SYMPOSIUM B

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

November 28 – 30, 2000

Chairs

Diana Farkas
Dept of MS&E
Virginia Tech
201 Holden Hall
Blacksburg, VA 24061-0237
540-231-4742

Harriet Kung
Maths Science & Technology Div
Los Alamos Natl Lab
MS G755 MST-8
Los Alamos, NM 87545
505-665-4005

Merrilea J. Mayo
Dept of MS&E
Pennsylvania State Univ
Steidle Bldg
University Park, PA 16803
814-863-7330

Helena Van Swygenhoven
Paul Scherrer Inst
Villigen-PSI, CH-5232
SWITZERLAND
41-56-3102931

Julia R. Weertman
Dept of Math Sci & Engr
Northwestern Univ
MLSB
Evanston, IL 60208-3108
847-491-5353

Symposium Support
†FEI Company
†JEOL USA, Inc.
Los Alamos National Laboratory
Office of Naval Research
†2000 Fall Exhibitor

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

*Invited paper
SESSION B1: MECHANICAL PROPERTIES AND DEFORMATION BEHAVIOR 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 effects at the interface surfaces. 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 phases 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 Rung, Los Alamos National Laboratory, MS-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, nanocrystalline 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 overaging 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. Relations between internal structure and deformation will be discussed, including the influence of grain size on dislocation activity and contrast shifts.


The advent of nanocrystalline materials has provided new opportunities to explore grain size dependent phenomena. Superplasticity is a grain size dependent phenomenon defined by the ability to maintain a high strain rate over microseconds or more. Superplasticity in nanocrystalline materials has been well characterized. The constitutive equations that describe 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 ultrafine-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 the 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-9658321.

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 30 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 (10) 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 orientation are observed. The deformation 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 mechanical alloying 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 (quasi-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 mechanical spectra by a combination of quasi-static and dynamic measurements allowed to trace a draft phenomenological modeling of the elastic-plastic behaviour of nanocrystalline metals. In particular, creep and stress relaxation experiments at low temperatures (T<0.25 Tm) show a large time dependent recoverable strain with a thermally activated character. The activation energy is comparable with this 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. Kieferling, M. Hoffmann, University of the Saarland, Dept. of Physics, Saarbrücken, 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 free surfaces we are able to displace residual stress and 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. Ungar, G. Tóth, Eötvös University Budapest, Budapest, HUNGARY, T.G. Sanders, R. Mítrai, 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 prepared 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, $\varepsilon_{\mathrm{ave}}$. The three apparent size parameters are converted into the log-normal size distribution of grains by a Student t square method. The dislocation structure is determined by the line density and the $\langle \rho \rangle$-dependent average contrast factors of dislocations. Analysis of the experimentally determined $\varepsilon$ values has shown the presence of the usual $t_{\text{dis}}$ dependence of $\varepsilon$ as the grain size decreases, and especially below about 50 nm, it is
revealed that dislocations with cubic Burgers vectors \( <100> \) become more and more frequent. It is assumed that these sessile Lamott-type dislocations reside mainly in the grain boundary regions.

**11:15 AM B1.8**

**STRUCTURAL EVALUATION OF FCC MATERIALS MECHANICALLY MILLED**

Jiang Hong, Enrique 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 microscopy 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 nonmonotonically, some grains developed 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 were in high density. Some of the faults were observed throughout milling process; this is thought to be associated with the development of the final grain structure. Based on the basis of the present findings, a mechanical milling mechanism is proposed as follows. First, grains in a powder particle are deformed 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 affected by strain damage. Third, fracture occurs in the regions affected by strain damage; in other words, strain damage leads to fracture of edge regions of the grains into nanosized grains, conversely, nanosized grains rotate relative to the original grain. Finally, the entire grain is divided into nanosized grains by strain damage fracturing.

**11:30 AM B1.0**

**MOLECULAR DYNAMICS SIMULATION OF NANO-SIZED CRYSTALLIZATION DURