 620 K, respectively. Thus, the thermal stability of the Fe-Si-B amorphous alloy is considered to be higher than that of the Ni-P amorphous alloy. This suggests that thermal stability of amorphous alloys affects the formation of nano-sized crystals during plastic deformation. The formation mechanism of nano-sized crystals during plastic deformation is also discussed.

SESSION B2: MECHANICAL PROPERTIES AND DEFORMATION BEHAVIOR II – BULK MATERIALS

Chair: Diana Farkas

Tuesday Afternoon, November 28, 2000

Hampton (Sheraton)

1:30 PM ***B2.1**

CYCLIC DEFORMATION AND FATIGUE PROPERTIES OF ULTRAFINE-GRAIN SIZE MATERIALS: A REVIEW OF THE CURRENT STATUS. Hael Mughrabi, Universitaet Erlangen-Nuernberg, Institut fuer Werkstoffwissenschaften, Erlangen, FEDERAL REPUBLIC OF GERMANY.

The study of the mechanical properties and the microstructure of ultrafine grain (UFG) size metals and alloys has become an important research area, since it has become possible to produce metallic compact bulk structural materials by new processing techniques such as equal channel angular (ECA) extrusion. The fatigue properties of these materials which are characterized by ultra-high strength are of considerable interest and have so far not been investigated in sufficient detail. One major problem encountered especially in strain-controlled tests is the pronounced instability of the strongly strain-hardened dislocation microstructure under conditions of cyclic deformation. Thus, impressive improvements of the fatigue strength have so far been limited mainly to stress-controlled fatigue (Wöhler/S-N plot), whereas strain-controlled fatigue (Manson-Coffin plot) tends to reduce the fatigue strength. The main reasons lie in marked cyclic softening related to localized grain coarsening and large-scale shear banding. Nonetheless, it appears worthwhile to attempt to improve this behaviour by better control of ECA extrusion and by suitable annealing treatments. Also, it should be explored which UFG materials have the best potential for enhancement of the fatigue properties. The problems outlined above can only be mastered by striving for a better understanding of the microstructural mechanisms through systematic mechanical and microstructural studies on different types of materials. The current status of these developments will be outlined in this presentation.

2:00 PM **B2.2**

CONSTITUTIVE MODELLING OF PLASTICITY OF NANO-CRYSTALLINE METALLIC MATERIALS. Yuri Estrin^a, Hyoung Seop Kim^b, Mark Bush^a. ^aDepartment of Mechanical and Materials Engineering, University of Western Australia, Nedlands, WESTERN AUSTRALIA; ^bDepartment of Metallurgical Engineering, Chungnam National University, Taejon, KOREA.

A constitutive model describing deformation behaviour of ductile nanocrystalline materials will be presented. The model is based on a phase mixture rule implying that the interiors of the grains and the grain boundaries can be considered as distinctly different phases of the material. A superposition of the dislocation glide and the diffusion mechanisms was assumed for the grain interior phase, while diffusion controlled plastic flow was considered as the mechanism operative in the grain boundary phase. Numerical simulation results obtained are in good accord with experimental data, particularly with regard to deviations from the Hall-Petch relation. Effects of porosity, e.g. in cold compaction of ultra-fine powders, will also be discussed.

2:15 PM **B2.3**

DEFORMATION MECHANISMS IN NANOCRYSTALLINE TiAl₃-X AND NiAl INTERMETALLIC ALLOYS. O. Coreno-Alonso, N. Mota-Solis, J.G. Cabanas-Moreno, H.A. Calderon, Dept. Ciencia de Materiales, ESFM-IPN, MEXICO; M. Umemoto, Toyohashi University of Technology, Toyohashi, JAPAN.

The deformation mechanisms in the nanocrystalline intermetallic alloys TiAl₃-X and NiAl have been experimentally investigated by

means of mechanical testing (compression and tension) at different temperatures ranging from 300 to 873 K and observation in transmission and scanning electron microscopy. These alloys were produced by mechanical milling in an inert atmosphere and consolidated by a spark plasma assisted technique. Single phase materials with ordered structures were obtained after sintering, TiAl₃-X alloys have an L1₂ structure and NiAl alloys have the B2 structure. The resulting average grain sizes (d) vary between 30 nm (Al₃Ti) and 150 nm. The value of d depends strongly on the sintering temperature. In the case of TiAl₃-X, the grain size was varied between 70 and 150 nm by means of heat treatment. The results of mechanical testing show relatively high yield stresses of approximately 1 GPa and a ductility that depends on the alloy system. As sintered TiAl₃-X shows no ductility, however heat treated alloys can be deformed in compression up to 15%. Crack formation parallel to the stress axis is commonly observed but such cracks grow slowly as expected for ductile materials. A finite strain hardening rate is found in all cases and at all testing temperatures investigated, indicating that most likely dislocations are involved in the deformation mechanism. However clear shear bands are also observed at angles between 45 and 55 degrees with respect to the loading axis indicating that the deformation takes place by other mechanisms as well. Serrated flow is characteristic of the deformation of these materials at higher testing temperatures. NiAl alloys were sintered at different temperatures and also show high values of yield strength. Nevertheless ductility of around 5% can only be observed at temperatures higher than 573 K. Analysis of the measured mechanical properties (strain hardening coefficient and yield stress dependence with temperature) will be used for discussion of the different contributions to the deformation of the present samples.

2:30 PM B2.4

UNIQUE MECHANICAL BEHAVIOUR OF SPD NANO-STRUCTURED ALLOYS. Ruslan Valiev, Aviation Technical Univ, Inst of Physics of Advanced Materials, Ufa, RUSSIA.

When severe plastic deformation (SPD) is applied to metallic alloys, the processed bulk materials can possess both very small grains with a mean grain size less than 100nm and highly dispersive particles of second phases having a size of about 20nm. This paper deals with a fabrication of several such Al and Cu-based alloys and nanocomposites, using SPD processing and their thorough microstructural characterization by TEM/HREM and X-ray, including SAXS. An especial attention is focussed on tensile mechanical behaviour of these SDP processed alloys at room and elevated temperatures. It is shown that the materials can demonstrate the unique combination of very high strength, ductility and superplasticity. Mechanisms of such unusual behaviour are studied through experiment and modeling and discussed.

3:15 PM *B2.5

MECHANICAL PROPERTIES OF ELECTRODEPOSITED NANOCRYSTALLINE COPPER. L. Lu, M.L. Sui, S.X. Li, and K. Lu, State Key Laboratory for RSA, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, CHINA.

In order to reveal the intrinsic mechanical properties of nanocrystalline (nc) metals and to understand their grain size effect in the nanometer scale, "ideal" nc specimens, i.e., porosity-free, impurity-free, and bulk in size, are necessary. In this work, we prepared a bulk nc copper specimen (grain size of 30 nm) with a high purity and full-density by means of electrodeposition. The mechanical properties of the nc Cu sample were investigated in terms of experimental measurements, including tensile properties (strength, ductility, and the strain rate effect) measured by using conventional-sized tensile specimens, extensibility at room temperature upon rolling, creep and fracture behaviors. Compared with the conventional nc Cu made by means of consolidation of ultrafine particles or ball-milling, our nc Cu specimens exhibit very different mechanical properties. A high tensile ductility with an elongation-to-fracture of as much as 50% was observed, which is much larger than that reported in the literature for the nc Cu with the same grain size. An extreme extensibility (as much as >5000% elongation) was obtained in cold-rolling at room temperature. These extraordinary mechanical behaviors of the nc Cu, which will be discussed in terms of the microstructure characteristics and other relative measurement results, shed new light on the deformation mechanism of nc materials and demonstrate new possibilities for scientific and technological advancements with nc materials.

3:45 PM B2.6

ELASTIC SOFTENING OF NANOCRYSTALLINE Pd-H DUE TO HYDROGEN REDISTRIBUTION AT GRAIN BOUNDARIES. P. Bunzel, C. Lemier, J. Weissmueller, Forschungszentrum Karlsruhe, Institute for Nanotechnology, Karlsruhe, GERMANY, and Universitaet des Saarlandes, Technische Physik, Saarbruecken, GERMANY.

At thermodynamic equilibrium, grain boundaries in nanocrystalline (n-) Pd-H alloys are known to be enriched in hydrogen, relative to the bulk. The enrichment causes local stresses and strains which, in a phenomenological approach, are described by stretch⁽¹⁾, an expansion of the grain boundaries along their normal, and by interface stress which must be balanced by opposite stresses in the bulk⁽²⁾. The interaction between composition and stress has important consequences for the phase diagram, such as a lowering of the critical point of the miscibility gap⁽³⁾. We have previously measured the interface stress and stretch in n-Pd-H⁽⁴⁾ and report here results of a study of the macroscopic elastic constants of the system, which may provide information on the stress-strain relation at the grain boundaries. Besides two slow processes, Coble creep and stress-driven exchange of hydrogen between the alloy and the external atmosphere, we find a softening of the spontaneous Young's modulus in n-Pd-H relative to pure n-Pd. We propose that the excess hydrogen is located in the first few interatomic planes adjacent to the grain boundaries, and that softening results from the additional compliance due to hydrogen redistribution across the boundary plane, from the layer in one of the two adjacent crystals to the other. We show that a driving force for this process follows from the difference in the elastic constants in the two (elastically anisotropic) adjacent crystallites. We present measurements of the width of the enriched layers by analysis of x-ray atomic density distribution functions for n-Pd-H.

¹M.E. Gurtin, J. Weissmueller, F. Larche; Phil. Mag. A 78 (1998), 1093. ²J. Weissmueller and J.W. Cahn; Acta mater. 45 (1997), 1899. ³J. Weissmueller and C. Lemier, Phil. Mag. Lett. 80 (2000), 411. ⁴J. Weissmueller and C. Lemier, Phys. Rev. Lett. 82 (1999), 213.

4:00 PM B2.7

EFFECT OF GRAIN SIZE DISTRIBUTION ON TENSILE PROPERTIES OF ELECTRODEPOSITED NANOCRYSTALLINE NICKEL. F. Ebrahimi, Z. Ahmed and K.L. Morgan, MS&E Department, University of Florida, Gainesville, FL.

We have produced dense nanocrystalline nickel using electrodeposition techniques. Various grain size distributions were obtained by changing the deposition parameters. The deposits are disc shape and have a diameter of 35mm and a thickness in the range of 25-45 micrometer. The tensile properties were measured using dog-bone specimens with a 10-mm gage length. Tensile strength values in the range 700 to 1700 MPa and maximum uniform plastic strains in the range of 0.5 to 5% were obtained at room temperature. The grain size distributions were measured using transmission electron microscopy techniques. The grain size varied from a few nanometers to over 100nm with average grain diameters in the range of 25 to 50nm. In this paper, the effects of grain size distribution on strain hardening behavior and tensile plastic instability are discussed and a model for deformation of nanocrystalline metals is suggested.

4:15 PM B2.8

MECHANICAL PROPERTIES OF NANOCRYSTALLINE METALS IN RELATION TO THEIR MICROSTRUCTURE. F. Dalla Torre¹, H. Van Swygenhoven¹, R. Schaeublin², M. Victoria², W. Wagner¹. ¹Paul Scherrer Institut, SWITZERLAND; ²EPFL Lausanne, Dept. of Fusion Technology CRPP, SWITZERLAND.

Mechanical properties of nanocrystalline Cu, Ni, Pd and Ni₃Al made by various synthesis techniques, such as IGC, Electrodeposition and Ball milling are presented. Due to the fact that the microstructure and chemical impurities of the materials, depend strongly on the synthesis method, e.g. voids in as compacted nanopowder or texture in electrodeposited samples, a detailed analysis of the various materials has been made using x-ray diffraction, conventional TEM and HREM techniques. Room temperature tensile tests made by a Microtensile Testing Machine for small 3 mm long specimens are performed. The change in microstructure before and after deformation and heat treatment is investigated to deduce information about the main deformation mechanisms taking place in nanocrystalline materials. The grain interior, its size and dislocation density and especially the character of the grain boundaries are studied using HRTEM for this purpose.

4:30 PM B2.9

COMPUTER SIMULATION OF MISFIT DISLOCATION MOBILITY IN Cu/Ni AND Cu/Ag INTERFACES. Richard J. Kurtz, Pacific Northwest National Laboratory, Richland, WA; Richard G. Hoagland, Los Alamos National Laboratory, Los Alamos, NM; Howard L. Heinisch, Pacific Northwest National Laboratory, Richland, WA.

Nanolayered bimetallic composites of semicoherent Cu/Ni and Cu/Ag prepared by co-deformation or physical vapor deposition techniques display near theoretical tensile strengths and also substantial ductilities. Arrays of misfit dislocations exist in semicoherent interfaces to accommodate lattice parameter mismatch between the layers. The mobility of misfit dislocations and their interaction with

glide dislocations are reported here from the results of a molecular dynamics study of cube-on-cube oriented Cu/Ni and Cu/Ag interfaces. The threshold shear stress to cause misfit dislocations to move is determined by observation of misfit response to an applied shear stress and by elastic band methods. The effect of distance from the chemical interface on misfit mobility is examined and compared to the glide resistance of perfect dislocations on {100} planes in pure Cu, Ni and Ag. Finally, the influence of the interface gamma surface on misfit core width and stress to move a misfit is computed and related to misfit mobility.

4:45 PM B2.10

INTERFACES IN NANOSTRUCTURED MATERIALS: GEOMETRIC AND CONTINUUM MODELS. Ilya Ovid'ko, Inst of Problems of Mechanical Engineering, Russian Academy of Sciences, St. Petersburg, RUSSIA.

This presentation briefly reviews geometric and continuum models that describe the specific structural and behavioral peculiarities of interfaces (intergrain and interphase boundaries) in nanostructured materials. The effects of interfaces on stress distributions and plastic deformation processes in nanostructured materials are discussed. Geometric models of solid/solid interfaces are concerned with their symmetries imposed by translational and rotational symmetries of adjacent solids. Continuum models use results of geometric and/or computer models in description of interfaces and interfacial defects as sources of internal stress fields and carriers of mechanical, transport and other properties of solids. In particular, geometric and continuum models of interfaces are very important in description of the relationships between the structure and the outstanding properties of nanostructured materials where the volume fraction of interfaces is extremely high (see, e.g., I.A. Ovid'ko, in: Nanostructured Films and Coatings. NATO Science Series. 3. High Technologies. Vol. 78, edited by G.-M. Chow, I.A. Ovid'ko and T. Tsakalakos, pp. 231-246). This is related to the fact that the effects of interfaces on structural stability, mechanical and other properties of nanostructured materials often can not be identified with the help of contemporary experimental research methods, in which case results of theoretical models are highly interesting for optimization of nanotechnologies. This review focuses on geometric and continuum models which describe the following: (i) Interphase boundaries and stress distributions in nanocrystalline films and nano-scale multilayer coatings. (ii) Grain boundaries and grain boundary defects in nanostructured materials. (iii) The role of grain boundaries in plastic deformation processes in nanocrystalline films and bulk solids.

SESSION B3: POSTER SESSION
Tuesday Evening, November 28, 2000
8:00 PM
Exhibition Hall D (Hynes)

B3.1

Abstract Withdrawn.

B3.2

MICROSTRUCTURE CHARACTERIZATION OF MECHANICALLY MILLED NANOGRAINED PrCo POWDERS WITH HIGH COERCIVITY. Yong Zhang, Zhongmin Chen, George C.

Hadjipanayis, Department of Physics and Astronomy, University of Delaware, Newark, DE.

Recently, PrCo₅-based nanograins have been developed using mechanical milling techniques. This produces high coercivity. In this article, the microstructure, magnetic domain structure, and magnetic properties of high-coercivity PrCo₅-based Pr_xCo_{100-x} (x=15.4-20.5) powders synthesized by mechanical milling and subsequent annealing were systematically studied as a function of Pr content, using TEM, lattice imaging, nanodiffraction, Lorentz imaging and magnetometry techniques. Microstructural studies reveal that a uniform nanoscale PrCo₅/Pr₂Co₁₇ microstructure with an average grain size of about 15-30 nm is developed in powders with increasing Pr content up to 19 at.%, which shows a peak coercivity of 24.1kOe. The volume fraction of the Pr₂Co₁₇ decreases with Pr content and a nearly single PrCo₅ structure is obtained in Pr₁₉Co₈₁ powders. When the nanodiffraction results are correlated with those of lattice imaging, it is found that further increase in the Pr content leads to the presence of the less-hard Pr₂Co₇ phase in the form of large grains of about 50 nm, resulting in lower coercivities. Lorentz imaging shows that the magnetic hardening originate from the high anisotropy field of the PrCo₅ phase and the uniform nanoscale microstructure developed by the processing used. This work is supported by Air Force Office of Scientific Research under Grant No. MURI F49620-96-1-0434.

B3.3

FEMTOSECOND ULTRASONICS FOR THE CHARACTERI-

ZATION OF LAYERED MICRO - AND NANOSTRUCTURES.

J. Vollmann, D. Profunser, J. Dual, Center of Mechanics, ETH Zurich, Zurich, SWITZERLAND.

Mechanical waves of few nanometers wavelength are excited by a Titanium sapphire laser, generating laser pulses of less than 70 femtoseconds at a repetition rate of 80 MHz. Thermal stress within an absorption layer of 10 to 20 nanometers transforms the energy of the laser pulse into mechanical waves. The pulsed laser beam is split into a pump beam, carrying approx. 90% of the energy and a 10% probe beam by a partly transmitting mirror. The pump pulses are used to excite the mechanical waves whereas the probe pulses are used to detect optical effects at the thin film surface which are caused by the echoes of the initial pulse. The time shift between a single pump-and-probe-pulse-pair is established by a variable optical path length of the pump beam. A dual frequency modulation technique is introduced in order to eliminate optical cross talk between the excitational pump pulse and the measuring probe pulse. Up to the 5th acoustic echoes in an aluminum film of 50 nm thickness on a sapphire substrate have been measured. Experimental results of further metallic thin-film substrate combinations are compared with numerical simulations obtained by an elastodynamic finite integration technique. Several approaches describing the opto-thermo-mechanical interaction are compared and discussed. Further directions of the on going research projects are presented.

B3.4

MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF Cu-Ag AND Cu-Co NANOCOMPOSITES SYNTHESIZED BY HIGH-ENERGY BALL-MILLING. S. Zghal, F. Wu, P. Bellon, Dept. of Materials Science and Engineering and Frederick Seitz Materials Research Laboratory, University of Illinois at Urbana-Champaign, Urbana, IL.

It is well documented that low-temperature ball-milling can force immiscible elements, e.g., Cu-Ag or Cu-Co, into solid solution. A slight increase on the milling temperature however results in a two- or even three-phase coexistence. This coexistence takes place at a nanometric scale and offers a direct route to synthesize nanocomposite materials. In this paper, we characterize these nanocomposites and relate their nanostructure to their hardness. Using transmission and scanning transmission electron microscopy (TEM, STEM), combining different analysis techniques (chemical analysis by EDS, nanodiffraction and Z-contrast imaging), the microstructure of the ball-milled powders is studied in detail. Low temperature milling results in the formation of a strong deformation texture, with a grain size in the 100 nm range, and in the presence of strong small scale (≈ 5 nm) distortions due to a large density of dislocations ($\approx 10^{13}$ cm⁻²), as revealed by high resolution TEM. When the milling is performed at elevated temperatures, decomposition takes place at a 10 to 30 nm scale and results in smaller grains with an almost isotropic orientation. Interestingly enough, only one phase appears to be percolated, and it is the mechanically soft phase, i.e., the Ag-rich phase in the Cu-Ag system and the Cu-rich phase in the Cu-Co system. Thermal annealing can also be used to induce nanoscale decomposition, but in this case both phases seem to be connected. The hardness of these nanostructures is measured by nanoindentation. Hardness values close to the theoretical shear strength are measured, i.e., up to 6 GPa in the Cu-Ag system. It is also observed that while the solid solution obtained by low temperature ball-milling exhibits already a large nanohardness (4 GPa in the Cu-Ag system), the formation of a nanocomposite with two connected phases leads to the highest strength in these materials.

B3.5

Abstract Withdrawn.

B3.6

HIGH GRAIN SIZE STABILITY OF A NANOCRYSTALLINE Al PREPARED BY MECHANICAL ATTRITION. Fei Zhou, Jongsang Lee, Enrique J. Laverna, Univ of California at Irvine, Dept of Chemical and Biochemical Engineering and Materials Science, Irvine, CA.

The grain growth in a nanocrystalline (nc) Al was studied using X-ray diffraction, transmission electron microscopy, differential scanning calorimetry. The nc Al sample with an average grain size of about 26 nm was produced by mechanically milling of pure elemental Al blends at the cryogenic temperature of -190°C. Grain growth kinetics was analysis using two different models of grain growth, one of which takes pinning forces on the grain boundaries into account. The time exponent n from $D^{1/n} - D_0^{1/n} = kt$ for the nc Al was found to be 0.04~0.28 in a range of 0.61 to 0.83 of melting temperature (T_M), exhibiting the same general trend as high purity conventional polycrystalline Al with n increasing toward 0.5 as annealing temperature increases. The average grain size in the sample remains to be less than 100 nm even after annealing at temperatures up to

0.80 T_M for relatively long times (e.g. 200 minutes). The activation energy for grain growth in the nc Al was determined to be about 79 KJ/mol at the low temperature range (0.61~0.78 T_M) and about 112 KJ/mol at the high temperature range. Possible mechanism for the high grain size stability was discussed.

B3.7

STRUCTURAL AND ELECTRONIC PROPERTIES OF GOLD NANOWIRES GENERATED BY MECHANICAL STRETCHING. Varlei Rodrigues^{1,2}, Daniel Ugarte¹. ¹Laboratório Nacional de Luz Síncrotron, Campinas, BRAZIL; ²Instituto de Física Gleb Wataghin - UNICAMP, Campinas, BRAZIL.

Metallic nanowires display interesting quantum phenomena that may be exploited to generate novel electronic devices. Recently, a great deal of effort has concentrated in the electrical measurements of ballistic nanocontacts, where the conductance is expected to be quantized. In this work, we have studied the structural properties of gold nanowires by in situ experiments in a high resolution transmission electron microscope (HRTEM - JEM 3010 UHR, 300 kV,) and their electronic properties using an ultra-high-vacuum mechanically controllable break junction (UHV-MCBJ). HRTEM atomic lattice images and time resolved observations have been used to study the crystallographic orientation effects in the nanocontact structure and dynamical stretching. We have observed that just before rupture the nanowires are crystalline and adjust their atomic arrangement such that one of three main gold zone axes ([111], [100] and [110]) lies approximately parallel to the elongation direction. Nanocontacts following [100] and [111] directions display a bi-pyramidal shape and evolve gradually to form one-atom-thick wires. On the other hand, nanowires along [110] axes show rod-like morphology and break abruptly when they are a few atoms thick. We have developed a model for the three possible atomic configurations and, predicted their conductance evolution and occurrence probability. We have correlated this model with the UHV-MCBJ conductance measurements and obtained a remarkable agreement. This is an important result, because it allows the association of nanowire structure with the conductance behavior.

B3.8

SIMULATION OF POSITRON CHARACTERISTICS IN NANOCRYSTALLINE MATERIALS. Jan Kuriplach, Charles Univ, Dept of Low Temperature Physics, Prague, CZECH REPUBLIC; Steven Van Petegem, Danny Segers, Charles Dauwe, Ghent Univ, Dept of Subatomic and Radiation Physics, Ghent, BELGIUM; Marc Hou, Free Univ of Brussels, Physics of Irradiated Solids, Brussels, BELGIUM; Eugenij E. Zhurkin, St. Petersburg State Technical Univ, Dept of Experimental Nuclear Physics, St. Petersburg, RUSSIA; Helena Van Swygenhoven, Paul Scherrer Institute, Villigen, SWITZERLAND; Alvaro L. Morales, Univ of Antioquia, Dept of Physics, Medellin, COLOMBIA.

Positron annihilation spectroscopy is a well established technique to study defects with the electron (atomic) density less than average. Theoretical calculations of the positron response to different types of defects can be very helpful in order to interpret properly experimental data. In this contribution a new computational technique to determine positron properties in nanocrystalline materials is presented. In such calculations realistic models of n-materials obtained using molecular dynamics are employed. The new technique is based on the so-called atomic superposition method where atomic densities are superimposed in a selected region of the model (virtual) sample to approximate the electron density of the system. For n-materials, however, an appropriate 'boundary condition' has to be applied to the positron potential before solving the Schrödinger equation for positrons. The regions of interest comprise grain boundaries including triple points, nano-voids, and bulk-like regions. We study the virtual samples of n-Cu, n-Ni, n-Ni₃Al, and n-NiAl, for which we calculate positron lifetimes and positron binding energies corresponding to defects located in selected regions of the samples. Obtained positron lifetimes are compared to the available experimental data.

B3.9

POSITRON LIFETIME MEASUREMENTS IN NANOSTRUCTURED Ni-Al SAMPLES. Steven Van Petegem, Danny Segers, Charles Dauwe, Ghent Univ, Dept of Subatomic and Radiation Physics, Ghent, BELGIUM; Florian Dalla Torre, Helena Van Swygenhoven, Paul Scherrer Inst, Villigen, SWITZERLAND; Mohammed Yandouzi, Dominique Schryvers, Gustaaf Van Tendeloo, Univ of Antwerp, Centre for Electron Microscopy and Materials Science, Antwerp, BELGIUM; Jan Kuriplach, Charles Univ, Dept of Low Temperature Physics, Prague, CZECH REPUBLIC; Marc Hou, Free Univ of Brussels, Physics of Irradiated Solids, Brussels, BELGIUM; Eugenij E. Zhurkin, St. Petersburg State Technical Univ, Dept of Experimental Nuclear Physics, St. Petersburg, RUSSIA.

Positron lifetime spectroscopy is an effective tool to study various

types of defects in materials including nanostructured ones. The size of free volumes associated with defects can be estimated using the lifetime components found in measured spectra. Positron lifetime experiments are performed on nanocrystalline Ni_xAl_{1-x} ($x = 0.73$ and $x = 0.62$) synthesized by the inert-gas condensation technique using different production parameters such as temperature and gas pressure. The samples are characterized by means of X-ray diffraction, electron diffraction and microscopy techniques as well as density measurements. In the lifetime spectra we observe three lifetime components. The first lifetime corresponds to positrons trapped in open volumes of the order of a mono-vacancy, which can be situated in the grains and in the grain boundaries. The second lifetime corresponds to the annihilation of positrons trapped in nanovoids situated at the intersection of interfaces. The size of these nanovoids can be varied by thermal treatment. A third nanosecond lifetime from the annihilation of ortho-positronium, formed in even larger open volumes, is also present. These lifetimes are compared with the results of simulations of positron lifetimes in virtual Ni-Al samples obtained using molecular dynamics.

B3.10

LOGARITHMIC RELAXATION OF ELECTRICAL RESISTANCE IN PLASTICALLY DEFORMED Au₈₀Fe₂₀ AT ROOM TEMPERATURE. Paolo Allia, Marco Coisson, Vincenzo Selvaggini, DIFIS-DISPE, Politecnico di Torino and INFM, Torino, ITALY; Paola Tiberto, Franco Vinai, IEN Galileo Ferraris and INFM, Torino, ITALY; Marcello Baricco, Enrica Bosco, Diego Falletti, Dip. IFM, Università di Torino and INFM, Torino, ITALY.

Rapidly solidified Au₈₀Fe₂₀ ribbons (thickness 60 μ m, width 2 mm) were prepared from a homogenized master alloy by planar flow casting in controlled atmosphere on a rotating drum. Magnetic measurements performed by a VSM showed that the magnetic response is described by a paramagnetic Curie-Weiss law at room temperature (RT) and above, indicating that the alloy is still a perfect solid solution of the two elements, as in the master alloy. The electrical resistance was measured at RT by the 4-contact method with pressure contacts using a digital bridge. The RT electrical resistivity may reach values as high as 500 $\mu\Omega$ ·cm in the considered ribbons. A logarithmic relaxation of the electrical resistance is observed at RT in all studied samples after severe plastic deformation. Typically, a 0.1% reduction of the initial R value is measured after one full day. Such a relaxation may be ascribed to deformation-enhanced compositional ordering processes of Fe atoms. In fact, these systems display a tendency towards segregation of Fe nanoclusters by effect of thermal treatments and/or plastic deformation. Magnetic measurements and X ray diffraction indicate that these nanoclusters may contain either bcc or fcc Fe. On the other hand, no relevant deformation-enhanced grain boundary re-arrangements are expected to occur, because these ribbons are characterized by the presence of large, stable grains. The electrical resistivity of Au_{100-x}Fe_x alloys is very sensitive to the Fe concentration; a resistivity change as observed is compatible with the loss of about 100 ppm of Fe atoms initially present in the solid solution. The electrical resistivity decrease related to Fe reduction in the solid solution turns out to overcome the slight resistivity enhancement originating from the nucleation of Fe clusters.

B3.11

EFFECT OF DEPOSITION PARAMETERS ON TENSILE PROPERTIES OF PULSE-PLATED NANOCRYSTALLINE NICKEL. K.L. Morgan, Z. Ahmed and F. Ebrahimi, MS&E Dept., University of Florida, Gainesville, FL.

Electrodeposition is a viable method for fabricating nanocrystalline metals. We have been able to fabricate nanocrystalline nickel through a control of deposition parameters and without the use of additives for grain refinement. The deposition was conducted using a sulfamate-based solution in a galvanostatic mode. The peak current density during pulse plating was varied between 0.025 A/cm² to 0.2 A/cm². The microstructure of the deposits was characterized by x-ray diffraction and transmission electron microscopy techniques. Tensile testing was conducted using dog-bone shaped specimens with a gage length of 10-mm. Scanning electron microscopy was applied for studying surface roughness and fracture behavior of the deposits. This paper presents the effect of current density on microstructure and tensile properties of electrodeposited nanocrystalline nickel produced via the pulse plating technique.

B3.12

DEFORMATION OF NANOCRYSTALLINE Ni INVESTIGATED BY TEM SIMULATION COUPLED TO MD SIMULATION. R. Schaublin, Fusion Technology - Materials, Centre de Recherches en Physique des Plasmas, Ecole Polytechnique Federale de Lausanne, Villigen PSI, SWITZERLAND; H. Van Swygenhoven, Paul Scherrer Institut, Villigen PSI, SWITZERLAND.

EM image simulations are used together with molecular dynamic

simulations in order to investigate the grain boundary structure of nanocrystalline Ni and Cu samples. The fcc material is a polycrystal which grains have a mean grain size between 5 to 12 nm and are nucleated from different seeds with random locations and orientations. TEM images are simulated using the multislice method. The dynamical approach based on the multislice technique gives access to the high resolution TEM image, diffraction contrast image and the diffraction pattern. The approach is applied in the analysis of the structure of the grain boundaries. This allows to simulate the bright field images to identify dislocations in the grain boundaries using classical TEM techniques. The results are discussed as a function of grain size and correlated to experimental images.

B3.13

EXTENSION OF HIGH CYCLE FATIGUE LIFE BY THE FORMATION OF NANO-SIZED MARTENSITE PARTICLES AT INTERSECTIONS OF DISLOCATIONS IN AN AUSTENITIC STAINLESS STEEL. Tomonari Inamura, Masayuki Shimajo, Kazuki Takahima, Yakichi Higo, Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, JAPAN.

Fatigue of metallic materials is mainly due to the accumulation of irreversible motions of dislocations under cyclic loading. Many strengthening methods for the improvement of tensile strength have been proposed. However, an increase in tensile strength generally decreases the ductility of the materials and is not very effective on improvement of fatigue properties. Then, pinning dislocations at their intersection after cold-work is considered to be effective for fatigue strengthening. The motion of pinned dislocations are expected to be bowing motion and the accumulation of irreversible motions of dislocations is supposed to be suppressed under cyclic stress. The effectiveness of this strengthening method for the fatigue life extension was investigated using an austenitic stainless steel in this study. It is well known that burst-like alpha-martensitic transformation occurs in austenitic stainless steels at Ms temperature during cooling. According to Bogers and Burgers, two {111} < 112 > shears transform f.c.c.-stacking into b.c.c.-stacking. Thus, it is suggested that b.c.c.-like stacking regions exist at intersections of partial dislocations and may transform easily into fine alpha-martensite particles at a certain cryogenic temperature above Ms temperature. By this cryogenic treatment, partial dislocations are supposed to be pinned at their intersections. A commercially available hot-rolled 316-type austenitic stainless steel (Fe-17Cr-10Ni-2Mo) was cryogenically treated above Ms temperature after cold-work. Transmission electron microscopy observation revealed that nano-sized alpha-martensite particles, with a diameter of 5 nm, were formed by the cryogenic treatment at 195 K. High cycle fatigue lives of the specimen was extended by the cryogenic treatment at 195 K without any decrease in ductility, compared to those of non-treated specimens. Effects of nano-sized alpha-martensite on the motions of dislocations under cyclic stress and on the progression of fatigue damage accumulation are discussed.

B3.14

MONTE CARLO SIMULATION OF GRAIN BOUNDARY SLIDING AND MIGRATION: EFFECT OF VACANCIES. Peter Ballo, Department of Physics, Slovak Technical University, Bratislava, SLOVAK REPUBLIC; Nicholas Kioussis and Gang Lu, Department of Physics, California State University, Northridge, CA.

We have carried out Monte Carlo (MC) simulations using the embedded atom potential to study the sliding and migration of the $\Sigma 5$ [001] (210) tilt grain boundary (GB) in aluminum and the effect of vacancies on the sliding properties. We find that the simulated annealing allows the system to gradually anneal to a global-minimum configuration, thus increasing the number of migrations and reducing the GB sliding energy barriers to about a factor of three compared to the corresponding "static" values. The distribution of atomic energies as a function of GB displacement, provide insight into which atoms are responsible for the GB migration. The vacancy formation energy is found to be lower when the vacancy is placed on the first layer to the boundary, in excellent agreement with *ab initio* electronic structure calculations. The sliding and migration properties depend very sensitively on the position of the vacancy in the GB core.

B3.15

BERYLLIUM STRENGTHENING AND STRENGTH ENHANCEMENT BY BIASED DEPOSITION. Alan Jankowski, Lawrence Livermore National Laboratory, Livermore, CA.

A mechanical testing technique is developed to load thin-walled spherical capsules of beryllium under uniaxial tension at constant strain. In addition to the measurement of elastic behavior, application of the tensile load to failure produces yielding and fracture. The capsules are prepared by magnetron sputter deposition of Be and Be alloys onto spherical polymer mandrels. The application of an applied bias to the substrate holder densifies the columnar microstructure and increases the material strength by a factor of three or more. A detailed

assessment of capsule mechanical properties is now available using this testing technique which will facilitate the design of spherical capsules as pressure vessels. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

SESSION B4: MECHANICAL PROPERTIES AND DEFORMATION BEHAVIOR III – MULTILAYERS

Chair: Harriet H. Kung

Wednesday Morning, November 29, 2000
Hampton (Sheraton)

8:30 AM *B4.1

DEFORMATION MECHANISMS IN SINGLE CRYSTAL METALLIC NANOLAMINATES: THEORY AND EXPERIMENT. Peter Anderson, Department of Materials Science and Engineering, The Ohio State University, Columbus, OH; and Tim Foecke, Metallurgy Division, NIST, Gaithersburg, MD.

This presentation details results from a study of deformation and fracture mechanisms that operate in metallic nanolayered materials that closely couples novel experiments with theories. In situ TEM observations of dislocation generation and motion in single crystal Cu/Ni nanolaminates are examined in terms of recently developed models. The experimentally observed generation mechanisms are simulated, and the effect of such microstructural factors as layer thickness and interfacial resistance are examined. Propagation of dislocations within single layers under confined layer slip (CLS) is detailed. The critical conditions for transmission across interfaces are calculated and are found to correlate well to experimental observations. Extensions are made to the onset of bulk yield in metallic nanolaminates as a function of layer thickness.

9:00 AM B4.2

DISLOCATION MODELS FOR STRENGTHENING IN NANOSTRUCTURED METALLIC MULTILAYERS. A. Misra, J.P. Hirth, H. Kung, R.G. Hoagland and J.D. Embury, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Metallic multilayers may be synthesized by physical vapor deposition techniques to have strength levels close to the theoretical strength. These materials are also ideal for investigating the effects of length scales in plastic deformation of metallic materials. Experiments on model systems such as Cu-Cr and Cu-Nb indicate that the strength of these materials increase with microstructural refinement according to the Hall-Petch model. However, as the layer thickness is reduced to the nm-scale, the number of dislocations in pile-up approaches unity and the pile-up based Hall-Petch model ceases to apply. Using dislocation theory, we develop a model to predict the critical values of grain size and layer thickness in a semi-coherent multilayer below which the continuum behavior is not applicable. For nm-scale semi-coherent multilayers, we hypothesize that plastic flow occurs by the motion of single dislocation loops, initially in the softer layer, that deposit misfit-type dislocation arrays at the interface thereby transferring load to the harder phase. The build-up of stress eventually leads to slip in the harder phase, overcoming the resistance from the misfit arrays at the interface. A model is developed within the framework of classical dislocation theory to estimate the strengthening from this mechanism. The model predictions are compared with experimentally measured strengths and observed dislocation substructures. This work is supported by the Office of Basic Energy Sciences, U. S. Department of Energy.

9:15 AM B4.3

CORRELATIONS OF MICROSTRUCTURE AND TEM OBSERVATIONS OF PLASTICITY IN METALLIC NANOLAMINATES. Donald Kramer and Tim Foecke, National Institute of Standards and Technology, Gaithersburg, MD.

It is well documented that nanolaminate materials exhibit enhanced mechanical properties beyond those expected according to rule of mixtures calculations. Several models have been proposed to explain this enhancement of strength, but conclusive experimental verification is hindered by the complex interaction between ingrown defects, in-plane microstructure and compositional modulation. Recently, electrodeposition has been used to fabricate epitaxial nanoscale Cu/Ni metallic multilayers on single crystal (001) Cu substrates. Transmission electron microscopy has been used to characterize the structure of these deposits. The low occurrence of growth defects, planarity of the layered structure and paucity of grain boundaries make this system ideal for studying the influence of compositional modulation on mechanical properties. The effects of modulation amplitude and interfacial misfit dislocations are demonstrated through observations of dislocation generation and motion during in

situ TEM straining experiments. These results are correlated with hardness measurements from nanoindentation testing.

9:30 AM **B4.4**

SUPERELASTIC DEFORMATION OF ADAPTIVE NANO-COMPOSITES. Julia Slutsker, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, MD; Alexander L. Roytburd, Dept Materials and Nuclear Engineering, Univ of Maryland, College Park, MD.

We model the superelastic deformation of multilayer nanocomposites consisting of thin adaptive layers of a shape memory alloy separated by the thin layers of a passive metal. It is shown that the martensitic transformation in thin constrained single crystalline layers proceeds through controlled evolution of polydomain microstructure resulting in superelastic deformation. The thermodynamic analysis of the evolution of an equilibrium microstructure in the active layers shows that the special composite architecture leads to the reversible deformation without static hysteresis. Strain-rate effects associated with kinetic hysteresis are analyzed by using the methods of irreversible thermodynamics.

10:15 AM ***B4.5**

ATOMIC SIMULATION OF THE RESISTANCE TO SLIP TRANSMISSION BY INTERFACES IN MULTILAYER METALLIC SYSTEMS. R.G. Hoagland, H. Kung, A. Misra, Los Alamos National Laboratory, Los Alamos, NM; J.D. Embury, McMaster University, Hamilton, Ontario, CANADA.

The strength of metallic nanolayered composites, even when constructed of combinations of relatively soft fcc metals, such as Cu, Ag, and Ni, can be very high, often exceeding 1 GPa while displaying Hall-Petch strength vs. wavelength (layer thickness) behavior for larger wavelengths (although, in some cases, the strength becomes independent of wavelength or is even observed to decrease when the wavelength becomes quite small). In typical systems, the strength derives from the interfaces which act as fairly strong barriers to slip. There are several contributions to this slip resistance, some of which involve nonlinear processes not amenable to linear elastic descriptions. This paper describes results of atomistic simulations that have been used to examine the interaction of glide dislocations with the structure of coherent and semi-coherent interfaces between two different fcc metals. Several different types of mechanisms are observed and the energetics and stresses needed to operate these mechanisms are discussed. In both types of interfaces, dislocation reactions at interfaces are observed. The misfit dislocations in the semicoherent interfaces, while serving to relax long-range coherency stresses present in thick layers, are particularly interesting because they are observed to both participate in and impede slip transmission. The core structures of misfit dislocations can also play an important role as is suggested by a comparison of Cu/Ag and Cu/Ni systems. This work was supported by the Office of Basic Energy Sciences, U. S. Department of Energy.

10:45 AM **B4.6**

EFFECT OF INTERDIFFUSION ON THE INTERFACE STRESS IN Ag-Ni(111) MULTILAYERED THIN FILMS. D. Josell, J.E. Bonevich, National Institute of Standards and Technology, Gaithersburg, MD; R.C. Cammarata, Johns Hopkins University, Baltimore, MD.

The interface stress is the thermodynamic quantity associated with the reversible interfacial work performed when both phases adjacent to an interface are elastically deformed by application of a uniform, uniaxial strain in the plane of the interface. Interface stresses have been measured using a variety of crystalline multilayered thin films over the past decade. Typically this evaluation involved transmission x-ray diffraction and/or wafer curvature measurements of multilayered thin films attached to thin substrates, the measurements aimed at determining the variation of the in-plane lattice spacings as a function of layer thickness. In most cases, a negative (compressive) value of the interface stress was reported. This is in contrast to the positive values generally obtained from computer simulations. One possible explanation for this discrepancy is the neglect of interdiffusion in modeling experimental results; an unaccounted for change in the reference lattice parameters associated with such alloying would affect the measured in-plane strains, possibly resulting in a sign change for the interface stress. In this study, results from transmission and reflection x-ray diffraction studies of free-standing Ag-Ni (111) textured multilayers fabricated by electron-beam evaporation are reported. These results, the first obtained via this technique, are analyzed to investigate the effect interdiffusion has on the interface stress measurements. When interpreted using a $\sin^2\psi$ analysis to obtain stress-free lattice parameters, the data indicate that interdiffusion must, in fact, be accounted for in evaluating the interface stress. We discuss these results in the context of measurements recently obtained by O. Thomas et al. from in-situ growth studies. We also discuss the associated, implicit assumption

that strains of the out-of-plane lattice parameter near the interface due to the interface stress are solely determined from the interface stress and the elastic moduli of the bulk (i.e., ignoring excess volume associated with the interface).

11:00 AM **B4.7**

MEASUREMENT OF INTERFACIAL FREE ENERGIES IN Ag/Ni MULTILAYERS. A.C. Lewis, A.B. Mann, D. Van Heerden, T.P. Weihs, Johns Hopkins University, Dept of MS&E, Baltimore, MD; D. Josell, National Institute of Standards and Technology, Metallurgy Division, Gaithersburg, MD.

Two types of solid-solid interfaces exist in nanolaminate or dual-phase nanocrystalline materials: grain boundaries within the individual layers and interfaces between the chemically dissimilar layers. Current understanding suggests that the ratio of the free energies for these two interfaces (γ_{gb}/γ_{int}) controls the stability of nanoscale and microscale multilayer materials. A large ratio favors deep grooves and layer pinch-off, while a small ratio favors shallow grooves and stable layers. Given the importance of these free energies in predicting microstructural stability, there is a strong need to measure grain boundary and interface energies. A unique experimental technique for doing so is presented here. Ag/Ni multilayers were deposited on sapphire substrates and the free energies of the Ag/Ni interfaces were determined from the curvature of the film-substrate couple at elevated temperatures (500°C) after equilibration. At these temperatures, deposition and thermal stresses decay to zero with time, but the Ag/Ni interfaces (and their drive to contract the sample) continue to exert a force that bends the substrate, producing the measured curvature. The interface free energy γ_{int} was determined from experiments using specimens with different numbers of layers along with geometric information obtained by transmission electron microscopy. This geometric information includes the average groove angles where the grain boundaries in the layers meet the interfaces between layers, as well as the average dimensions of grains.

11:15 AM **B4.8**

ATOMISTIC SIMULATIONS OF STEPS IN BIMETALLIC INTERFACES. Chuck Henager, Jr., Howard Heinisch, Jr., Rick Kurtz, Pacific Northwest National Laboratory, Richland, WA.

Bimetallic, nanolayered materials formed by PVD processes consist of curved interfaces containing atomic-scale steps. It is important to determine the characteristics of these steps and ledges and their role in the mechanical properties of these nanolayered materials. The results of an atomistic study of the structure and energies of steps on (001) (cube on cube) oriented Cu/Ni and Cu/Ag interfaces are presented. Step height and orientation are systematically varied, and the excess interface energies associated with the steps are determined. In addition, the interaction between steps and interface (misfit) dislocations is explored. The atomistic results are compared to elastic calculations of the forces between steps and dislocations.

11:30 AM **B4.9**

COMPUTER SIMULATION OF INTERACTION BETWEEN DISLOCATIONS AND MISFIT INTERFACE. Antti Kuronen, Kimmo Kaski, Leonel Perondi, and Jari Rintala, Laboratory of Computational Engineering, Helsinki University of Technology, Espoo, FINLAND.

Misfit dislocations in a lattice mismatched system may appear either as a consequence of the gliding of threading dislocations, by nucleation of dislocation half-loops from the overlayer surface or by dislocation multiplication. All these mechanisms involve gliding of a dislocation from the substrate or overlayer to or along the misfit interface. While there has been a substantial number of studies concerning static properties of the misfit dislocations there seems to exist very few studies, at the atomic level, concerning the migration of dislocations from the substrate or overlayer to the interface and associated processes. In this work we have investigated the effect of the misfit interface on the dislocations already existing in the system using a two-dimensional Lennard-Jones system and molecular dynamics simulation technique. Since this is an exploratory investigation, we perform simulations in a two-dimensional system, thus avoiding the geometrical complexities involved in the simulation of three-dimensional dislocations while retaining part of the essential features of the problem under consideration. The simulations indicate that the misfit interface attracts only those dislocations that are able to relieve the strain. Moreover, the interaction between dislocations was observed to cause dislocation reactions resulting in dislocations that have the optimum Burger's vector for strain relief. The reactions between dislocations proceed through creation of two partial dislocations and a stacking fault between them and a slip of atomic rows. For large mismatches dislocation nucleation from the overlayer edges and from the free surface of the overlayer was also observed.

11:45 AM B4.10

COHERENCY STRAIN AND A NEW YIELD CRITERION. N.B. Jayaweera, Dept of Physics, Queen Mary and Westfield College, University of London, London, UNITED KINGDOM; J.R. Downes, Dept of Physics, Queen Mary and Westfield College, University of London, London, UNITED KINGDOM; A.J. Bushby, Dept of Materials, Queen Mary and Westfield College, University of London, London, UNITED KINGDOM; P. Kidd, Philips Analytical Research Centre, Redhill, UNITED KINGDOM; A. Kelly, Dept of Materials Science and Metallurgy, University of Cambridge, Cambridge, UNITED KINGDOM; and D.J. Dunstan, Dept of Physics, Queen Mary and Westfield College, University of London, London, UNITED KINGDOM.

We have reported results on the onset of plasticity in semiconductor-strained-layer superlattices, using nanoindentation with spherical indenters to observe the full stress-strain curve. These structures have alternating layers of a few nanometres thickness with strains of opposite sign. By balancing the strains, superlattices a few microns deep can be grown. The yield pressure is reduced by as much as a factor of two by the presence of the coherency strain. Varying the thicknesses and strains of the layers enables us to show that both sets of layers, compressive and tensile, influence the yield pressure. This requires that a yield criterion must be satisfied over a finite volume, large enough to include layers of both signs. This is a completely different approach to pointwise yield criteria, such as von Mises, and is necessary to describe yield within inhomogeneous strain fields. We show that the relevant yield criterion for our experimental data is the rate of change of elastic strain energy with plastic relaxation, integrated over a volume of the order of a micron across. The result has implications for finite-element modelling of point contact, and for the design and understanding of materials with coherency-strained microstructure.

SESSION B5/W4: JOINT SESSION

Chairs: Daryl C. Chrzan and Richard G. Hoagland
Wednesday Afternoon, November 29, 2000
Room 310 (Hynes)

1:30 PM *B5.1/W4.1

THE INVERSE HALL-PETCH EFFECT - FACT OR ARTIFACT? Carl Koch, North Carolina State University, Dept. of MS&E, Raleigh, NC.

The first observations of Chokshi et al (1) of softening with decreasing grain size for nanocrystalline Cu and Pd stimulated interest in this apparent inverse Hall-Petch behavior for decreasing grain size in the nanoscale regime. However, it was subsequently pointed out that for most of the experiments where inverse Hall-Petch behavior was observed, the grain size was changed by annealing the initially finest grain size specimens to grow the grains. It was suggested that thermally treating nanocrystalline samples may cause changes in the structure (eg. decreases in porosity, changes in grain boundary structure, etc.) along with the grain growth, and these changes may be responsible for the inverse Hall-Petch behavior. In spite of the uncertainty in the experimental evidence for the inverse Hall-Petch effect, a number of mechanisms have been proposed for its occurrence. Molecular dynamics simulations have also predicted the inverse Hall-Petch effect. Since conventional dislocation-induced deformation mechanisms are unlikely at the finest nanoscale grain sizes, changes in the grain size dependence of hardness and strength are not surprising. This paper will critically review the available experimental evidence for the inverse Hall-Petch effect and discuss it in the context of the simulation studies and the various proposed mechanistic models.

1. A.H. Chokshi, A. Rosen, J. Karch, H. Gleiter, Scripta Metall. 23, 1679 (1989).

Support from NSF under grant no. DMR-9871980.

2:00 PM B5.2/W4.2

LARGE TENSILE DUCTILITIES AND ULTRAHIGH YIELD STRENGTHS AT ROOM TEMPERATURE IN NANOCRYSTALLINE FeCo ALLOYS. Chang He Shang, D. Van Heerden, R.C. Cammarata, The Johns Hopkins University, Department of MS&E, MD; C.L. Chien, The Johns Hopkins University, Department of Physics and Astronomy, Baltimore, MD; T.P. Weihs, The Johns Hopkins University, Department of MS&E, Baltimore, MD.

Researchers have pursued nanocrystalline materials for structural applications for more than a decade because materials with grain sizes below 1 micrometer have been predicted to display and, in some case, have shown significant improvements in mechanical properties compared to coarse-grained materials. High yield strengths and superplasticity have been demonstrated, and enhanced ductility has been predicted for nanocrystalline intermetallics. However, while nanocrystalline Cu has shown enhanced ductility at room

temperature, nanocrystalline intermetallics typically fail in the elastic regime with less than 1% strain-to-failure. The extreme brittleness in nanocrystalline intermetallics is generally attributed to flaws or porosity that are produced during fabrication. In this study, we have investigated the mechanical properties and microstructures of fully dense, single-phase nanocrystalline FeCo alloys that were fabricated using bulk processing techniques. The nanostructured alloys possess very attractive mechanical properties at room temperature with tensile yield strengths as high as 2.2 GPa and tensile ductilities ranging from 3% to 18%. Both tensile strengths and hardnesses follow Hall-Petch relationships, and the fracture surfaces show ductile features. Results from TEM, SEM, and X-ray diffraction investigations will be used to describe the microstructure of these flaw-free, nanocrystalline materials.

2:15 PM B5.3/W4.3

DEFORMATION BEHAVIOR AND FAILURE MODE OF CONSOLIDATED FULL-DENSITY NANOPHASE Fe AT QUASI-STATIC AND DYNAMIC STRAIN RATES. D. Jia¹, Y.-M. Wang², K.T. Ramesh^{1,2} and E. Ma¹. ¹Department of Mechanical Engineering, ²Department of MS&E, Johns Hopkins University, Baltimore, MD.

Full density Fe with grain sizes in the nanophase to submicron range has been consolidated from mechanically milled powders. The deformation behavior of such materials, as a function of grain size, strain rate, and temperature, have been studied using quasi-static and high strain rate (Kolsky bar) tests. With ultrafine grain sizes, Fe exhibits high strength, little work hardening, and plastic strains localized in shear bands. Shear banding appears to be the dominant mode from the onset of the plastic deformation in our consolidated materials. Little strain rate sensitivity of the flow stress is observed over a wide strain rate range (up to 5E5/s). These behaviors are contrasted with those of conventional bulk Fe, which shows uniform deformation, significant work hardening, and strong strain rate sensitivity. The underlying deformation and failure mechanisms are discussed based on these observations. With increasing grain sizes and/or temperature, the yield strength decreases and plastic strain increases. These findings are compared with the Hall-Petch relationship, and examined to derive the validity range, in terms of grain size, temperature, strain rate, and particle bonding strength, of different deformation mechanisms (including grain boundary mechanisms). In addition, we comment on the potential advantage of related bcc alloys in military kinetic energy penetrators where the shear banding mode and self-sharpening capability during high rate deformation are desired.

2:30 PM *B5.4/W4.4

THE LIMIT OF GRAIN REFINING STRENGTHENING IN IRON. Setsuo Takaki, Kenji Kawasaki, Kyushu Univ, Dept of MS&E, Fukuoka, JAPAN; Yuuji Kimura, National Research Institute for Metals, Ibaraki, JAPAN.

Mechanical milling (MM) using a high energy ball mill was applied to iron powder to obtain ultra fine-grained structure. The MM treatment causes a marked increase in the hardness of iron powder from Hv_{0.5}GPa to Hv_{9.5}GPa. Initial hardening is due to well-known dislocation strengthening but the latter hardening (above Hv₆GPa) is mainly due to grain refining strengthening. The grain size of iron powder with the hardness Hv_{9.5}GPa was about 25nm. In the Hall-Petch plot of such ultra fine-grained iron, a linear Hall-Petch relationship was confirmed to around 100nm but the plots below 100nm in the grain size deviated toward lower hardness side from the extended Hall-Petch line. In Fe-0.8%C powder, grain size was refined to 10nm but the hardness was also much smaller than the value expected by the extended Hall-Petch line and showed a tendency of levelling-off at Hv₁₂GPa. As a result, the grain size of iron is seemed to be refined to around 10nm by severe deformation and the hardness achieved by grain refining is thought to be about Hv₁₂GPa (expected yield strength: 3.6GPa).

3:30 PM *B5.5/W4.5

ATOMISTIC STUDIES OF PLASTICITY IN NANOPHASE METALS. A. Caro, Centro Atomico Bariloche, Bariloche, ARGENTINA; H. Van Swyghoven, P. Derlet, Paul Scherrer Institute, Villigen, SWITZERLAND; D. Farkas, Dept. of MS&E, Virginia Polytechnic Inst. & State Univ., Blacksburg, VA; M.J. Caturla and T. Diaz de la Rubia, Lawrence Livermore Natl. Lab., Livermore, CA.

We have studied structural, energetic, elastic, and plastic properties of a family of computer generated nanophase samples of Cu and Ni, continuously increasing the average grain size, covering now the range from 20 to 3.5 nm. Properties that can directly be compared to experiments, like density, excess enthalpy, and elastic constants, show a remarkable agreement suggesting that the computer modeled materials are able to capture the essential features of the real

nanophase metals. Investigating the structure of the grain boundaries on the atomic level, we found that grain boundaries in nanophase metals are essentially similar to those found at the micro scale, i.e. similar structural units are found, providing evidence against the view of grain boundaries in nano-crystals as amorphous or liquid-like interfaces. One of the most striking features is the observation of a change in plastic regime as the grain size approaches the ~ 10 nm region. In fact, intragrain dislocation activity ceases to operate below this size and grain boundary sliding, a process based on mechanical and thermally activated single atomic jumps, dominates the contribution to deformation. It in turn implies that deformation becomes easier as the grain size further decreases. This observation is of primary importance in the interpretation of the 'inverse Hall-Petch relation' reported experimentally. The computer simulations provide access to a microscopic view of the deformation processes. In this way we obtain a detailed description of the structure and energetics of grain boundaries and triple junctions, identifying the regions where dislocations are emitted when the grain size is large enough, as well as the mechanism on atomic level of grain boundary sliding. We extract quantitative information about the conditions for dislocation emission and the role of the atomic structure of the grain boundaries.

4:00 PM *B5.6/W4.6

GRAIN-BOUNDARY CONTROLLED DEFORMATION OF NANOCRYSTALLINE MATERIALS BY MOLECULAR-DYNAMICS SIMULATION. Dieter Wolf, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Molecular-dynamics (MD) simulations of model nanocrystalline microstructures demonstrate that the high-temperature plastic-deformation process involves grain-boundary diffusion creep. In order to suppress grain growth and thus to enable steady-state deformation to be observed on a time scale accessible to MD simulations (of typically 10-9s), our input microstructures were tailored to (i) have a uniform grain shape and a uniform grain size of up to about 10 nm and (ii) contain only high-energy grain boundaries which are known to exhibit rather fast, liquid-like self-diffusion. Our simulations reveal steady-state diffusion creep that is homogeneous (i.e., involving no grain sliding), with a strain rate that agrees quantitatively with that given by the Coble-creep formula. The extension of these results to lower temperatures, with emphasis on the crossover in the Hall-Petch effect, is discussed. Work supported by the U.S. Department of Energy, Office of Science, under Contract W-31-109-Eng-38.

4:30 PM B5.7/W4.7

GRAIN MICROSTRUCTURE EVOLUTION IN NANOSCALE MATERIALS - RECENT SIMULATION RESULTS. Moneesh Upmanyu^{1,2,3} and David J. Srolovitz^{1,2}. ¹Princeton Materials Institute, Princeton, Princeton University, NJ. ²Dept. of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ. ³Dept. of MS&E, University of Michigan, Ann Arbor, MI.

The mechanical properties of many materials varies with grain size. For example, the yield strength varies with grain size as in the well-known Hall-Petch relation and creep properties vary with grain size in Coble creep. Such effects are especially important on the nano-scale. In this study, we examine several issues associated with the evolution of the grain structure. In particular, we study several important issues in grain size evolution that only become important when the grain size is very small. These studies were performed using molecular dynamics simulation in two- and three-dimensions. We examine the range of grain sizes over which the classical concepts of grain boundary mobility and curvature driven growth apply and the effects of grain boundary triple junctions and grain rotation on grain boundary migration. These latter effects are commonly neglected in models for grain microstructure evolution. The present results suggest that the boundary velocity is proportional to boundary curvature, at large grain sizes, but increases superlinearly with decreasing grain sizes at very small grain sizes. We have also explicitly extracted grain boundary triple junction mobilities for a large number of grain misorientations. We find that while the triple junctions provide a drag on boundary motion, this drag is negligible at all grain sizes (down to a couple of nanometers). However, we have identified several tricyrystallographies where the triple junction mobility is low enough to significantly slow boundary migration. Finally, simulations of shrinking grains demonstrate that at sufficiently small size, grains rotate as they shrink. Interestingly, this rotation is not completely consistent with expectations based on the misorientation dependence of the boundary energy and does not require the motion of lattice dislocations.

4:45 PM B5.8/W4.8

THE HALL-PETCH RELATION IN NANOCRYSTALLINE METALS. J. Schiøtz, Center for Atomic-Scale Materials Physics and

Department of Physics, Technical University of Denmark, Kongens Lyngby, DENMARK.

It has not yet been settled to what extent nanocrystalline metals obey the Hall-Petch relation, i.e. whether the yield stress and hardness increase with decreasing grain size. There are conflicting experimental data concerning a possible reverse Hall-Petch effect, a softening with decreasing grain size at the smallest grain sizes. The experimental ambiguities are probably caused by the extreme difficulty of manufacturing samples of sufficient quality. We have previously used atomic-scale simulations to demonstrate that nanocrystalline copper and palladium with grain sizes below 13nm exhibit a reverse Hall-Petch effect, caused by sliding in the grain boundaries [Schiøtz et al., *Nature* **391**, 561 (1998); *Phys. Rev. B* **60**, 11971 (1999)]. Based on these simulations, a model is presented that attempts to give a unified description of the yield stress of polycrystalline metals for all grain sizes. The model is based on a competition between two deformation mechanisms: activation of dislocation sources in the grains and grain boundaries, and grain boundary sliding.

SESSION B6: POSTER SESSION
Wednesday Evening, November 29, 2000
8:00 PM
Exhibition Hall D (Hynes)

B6.1

NOVEL TUNGSTEN CARBIDE NANOCRYSTALLINE COMPOSITES BY PULSED LASER DEPOSITION. Ravi K. Venkatesan, A.K. Sharma, A. Kvit, J. Narayan, NSF Center for Advanced Materials and Smart Structures, North Carolina State University, Dept. of Materials Science & Engineering, Raleigh, NC; Q. Wei, J. Sankar, North Carolina A&T State University, Greensboro, NC.

We have developed a novel processing technique to fabricate artifact free Tungsten Carbide nanocomposites. In this method, a modified pulsed laser deposition of WC in conjunction with a few monolayers of NiAl is used to control the grain size of nanocrystalline composites. The grain size of WC was controlled by the relative amounts of tungsten carbide and nickel aluminide deposited in each layer. The role of NiAl is to enhance the nucleation sites of WC and ensure the three-dimensional nucleation of Tungsten Carbide islands. It has a high surface energy and is also insoluble in Tungsten Carbide. Using this approach, we have fabricated nanocomposites with grain sizes as low as 9 nm. The potential is to go even lower by playing with the parameters. The WC grains are fairly uniform and free from defects. The presence of NiAl is below the detection limit of TEM diffraction measurements. Thus, the amount of NiAl was enough to affect nucleation and reduce the grain size of WC to very low values, yet small enough not to show up in the diffraction patterns. The role of NiAl in grain boundary deformation is of particular interest in strengthening the nanocrystalline composites. The correlation between the grain sizes and hardness values obtained by nanoindentation techniques will also be discussed.

B6.2

CERAMIC NANOALLOYS FROM EUTECTIC MELTS: THE SYSTEM Al_2O_3 - Y_2O_3 - ZrO_2 . Jose Calderon-Moreno, Masahiro Yoshimura, Materials and Structures Laboratory, Tokyo Institute of Technology, Tokyo, JAPAN.

For the first time a ternary ceramic eutectic composite was prepared, in the system alumina-yttria-zirconia. The composite was self-assembled in one quick step by rapid quenching of the multioxide alumina-YAG-zirconia eutectic melt in a solar furnace. The eutectic composite has a nanolayered structure of eutectic lamella, with lamella sizes from 20 nm, two orders of magnitude thinner than in directionally solidified eutectics. New eutectic composites with very fine structure – i.e. fine multilayered or nanowired composites – will have excellent thermomechanical stability at high temperature compared to any sintered ceramics, and excellent prospectives for applications as in-situ coating and high temperature component. Design of rapidly supercooled ceramic eutectics opens a new multidisciplinary challenge of obtaining kinetically controlled nanostructures tailored to specific applications. In this work we address the fabrication of new ultra-high temperature ceramics by rapid quenching of eutectic oxide melts and show the feasibility of obtaining very fine nanolayered self-assembled eutectics.

B6.3

GRAINS WITH THE RUTILE STRUCTURE. A TIGHT-BINDING CALCULATION OF THE ELECTRONIC STRUCTURE. A.M. Mazzone, C.N.R.-Istituto LAMEL, Bologna, ITALY.

It has been recognized for some time that the unusual dielectric and

optical properties of oxides in the rutile family can be used advantageously in a variety of electronic devices and gas sensors. The importance of these materials has led to many structural studies on the crystalline state of the materials of the rutile group. In spite of the importance of these contributions, they seem insufficient for the current applications of metallic oxides. In modern technologies, in fact, thin films are used which have a granular structure with grains of size 20-50 nm. Significant alterations of the electronic properties with respect to the ones of the crystalline material are obviously expected. Many current studies gain insight on the properties of micro-crystalline films from the properties of its basic building block, that is a fiducial grain which is described at quantum mechanical level. This approach has been adopted also in this contribution. In this work the electronic properties of crystalline grains of TiO_2 and SnO_2 are studied with a quantum mechanical method of the tight binding type. The grains have a realistic size and their structure is either spherical or polygonal, with edges and corners. The focus of the calculations is on their bonding properties and we evaluate binding energies, surface tensions and migration barriers. These quantities should lead to useful informations on the stability of the film under operative conditions.

B6.4

A NEW APPROACH TO THE ESTIMATION OF STRAINS IN COMPOSITIONAL SEMICONDUCTOR SUPERLATTICES.

Vadim Mirovitskii, Institute of Applied Physics, Dept of Laser Characterization of Materials, Kishinev, MOLDOVA.

The universal method is developed to the calculation of strain fields in the $(AC)_m(BC)_n$ superlattice (SL) with ultra-short period. It is based on the idea to consider such SL as a complex single crystal formally "derived" from a simpler "initial" one. Namely, we represent an SL composed of the $III-V$ (or $II-VI$) zinc-blende semiconductors as a result of alternative A/B atoms ordering in the $A_xB_{1-x}C$ solid solution with $x = m/(mn)$. This artificial ordering is described by two-components order parameter and accompanied by both a homogeneous deformation of "initial" crystal structure and mutual shifts of its splitted sublattices. By formal analogy with phase transitions both these types of distortions are calculated in the spirit of Landau theory as functions of order parameter. The approximate analytical expressions for phenomenological coefficients in these dependences are found out by the methods of statistical mechanics applied to the quite realistic microscopic model of the solution. Thereby it is demonstrated that previously bulky problem is reduced to simple one. It is shown, that all we need to know for a numerical estimation of strains are only the SL geometry and some physical characteristics (such as elastic moduli and total energy) of the "initial crystal" (disordered solid solution).

B6.5

YTTRIUM ALUMINUM GARNET NANOPOWDERS. J.C.Marchal, T. Hinklin, R.M. Laine, Depts. of Materials Science and Engineering and Chemistry, University of Michigan, Ann Arbor, MI.

Nanosized YAG powders can be produced by flame spray pyrolysis (FSP) from very simple yttria and alumina precursors, that can be doped with various metals. This scaleable synthesis route produces single crystal nanopowders at > 100 g/h with surface areas of 50-100 m^2/g . Typical powders consist of unaggregated partially amorphous particles, with sizes 20-80 nm diameter. As-shot powders are not crystalline YAG, but are converted to nanocrystalline unaggregated YAG powder after low temperature heat treatment. These powders can be used to form fine grained YAG monoliths, fibers, and novel photonic materials. Details concerning flame spray pyrolysis, and the influence of the precursors will be discussed.

B6.6

SINGLE CRYSTAL MULTIMETALLIC OXIDE NANOPARTICLES.

T.R. Hinklin, J.C. Marchal and R.M. Laine, Dept. of Materials Science & Engineering and Dept. of Chemistry, University of Michigan, Ann Arbor, MI.

We recently described methods of producing nanosized oxide powders by flame spray pyrolysis of novel alkoxide aluminate and silicate complexes as well as other soluble metal complexes. This scaleable synthesis route provides reproducible, high purity nanosized powders. We have previously studied single crystal TiO_2 , CeO_2 , crystalline mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), β'' alumina and amorphous strontium aluminosilicate ($\text{SrO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) powders with controllable average particle sizes of 2-200 nm and surface areas = 40-120 m^2/g at production rates ≈ 250 g/h. The powders are characterized using a variety of techniques. This poster will focus on the utility of the FSP method to provide control of multimetallic concentrations from line compounds down to ppm level, the formation of selected matrices and the potential to optimize sintering, catalytic, magnetic, optical and other desired properties.

B6.7

PROCESSING AND CHARACTERIZATION OF $\text{Cu-Al}_2\text{O}_3$ NANOSCALE COMPOSITES SYNTHESIZED BY IN-SITU REDUCTION.

M.S. Motta, E.A. Brocchi, P.K. Jena and I.G. Solurzano, Catholic Univ of Rio de Janeiro, Dept of Materials Science and Metallurgy, BRAZIL.

The copper-alumina system is reported to have excellent combination of properties that are essentially dependent on their microstructure, which, in turn, will vary according to the preparation method. In this work, the synthesis of the $\text{Cu-Al}_2\text{O}_3$ (5 wt%) nanoscale composites was carried out by in-situ reduction of a powder mixture consisting of CuO and Al_2O_3 , previously obtained through a chemical route. The procedure imparts to the product special features in terms of microstructure. FEG SEM observations have suggested the formation of a composite with nanoscale alumina particles dispersed in the copper matrix. Although most of the microstructure has a homogeneous distribution of Cu, Al and O, the x-ray STEM elemental mapping revealed a tendency for clustering in specific regions. TEM bright field and centered dark field imaging have shown well-developed copper crystals surrounded by Al_2O_3 particles, which are in the order of 10 nm in size. Copper crystals ranging from 50 to 250 nm are dispersed and exhibit twinning despite their small size. Current investigation is being developed to assess the evolution of the microstructure resulting from cold rolling followed by annealing.

B6.8

STRUCTURE AND MECHANICAL PROPERTIES OF FLEXIBLE INORGANIC/ORGANIC HYBRID SHEETS. Shingo Katayama and Noriko Yamada, Nippon Steel Corporation, Advanced Technology Research Labs, Futtsu, JAPAN.

Inorganic/organic hybrids prepared from polydimethylsiloxane (PDMS) and tetraethoxysilane (TEOS) have flexible and rubbery properties. However, they have only an inorganic component of the siloxane network derived from TEOS. We successfully fabricated flexible sheets of PDMS-based hybrids from PDMS and chemically modified metal alkoxides as a precursor of the inorganic components. The hybrid sheets were flexible, homogeneous and transparent without inorganic particles precipitated by hydrolysis of metal alkoxides. The inorganic component derived from metal alkoxides was homogeneously present as fine oxide-like clusters with several nm or below by results of EXAFS, SAXS, and HRTEM. The inorganic component was also bonded to PDMS chains via M-O-Si bonds such as a cross-linking agent of PDMS chains by results of FT-IR and NMR. The hybrid sheets showed high elongation of 100% and high strength of 3.0MPa. Since the storage modulus of 10^7 - 10^8 Pa in rubbery region was approximately constant at temperatures of 0-300°C, the hybrid sheets may have high temperature stability in flexibility. This work has been supported by NEDO, as part of the Synergy Ceramics Project promoted by AIST, MITI, Japan.

B6.9

FABRICATION AND STRUCTURAL CHARACTERISATION OF NANOSTRUCTURED NETWORK OF GAMMA IRON OXIDE.

S.A.M. Tofail, I.Z. Rahman, M.A. Rahman, Materials and Surface Science Institute (MSSI), University of Limerick, IRELAND.

Nanofabrication technology has recently been opening up new opportunities for innovative magnetic materials and devices. It is anticipated that novel nanostructured magnetic arrays produced by such techniques can be useful as recording media for future high-density recording and can increase the recording density beyond 100 Gbits/in². Negative anti dot structures e.g. nanonetworks are believed to overcome the recording limit which is incurred by superparamagnetism in such media. We report the fabrication of nanostructured network of an important recording media, gamma iron oxide ($\gamma\text{-Fe}_2\text{O}_3$) by rf-magnetron sputtering and reactive rf-magnetron sputtering of an iron target with the help of a template, nanochannel alumina (NCA). It has been found from X-ray Diffraction (XRD) phase analyses that the nano size pores of the NCA template enhanced the in-situ formation of the gamma iron oxide phase even without any further air annealing. The nanonetworks deposited at lower substrate temperatures and oxygen pressures are found to be under tensile strains due to the porous nature of the film. The gamma iron oxide nanonetworks deposited at higher substrate temperatures and oxygen pressures, in contrast, are under compressive strain indicating an in chamber annealing of the films. The surface topography of such nanonetworks by Atomic Force Microscopy (AFM) shows that the nanonetworks are very rough compared to the continuous films deposited on Si and glass substrates and this roughness originates from the fabrication history of NCA.

B6.10

MODELING BONDING IN LEAD MAGNESIUM NIOBATE USING DENSITY FUNCTIONAL THEORY. George Kavarnos, EG&G, Inc.,

Groton, CT; Roger Richards, Naval Undersea Warfare Center Division, Newport, Newport, RI.

Lead magnesium niobate (PMN) ceramic is a relaxor material used in underwater high-power sonar drivers. This material has drawn considerable interest because of its high strains and energy densities when stressed by large electric fields. Nonetheless, despite their outstanding electromechanical properties, PMN-based transducers have been predisposed to premature failure. An important issue here is to understand the origins of these failure mechanisms. We have applied first-principles Density Functional Theory (DFT) to understand the nature of bonding in PMN solid solutions with lead titanate (PT). This high-level quantum mechanical approach has been used to analyze the role of bonding, ionicity, and electronic distribution in PMN-PT model structures. In addition, we used this approach to calculate the energetics of crystal structures containing various atomic defects. It will be shown that local order and bonding are sensitive to the presence of substitutionals such as strontium and barium.

B6.11 RELATIONSHIP BETWEEN FILTRATION RATE AND CRYSTAL SIZE OF 3-D SELF-ASSEMBLED COLLOIDAL CRYSTALS.

Siu-Wai Chan, Feng Zhang, Columbia Univ, Henry Krumb School of Mines, Materials Science and Engineering Program, New York, NY.

Filtration method is important in processing 3-D self-assembled colloidal crystals. It is found that filtration rate is one of the significant factors of controlling crystal qualities. The grain size increases with the decrease of filtration rate up to a certain grain size. The final grain size is related to the particle kinetics in the media. Certain universal features are observed in nano-particle systems, such as SiO₂ and polystyrene, with different sphere sizes from nanometer to sub-micron size.

B6.12 ORGANIC/INORGANIC NANOCOMPOSITES FROM SILSESQUIOXANE. J. Choi, R. Tamaki and R.M. Laine, Dept of MS&E Chemistry and the Macromolecular Science and Engr. Center, University of Michigan, Ann Arbor, MI.

Development of hybrid materials from cubic silsesquioxanes [(RSiO)_{1.5}]₈, cubes] is a subject of intense research. Using these silsesquioxanes, hybridization of organic and inorganic components becomes possible at a molecular level providing the potential to obtain novel properties. Incorporation of cubic silsesquioxanes in polymer backbones or as pendant groups provides improved thermal stability. Recently, functionalized cubic silsesquioxanes were examined as potential building blocks for hybrid composites wherein the architecture and length of the organic tether between cubes can be completely defined. Our goal is to develop structure-property-processing relationships in nanocomposites prepared using octa-functionalized silsesquioxanes [(RSiO)_{1.5}]₈, cubes] to establish a complete understanding of the behavior of "interphase" materials. Interphase materials normally consist of the species at the interfaces between adjacent phases. In our approach, organic functional groups (R = epoxy, methacryloyl, etc) are appended to inorganic cubes and polymerized to give organic/inorganic nanocomposites wherein both components are present at the 1~2 nm scale and discontinuous. Their properties will be governed not only by the nature of but also by the scale of each phase. Thus, all organic and inorganic species are at discontinuous interfaces at length scales typical of interphase materials and the composites should be wholly interphase.

B6.13 SYNTHESIS AND CHARACTERIZATION OF PERIODIC NANOSTRUCTURED ORGANOSILICATES BY USING BLOCK COPOLYMER TEMPLATES. Eun-Bum Cho, Daehwan Kim, Sang-Hyon Chu, Kookheon Char, Seoul National University, School of Chemical Engineering, Seoul, KOREA.

Nanostructured organic-inorganic hybrids and nanoporous materials can be achieved by sol-gel chemistry using amphiphilic molecules as structure-directing agents. With amphiphilic block copolymers in a selective solvent, hydrolysis and polymerization of inorganic species occur in one of the microphase separated amphiphilic molecules by a preferable interaction at the block copolymer-solution interface. The ordering length scales and periodicities of the porous silica materials are established by the dimensions and arrangements of the micellar core aggregates. Tetraethyl orthosilicate (TEOS) has been widely used for various types of ordered mesoporous silica materials. Recently, in order to realize superior physical properties, the synthesis of porous materials incorporating a variety of organic or organometallic species becomes a new trend in this area. Nanoporous organosilicates showing well-defined hexagonal or cubic phases can be applied to a wider range than materials obtained only by TEOS. We have tried to prepare more mechanically stable porous materials by using organosilicates

such as methyltriethoxysilane (MTES), dimethoxy-dimethylsilane (DMDMS), and 1,2-bis(trimethoxysilyl)ethane (BTMSE). Methyl moieties substituted in MTES, DMDMS, and BTMSE demonstrate morphologies different from the morphologies typically obtained with TEOS after reactions with the block copolymers, and also show improved mechanical property of the stable thin films. In present study, we have used commercial or specially designed block copolymers as nanotemplates and use TEOS, MTES, DMDMS, and BTMSE as inorganic precursors. We have investigated the reaction mechanism between the block copolymers and the organosilicates, and obtained nanocomposite films as well as nanoporous organosilicate powders by varying experimental conditions. We have also studied the pore shape and packing structure of the nanoporous materials using small angle X-ray scattering (SAXS), BET isotherm, transmission electron microscopy (TEM), and nuclear magnetic resonance (NMR).

B6.14 PROPERTIES OF COLLOID-LIQUID CRYSTAL COMPOSITES. Jason Crain, R.J. Lavery, S.P. Meeker, W.C.K. Poon, Department of Physics, University of Edinburgh, UNITED KINGDOM; H. Nakamura and Y. Taira, IBM Research, Tokyo, JAPAN.

This paper explores the mechanical, optical and structural properties of dense dispersions of spherical colloidal particles in liquid crystalline solvents. In the isotropic phase, these systems behave for the most part as normal colloidal suspensions showing approximately single-particle Brownian diffusion as observed using light scattering techniques. Upon cooling toward the isotropic-nematic phase transition the colloidal particles are expelled from the emerging nematic droplets and become increasingly confined to interfacial regions between nematic domains. This partial phase separation occurs because the particles introduce unfavorable high-energy topological defects in the nematic phase. The resulting microstructure, at temperatures well into the nematic stability range, is one characterized by a percolating network of particle-rich interfaces between nematic regions. The viscoelastic moduli of this composite are unusually high even for relatively low particle concentrations and can reach values of 10⁵ Pa at 20% volume fraction. Such composites are mechanically rigid and self-supporting. A rigorous theoretical description of the mechanical properties of this class of materials has not yet been developed but a promising model will be outlined. Optically, these composites are more opaque (turbid) than is the pure nematic liquid crystal host but the underlying optical anisotropy of the liquid crystal is preserved opening the possibility of exploiting these materials for optical applications. The microstructure and physical properties have been characterized by confocal microscopy, small amplitude viscoelastic measurements and electro-optical response. The nature of the microstructure and the physical properties of the composites depend sensitively on particle concentration and phase transition kinetics. This dependence will be discussed as will potential applications in liquid crystal display devices.

B6.15 MILLIMETER WAVE GYROTRON BEAM PROCESSING OF NANOCRYSTALLINE METALS USING THE POLYOL PROCESS. L.K. Kurihara, D. Lewis III, Mat Sci and Tech Div, R.W. Bruce, A.W. Fliflet, Plasma Physics Division, Naval Research Laboratory, Washington, DC.

A new continuous-wave (CW) gyrotron-based system has been set up at the Naval Research Laboratory (NRL) to investigate rapid, selective millimeter-wave heating of materials. This facility is being applied to important areas of material processing including coating of materials, joining, and treatment of ceramics and polymers. Nanocrystalline metallic powders and films have been prepared by reducing metal salts in a polyol using millimeter-waves as the heating source. A few of the metals synthesized in this study include: Co, Ni, Cu, Ru, Pt, Au, and Cu-Ni alloy. Additionally, a Cu film was deposited on a AlN substrate. Conventionally, the metal salt-glycol solution is brought to reflux using a heating mantle as the heat source. In this study, a millimeter-wave beam provided by a Gycom, Ltd., 83 GHz CW gyrotron [operated at less than 200W output power] was used. The experiment was set up in the processing chamber, the beam was directed into the center of the solution and it was brought to reflux. The rate of reflux was easily controlled by varying the power input. The differences in nucleation and growth of the metal particles by conventional and millimeter-wave heating methods will be discussed. Sponsored by the Naval Research Laboratory

SESSION B7: CERAMIC MATERIALS
Chair: Merrilea J. Mayo
Thursday Morning, November 30, 2000
Hampton (Sheraton)

8:30 AM *B7.1 BREAKING THE NANOGRAIN BARRIER IN SINTERED

CERAMICS. Bernard H. Kear, Rutgers University, Dept. of Ceramic and Materials Engineering, Piscataway, NJ.

Bulk nanocrystalline Al_2O_3 (corundum) and TiO_2 (rutile) samples with a relative density >98% and a grain size <50 nm have been produced by high pressure (2- 8 GPa)/low temperature ($\sim 0.35 T_m$) sintering. Metastable nanophase powders, produced by a chemical vapor condensation method, are used as starting materials. During sintering, a multiplicity of nucleation events in the parent phase at high pressure creates a nanoscale grain size, whereas grain growth is limited by the low temperature. Thus, we have demonstrated the feasibility of making dense sintered ceramics with grain size comparable to or even smaller than that of the starting powder particle size. High pressure sintering has also been used to produce a new class of ceramic nanocomposites, starting with plasma-melted and quenched metastable powders. For example, rapidly solidified $\text{Al}_2\text{O}_3/13\text{TiO}_2$ powder has a metastable spinel structure, which decomposes under pressure into an $\text{Al}_2\text{O}_3/\text{TiO}_2$ nanocomposite. This method appears to be applicable to many ceramic systems. It is currently being investigated as a means to produce ZrO_2 -base ceramics for applications requiring high strength and toughness.

9:00 AM **B7.2**

PROCESSING AND MECHANICAL PROPERTIES OF NANO-SCALE COMPOSITES FROM POLYMER PRECURSOR, HIGH-PRESSURE AND SPARK PLASMA SINTERING ROUTES. Amiya K. Mukherjee, Julin Wan, Matt J. Gasch, Joshua D. Kuntz, University of California, Dept. of Chemical Engineering & Materials Science, Davis, CA.

The engineering of superior nanostructured materials is now possible with the advent of several novel processing routes. In comparison to conventional ceramic sintering techniques, polymer precursors offer new methods for making silicon nitride/silicon carbide ceramic composites. To understand the structural evolution from polymer to nano-ceramic, two processing routes have been developed to produce silicon nitride/silicon carbide nanocomposites with varied microstructural features. Method 1 involves pyrolysis and in-situ consolidation of compacted polymer into Si-C-N amorphous materials. Crystallization of such specimens yields silicon nitride/silicon carbide composites with nanometric grains. Method 2 involves the spark plasma sintering of pyrolysis-derived amorphous powder with oxide additives. Results, so far, indicate remarkable flexibility in microstructural design and possibly improved high temperature properties of nanocomposites produced from polymer precursors. Nanocrystalline alumina composites have been formed using high-energy ball milling powder processing, and either high pressure sintering or spark plasma sintering as consolidation routes. The composite systems under investigation are either nano-ceramic particle or metallic phase toughened composites. The fully dense nano-alumina matrix increases strength and hardness while the second phase is designed to improve the composites' toughness. The ceramic particle reinforcements of interest are silicon carbide, zirconia, and diamond. The particle morphology is manipulated to introduce various toughening mechanisms including: residual stress, transformation, and crack bridging. The metallic phase added is a niobium alloy, composing between 5 and 25 vol. % of the composite. Research was supported by U.S. Office of Naval Research Grant #N00014-00-1-0186. TEM work was supported by the Director, Office of Science, Office of Basic Energy Sciences, at the National Center for Electron Microscopy, Lawrence Berkeley Laboratory, under U.S. Department of Energy Contract #DE-AC03-76SF00098.

9:15 AM **B7.3**

MULTIMODAL POWDERS AND COATINGS: A NEW APPROACH TO THERMAL SPRAYING. Ganesh Skandan, Ruvee Yao, Nanopowder Enterprises Inc., Piscataway, NJ; Bernard H. Kear, Rutgers-The State University of New Jersey, Piscataway, NJ; Yunfei Qiao, Lucy Liu and Traugott Fischer, Dept. of Chemical, Biochemical and Materials Engineering, Stevens Institute of Technology, Hoboken, NJ.

We are developing a new class of High Velocity Oxy-Fuel (HVOF) thermal spray feedstock powders, which consist of aggregates of hard ceramic particles that are either mixed or coated with a more readily fusible nanophase binder. Thus, during thermal spraying, the nanostructured material undergoes rapid melting while the aggregated material is heated but not necessarily melted. A dense coating is formed when the molten nanomaterial fills the available pore spaces between the heated and softened aggregates, providing a strong and tough matrix for the spray deposited material. Such multimodal coatings combine moderate hardness with exceptional abrasion wear resistance. Multimodal coatings exhibit properties that are different from those of their unimodal counterparts. In some cases, even the wear mechanism for the multimodal coatings is entirely different. Characteristics of WC/Co multimodal feed powders, along with the structure and properties of the resulting coatings, will be discussed.

9:30 AM **B7.4**

PROCESSING AND PROPERTIES OF SINGLE- AND MULTI-PHASE BULK NANOCERAMICS. James Colaizzi, NanoPac Technologies, Inc., Allentown, NJ; William E. Mayo and Bernard H. Kear, Rutgers Univ, Center for Nanomaterials Research, Piscataway, NJ.

The fabrication of starting material for and production of bulk ceramics with nanoscale grain size is described. Flat flame spraying and plasma spraying are used to homogenize starting materials. Quenching of the spray stream on a chilled metal plate rapidly cools the homogenized liquids to create single phase metastable materials from multi-components systems. These single phase materials of various oxide systems including alumina/titania and zirconia/yttria/alumina are used to create bulk nanoscale ceramic objects using the Transformation Assisted Consolidation (TAC) method. Compacts of these powders are sintered under high pressure, up to 8 GPa. The high pressure creates high green density before high temperature is applied, which allows rapid densification to commence immediately. Sintering is performed by electrical resistance heating of the graphite crucible surrounding the sample while still under high pressure. High pressure and relatively low temperature (30 to 60 percent of the melting temperature) maximize nucleation of the stable phase(s) and minimize grain growth. Both single and multi phase structures can be created. In multi-phase structures, the various nucleating phases inhibit growth of the other phases. Systems that normally exhibit two phases in equilibrium can reveal high pressure phases as well. This results in several phases being created in the structure, all with grains nanoscale in size and distributed uniformly. The usual property enhancements associated with smaller grain size and composite structures are augmented at the nanoscale. Enhanced mechanical behavior of these structures is discussed, including improved wear resistance, friction and hardness.

10:15 AM ***B7.5**

NANOCRYSTALLINE TETRAGONAL ZIRCONIA WITH LOW YTTRIA CONTENT. Alfonso Bravo-Leon, Dept. de Física de la Materia Condensada, Universidad de Sevilla, SPAIN; Merrilea J. Mayo, Dept. of MS&E, The Pennsylvania State University, University Park, PA.

Nanocrystalline tetragonal zirconia samples are shown to exhibit excellent mechanical properties at room temperature, provided the conventional levels of 3 mol% yttria additive are lowered to 1.0 or 1.5 mol%. For these compositions, the maximum value of the toughness occurs at a grain size just below the critical grain size for spontaneous transformation to the monoclinic phase which is 90 nm for 1.0 mol% and 110 nm for 1.5 mol% yttria content. Thermodynamic analysis predicts a linear relationship between yttria content and the inverse of the critical grain size, which is shown to apply into the nanocrystalline regime. Literature analysis suggests a power law relationship between grain size and toughness increment which is also obeyed by the present data.

10:45 AM **B7.6**

VARIABLE-CHARGE MOLECULAR-DYNAMICS SIMULATIONS OF SINTERING OF TiO_2 NANOPARTICLES. Shuji Ogata, Yamaguchi Univ, Dept of Applied Sciences, Ube, JAPAN; Hiroshi Iyetomi, Niigata Univ, Dept of Physics, Niigata, JAPAN; Kenji Tsuruta, Okayama Univ, Dept of Electrical and Electronic Eng, Okayama, JAPAN; Fuyuki Shimojo, Hiroshima Univ, Faculty of Integrated Arts and Sciences, Higashi-Hiroshima, JAPAN; Chun K. loong, Argonne National Lab, IPNS, Argonne, IL; Aiichiro Nakano, Priya Vashishta, Rajiv K. Kalia, Louisiana State Univ, Dept of Physics and Astronomy, Baton Rouge, LA.

Grain-boundary structure and space-charge distribution in sintering TiO_2 -nanoparticles are investigated for both rutile and anatase phases using a variable-charge interaction potential in which atomic charges vary dynamically depending on their environment. For the nanoparticles with size $\sim 6\text{nm}$, we find the dynamic charge transfer: (i) enhances atomic diffusion at surfaces of the spherical nanoparticles at high temperatures; and (ii) creates additional repulsive force between the two nanospheres through formation of a double-charge surface layer in each nanosphere. The surface diffusion due to the charge transfer clearly distinguishes the two nanospheres with different underlying crystalline structures; the surface diffusion constant of the anatase sphere is almost three times as great as that of the rutile sphere. Variable-charge molecular dynamics simulations of sintering of two TiO_2 -nanospheres demonstrate that the enhanced surface diffusion in the anatase nanosphere at high temperatures significantly promotes neck formation between the two spheres. We then perform larger-scale simulations of nanoparticle-assembled systems to investigate evolution of microscopic structures during sintering. We compare present results for the nanophase TiO_2 with the corresponding experimental data on phonon density-of-states obtained by the neutron scattering techniques.

11:00 AM B7.7

STRUCTURAL DISORDER IN THE ANION LATTICE OF NANOCRYSTALLINE ZIRCONIA AND HAFNIA PARTICLES. Dieter Vollaht, D. Vinga Szabo, Michael Hagelstein, Forschungszentrum Karlsruhe, Karlsruhe, GERMANY; Manfred Forker, University Bonn, Bonn, GERMANY.

Nanoparticles often crystallize in different structures than bulk materials. Typical examples are zirconia, ZrO_2 and hafnia, HfO_2 , crystallizing monoclinic as bulk material at room temperature. Nanoparticles with sizes around 5 nm are - depending on the production route - found in the cubic or tetragonal phase. To shed some light into this difference, an analysis of crystal structure, distances, and symmetry around the cations was performed on alumina coated nanoparticles prepared by the microwave plasma process. Within the range of accuracy, electron-diffraction and -microscopy revealed a possibly cubic cation lattice for both materials. The evaluation of the magnitude of the Fourier transformed EXAFS data lead to a structure with a very broad distribution of oxygen ions in the first shell of neighbors. For the cubic structure one narrow and for the tetragonal one, a split radial distribution peak is expected. This result indicates a random distribution of the oxygen ions in the vicinity of the lattice sites. Comparing the imaginary part with model calculations, a distinct similarity with the cubic structure is obvious. In the second shell consisting of metal ions, one radial distribution peak is expected and found experimentally for both structures. In perfect crystallized cubic zirconia, model calculations predict a shoulder stemming from multiple scattering on the peak for the next cation neighbor. This shoulder was neither observed for coarse-grained yttria stabilized cubic zirconia nor for nanocrystalline material. The results from perturbed angular correlation (PAC) indicate clearly a random distribution of the first neighbors of the metal ions. Above 500°C a reversible transformation to perfect tetragonal symmetry is observed. Therefore, one has to conclude that the cubic structure with disordered anion lattice is more stable than the tetragonal one at room temperature. These structural relationships found in zirconia and hafnia are unknown in any coarse grained material.

11:15 AM B7.8

MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF CERAMIC/CERAMIC NANOCOMPOSITE THIN FILMS BY XANES, EXAFS, SAXS, HRTEM, AND NANOINDENTATION. W.J. Meng, D.E. Alexander¹, D.M. Cao, B. Feng, R.C. Pittsworth², Mechanical Engineering Department, Louisiana State University. ¹Materials Science Division, Argonne National Laboratory; ²Center for Advanced Microstructures and Devices, Louisiana State University, LA.

Using Ti-containing hydrocarbon (Ti-C:H) thin films deposited by plasma assisted vapor phase techniques as an example, we demonstrate a general experimental methodology for in-depth characterization of the microstructure and mechanical properties of ceramic/ceramic thin film nanocomposites. Combining high-resolution transmission electron microscopy (HRTEM) and x-ray absorption near edge structure (XANES) spectroscopy, we have for the first time measured the dissolution limit for Ti atoms in an amorphous hydrocarbon (a-C:H) matrix. Beyond the dissolution limit, precipitation of B1-TiC occurs, and Ti-C:H thin films are in-fact TiC/a-C:H nanocomposites. The local atomic bonding environment of Ti atoms as a function of Ti composition has been determined by extended x-ray absorption fine structure (EXAFS) spectroscopy. The size distribution of TiC nanoclusters as a function of Ti composition has been determined by small angle x-ray scattering (SAXS) measurements. Mechanical properties of TiC/a-C:H thin films with widely varying TiC volume fractions have been measured by nanoindentation. Despite the nanocrystalline microstructure, it is shown that measured elastic stiffness and hardness of TiC/a-C:H nanocomposites conform well to macromechanical bounds for two-phase materials. Our results on TiC/a-C:H nanocomposite thin films serve as a basis with which data on other nanocomposite thin films are compared.

11:30 AM B7.9

NANOMECHANICAL PROPERTIES OF NACRE - A BIOMIMETIC MODEL FOR LAYERED COMPOSITES: EXPERIMENTS AND MODELING. Jeffrey M. Sopp, William N. Mercer, Hanson Fong, and Mehmet Sarikaya, Materials Science and Engineering, University of Washington, Seattle, WA; Kalpana S. Katti, and Dinesh R. Katti, Civil Engineering, North Dakota State University, Fargo, ND.

Organisms produce biocomposites of inorganics and macromolecules, intricately architected hierarchically, at all dimensional scales exhibiting physical properties superior to synthetic materials with similar phase compositions. These include bone, dental tissues, mollusk shells, spicules, spines and bacterial particles. These biocomposites are excellent models for nanostructured engineering

materials. A truly biomimetic design requires a knowledge of detailed internal and interface structures and local properties of individual biocomponents. Using a nanoindentation system attached to an atomic force microscope, we determined mechanical properties of both the aragonite platelets (250 nm-thick) and the thin-film (20 nm) organic matrix in nacre (mother-of-pearl) of mollusk shells. Furthermore, we simulated the mechanical response using 3D finite element analysis that models detailed nacre structures. These results revealed hitherto unknown properties, potentially the basis for biomimetic design of layered hybrid composites. Here, a sharp nanoindenter (diamond Berkovich tip) was used to determine local hardness and elastic modulus of the biomineral aragonite crystallites and these are compared with those of large single crystal geological mineral over a range of indent sizes. Similarly, the properties of the organic matrix are determined using specially-engineered blunt, semispherical (metallic) tips at very small stresses on cleaved surfaces, and compared to those of well-known synthetic polymeric thin films. The capabilities of using small indents and low stresses (nanoNewton) result in isolated properties (e.g., elastic modulus) of individual components. Based on these experimental results, we constructed a nanoscale three-dimensional model nacre using non-linear finite element method (FEM) to simulate strain-rate effects and large deformations in organic phase. We incorporated interface mechanics at nanoscale as related to macro-scale response. More specifically, the scale effects, i.e. influence of dimensional scaling-up on mechanical response, were evaluated in the context of future nano-layered materials design.

11:45 AM B7.10

MECHANICAL PROPERTIES OF NANOPHASE HYDROXYAPATITE VIA INSTRUMENTED INDENTATION. K.J. Van Vliet, S. Suresh, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA; E.S. Ahn, J.Y. Ying, Massachusetts Institute of Technology, Dept of Chemical Engineering, Cambridge, MA.

Hydroxyapatite (HAP) is used in bioactive coatings for metallic orthopedic implants and other non-load bearing applications. However, the non-stoichiometry, inhomogeneities, and impurities introduced during conventional processing of this ceramic limit its mechanical strength. Alternatively, nanostructure processing of HAP has achieved microstructural and chemical homogeneity resulting in a nanophase HAP monolith (125 nm grain size). Because of this ultrafine microstructure, flaw sizes are reduced and mechanical properties are enhanced. Furthermore, additional toughening of HAP can be achieved through nanocomposite processing which incorporates highly dispersed tetragonal zirconia nanoparticles. Here, we present the dependence of HAP mechanical properties (hardness, elastic modulus, etc.) on the weight fraction of both yttria-stabilized and unstabilized tetragonal zirconia, as assessed via sharp and spherical microindentation. These results are compared with theoretical predictions and experimental results from more traditional mechanical testing methods for brittle materials to assess the extent to which the incorporation of highly dispersed zirconia increases the toughness of our HAP-based systems. In addition, as the microindentation technique samples very small volumes, we will also evaluate the extent to which our processing has achieved microstructural uniformity.

SESSION B8: CLUSTERS AND OTHER NANOSTRUCTURES

Chair: Julia R. Weertman
Thursday Afternoon, November 30, 2000
Hampton (Sheraton)

1:30 PM *B8.1

MECHANICAL PROPERTIES OF NANOSTRUCTURED ALUMINA-TITANIA COATINGS. L. Shaw¹, D. Goberman¹, Y.H. Sohn¹, E.H. Jordan² and M. Gell¹. ¹Department of Metallurgy and Materials Engineering; ²Department of Mechanical Engineering, University of Connecticut, Storrs, CT.

Nanostructured coatings have potentials to provide significant improvements over coarse-grained counterparts in load bearing applications owing to enhanced hardness and toughness. In this paper, Al_2O_3 - 13 wt.% TiO_2 coatings formed via thermal spray approach using reconstituted nanosized Al_2O_3 and TiO_2 powder feeds are described. Microhardness, indentation crack growth resistance, adhesion strength, spallation resistance during bend- and cup-tests, abrasive wear resistance, sliding wear resistance and single scratch resistance of the coatings obtained from the nano-powder feeds have been characterized and compared to those obtained from commercial coating counterparts. The properties of the coatings obtained from nano-powder feeds are discussed and related to their microstructures and thermal spray conditions.

2:00 PM B8.2

ATOMIC SCALE MODELING OF SUPPORTED AND ASSEMBLED NANOPARTICLES. E. Zhurkin¹, M. Hou², H. Van Swygenhoven³, B. Pauwels⁴, M. Yandouzi⁴, D. Schryvers⁴, G. Van Tendeloo⁴, P. Lievens⁵, G. Verschoren⁵, J. Kuriplach⁶, S. Van Peteghem⁷, D. Segers⁷ and C. Dauwe⁷. ¹St. Petersburg State Technical University, Department of Experimental Nuclear Physics, St. Petersburg, RUSSIA; ²Physics of Irradiated Solids, Free University of Brussels, Brussels, BELGIUM; ³Paul Scherrer Institute, Villigen, SWITZERLAND; ⁴EMAT, University of Antwerp, RUCA, Antwerp, BELGIUM; ⁵Laboratory for Solid State Physics and Magnetism, Catholic University of Leuven, Leuven, BELGIUM; ⁶Department of Low Temperature Physics, Charles University, Prague, CZECH REPUBLIC; ⁷NUMAT, Subatomic and Radiation Physics Department, Ghent University, Ghent, BELGIUM.

The properties of elemental and bi-metallic free, supported and assembled nanoclusters are modeled at the atomic scale and the models are discussed on the basis of experimental observations. This way, the memory of some free cluster properties in nanostructured materials may be evaluated. The combination of molecular statics with High Resolution Transmission Electron Microscopy (HRTEM) allows to predict fine detail of the lattice relaxation of a truncated octahedral gold cluster deposited on MgO. Metropolis Monte Carlo (MC) predicts that a lattice mismatch may contribute to disordering in deposited Cu_3Au nanoclusters. In both Cu-Au and Ni-Al free clusters, onset of equilibrium stoichiometry may result in segregation of Au or Al at the cluster surface. An ordered stoichiometric core is surrounded by a disordered mantle where the excess specie resides. Different modeling methods predict different nanometer scale textures. Therefore, cluster assembled Ni_3Al alloys formed by condensation and pressing are modeled in two different ways. Both make use of a combination of Molecular Dynamics and MC. Whatever the model nanostructure, the segregation properties of free clusters remain in the nanostructured material. The segregation is found to inhibit the formation of a metastable martensitic phase as observed in bulk Ni-Al alloys. The prediction is consistent with HRTEM observation, which indicates that the martensitic phase only occurs in grains larger than the modeled ones. The occurrence of vacancy clusters and voids is hardly identified by HRTEM. On the other hand, their distribution and sizes are sensitive to the nanostructure modeling. Therefore, a new characterization method is developed, which combines positron lifetime spectroscopy with the calculation of positron lifetimes from selected areas of the model samples. The first results are discussed.

2:15 PM B8.3

ATOMIC SCALE CHARACTERIZATION OF SUPPORTED AND ASSEMBLED NANOPARTICLES. B. Pauwels, M. Yandouzi, D. Schryvers, G. Van Tendeloo, EMAT, University of Antwerp, RUCA, Antwerp, BELGIUM; G. Verschoren, P. Lievens, Laboratory of Solid State Physics and Magnetism, Catholic University of Leuven, Leuven, BELGIUM; M. Hou, Physics of Irradiated Solids, Free University of Brussels, Brussels, BELGIUM; H. Van Swygenhoven, Paul Scherrer Institute, Villigen PSI, SWITZERLAND.

Different structural configurations of nanoparticles have been investigated by advanced TEM techniques. For Au clusters, produced in a laser vaporization source and deposited with low energy on MgO cubes and on amorphous carbon, the cluster-surface interaction was investigated by high resolution transmission electron microscopy (HRTEM). Special attention was paid to the influence of this interaction on the morphology of the Au clusters. Not only have the clusters different morphologies for the two different surfaces, but also a dilation of the Au lattice is measured for clusters deposited on the crystalline surface of MgO. This dilation perfectly accommodates the misfit with the MgO lattice. This behavior is modeled with classical molecular dynamics (MD) and the resulting structures are used as input for HRTEM simulations: good agreement between experiment and modeling is found. The same methods are used to produce and deposit clusters of different Au-Cu alloys in order to study the chemical ordering. Electron diffraction ring patterns obtained from Au_3Cu , $AuCu$ and Cu_3Au clusters lying on amorphous carbon, are indexed as face centered cubic with a lattice parameter of 3.87 Å, 3.82 Å, and 3.76 Å, respectively. This indicates that clusters of Au-Cu alloys are solid solutions and no ordering takes place in the clusters. For Ni-rich Ni-Al nanoparticles, prepared by the inert gas condensation technique and cluster assembled under an external pressure, the crystalline structure, grain size distribution and grain boundary structure are investigated. Non-stoichiometric 62 at%Ni samples contain B2, $L1_2$ as well as martensitic $L1_0$ grains with slight composition differences whereas close to stoichiometric 73at%Ni material consists completely of $L1_2$ grains, both correlating well with the phase diagram. Some amorphous areas averaging around 5 nm in diameter as well as incoherent and semi-coherent interfaces are observed in between adjacent grains.

2:30 PM B8.4

SIMULATION OF SURFACE MORPHOLOGY AND DEFECT STRUCTURE IN COPPER NANOPARTICLES. Yoshiaki Kogure, Masao Doyama, Teikyo University of Science and Technology, Uenohara, Yamanashi, JAPAN.

Formation of copper nanoparticles and defect structure in the particles are investigated by means of molecular dynamics simulation. The numbers of atoms in the particles range 100 and 10000. The EAM potential developed by the present authors is adopted to express the interaction between atoms in the simulation. The nanoparticles are formed by two methods. One is to coagulate the clusters consisted of dozens of atoms, which may correspond to the vapor deposition method. The other is to cool the molten state slowly and then to anneal the particle. Surface morphology of the nanoparticles are investigated in relation to surface energy. The generation of surface roughness at elevated temperatures is also observed in the simulation. It is related to the thermal stability of the nanoparticles. The defect structures such as grain boundaries, dislocations and point defects, in the particles are visualized through the potential energy and the local crystalline order, which distinguishes a fcc atom from the atoms of hcp or other crystal structures. The cyclic temperature variations are tried to eliminate the defects.

3:15 PM *B8.5

ACHIEVING SUPERPLASTICITY AND SUPERPLASTIC FORMING THROUGH SEVERE PLASTIC DEFORMATION. Terence G. Langdon, University of Southern California, Depts of Materials Science and Mechanical Engineering, Los Angeles, CA.

The application of severe plastic deformation to metals provides a convenient procedure for achieving nanometer and submicrometer microstructures. The most convenient procedure is through Equal-Channel Angular Pressing (ECAP) in which a sample is pressed through a die and experiences a very high strain with no change in the cross-sectional dimensions of the work-piece. This paper describes the use of ECAP in attaining materials with ultrafine grain sizes. It is shown that, provided precipitates are present to retain these small grain sizes at the high temperatures where diffusion becomes important, it is possible to achieve remarkably high superplastic elongations after ECAP. Furthermore, optimum superplasticity occurs at very rapid strain rates which provide the potential for making use of this procedure in developing a high strain rate forming capability. Examples of superplastic forming are presented and the results are compared with other attempts to achieve a high strain rate forming capability using conventional thermo-mechanical processing.

3:45 PM B8.6

SIZE-DEPENDENT MELTING OF MATRIX-EMBEDDED Pb-NANOCRYSTALS. H. Ehrhardt, G. Wilde and J. Weissmueller, Forschungszentrum Karlsruhe, Institute of Nanotechnology, Karlsruhe, GERMANY.

The melting point of any finite sample depends in a significant way on its size. For isolated particles the decrease of the melting temperature has been attributed to the reduced number of bonds for the atoms on the surface. Recent theories relate the melting-point variation as well as the reduction of the melting enthalpy more specifically to the size dependence of the mean square relative displacement (MSRD) of the atoms near the surface of the particle. The theories also consider matrix-isolated particles, where the interactions at the interface between matrix and particle, i.e. the changes in the binding energy as well as the topography of the interface have to be taken into account. We prepared matrix-embedded nanocrystal dispersions of Pb in Al by high-energy ball milling and examined the melting behavior of nanocrystalline Pb as a function of the average grain size. The mean-square relative displacement of the lead atoms was determined by analysis of x-ray radial distribution functions. Additional measurements of the melting temperature and melting enthalpy by DSC experiments on the same samples lead to a quantitative characterization of the size dependence of the macroscopic thermodynamic properties. This complete set of data is used to compare in detail theoretical model predictions on the size-dependent melting behavior with the current experimental results.

4:00 PM B8.7

STACKING FAULTS CREATED BY MECHANICAL MILLING IN NANOSTRUCTURED WC-Co COMPOSITE POWDER. Zhimin Yang, Jun Du, Changhui Mao, General Research Institute for Non-Ferrous Metals, Beijing, CHINA; Daniel Michel, Yannick Champion, Serge Hagege and Martin Hytch, Centre d'Etudes de Chimie Metallurgique / CNRS, Vitry sur Seine, FRANCE.

Nanostructured WC-Co powder, obtained by mechanical milling, was investigated in detail by combination of X-ray diffraction (XRD) and high-resolution transmission electron microscopy (HREM) techniques. HREM image analysis shows that in the as-milled nanostructured

powder, some WC grains are seriously faulted in terms of the stacking sequence of the perfect structure for WC crystal. The dominant faults are characterised by stacking faults lying periodically on the plane of {100}, which were created by mechanical milling process. Based on the HREM observation, a structure model for the faulted WC grains with stacking faults was proposed, which is in fact equivalent to a structure with space group of $R\bar{3}m$. Together with the perfect hexagonal structure (space group $P-6m2$) for the unfaulted WC grains, the new structure model used to describe the faulted WC grains was then put into the refinement procedure of the XRD experimental patterns using Rietveld method. The agreement between the calculated profile and the experimental one allowed to obtain the lattice parameters, grain size, microstrain and other structural information of the as milled powders.

4:15 PM B8.8

HOW CAPILLARY PRESSURE AFFECTS MATERIALS PROPERTIES INSIDE NANOCUSTERS. Ralf Meyer, Université de Montréal, Département de Physique, Succursale Centre-Ville, Montréal, Québec, CANADA.

Nanoclusters of Ag, Cu, and Ni with diameters in the range between 2 and 10 nm have been studied using molecular-dynamics simulations. Inside the clusters large capillary pressures build up, whose values are in good accordance with experimental values of surface tension. These pressures of the order of GPa are strong enough to influence materials properties and might thereby enable the fabrication of cluster assembled materials with specifically tailored properties. It is shown that the local vibrational density of states inside the clusters is shifted to higher energies by the capillary pressure and that it is well described by the corresponding bulk system under pressure.

4:30 PM B8.9

MOLECULAR DYNAMICS SIMULATION ON COALESCENCE OF SEVERAL EXPERIMENTALLY OBSERVED GOLD NANO CLUSTERS. Hong Bo Liu, Jorge A. Ascencio, Mario Perez-Alvarez, Miguel Jose Yacamán.

To reveal the coalescence processing and its rule of small nanoclusters is of importance and essence to understand and hence control the formation of relative large cluster and even assembled materials, in particular the growth of thin film. In the recent years, there are many attentions paid on this phenomena for many simulators, in which gold model nanoclusters are often taken as sphere morphology with fcc lattice or disordered arrangement. Actually, there are several different characteristics of isomers in gold, furthermore, small nanoclusters generally behave faceting, which likely have some certain effects on the coalescence processing and even is a dominant factor. Based on the above consideration, we deliberately select several experimentally observed gold nano clusters featuring with structural types and different sizes as our research objects and models. In our simulation, tight-binding potential is employed to describe the interatomic interaction of gold atom. In summary, the coalescence processing between nano clusters combining-pairs are captured in detail during our simulation, the behavior and rule of transition and reorientation are revealed. on the other hand, diffusion behavior, particularly collective diffusion are observed and quantified. Further, the time scale of the coalescence processing is monitored and finally clarified. This theoretical analysis is also corroborated by using HREM images of dynamical behavior of gold nanoparticles prepared by bio-precipitation.

4:45 PM B8.10

TIME RESOLVED HRTEM STUDY OF ATOMISTIC PROCESS IN SUSPENDED ONE-ATOM THICK GOLD ATOM CHAINS. Varlei Rodrigues^{1,2}, Daniel Ugarte¹; ¹Laboratório Nacional de Luz Síncrotron, Campinas, BRAZIL; ²Instituto de Física Gleb Wataghin - UNICAMP, Campinas, BRAZIL.

The understanding of the mechanical properties of nanometric junctions has huge interest for many domains such as electronics, wear, adhesion, friction, etc. Recently, the suspended chains of gold atoms have been identified as the ultimate junction atomic structure. Here, we present a real-time high resolution transmission electron microscopy (HRTEM) study of the atomistic process during the evolution of one-atom-thick gold chains. Nanojunctions were generated in situ in the TEM by using the electron beam to produce a nanometric bridge between neighboring holes in a self-supported polycrystalline gold thin film. Using this procedure, we have been able to experimentally get direct real space information on atomic positions, bond lengths, and also how they change during a rupture process. Atomic chains display extremely long interatomic distances (0.30-0.36 nm), but they are remarkably stable and their attachment points on a metal surface are capable of moving rather easily. The mobility of the chain fixing points may have important effects, for example it may change the atomic wire-apex electronic coupling and generate small variations (ex. substructures) in the quantum conductance response.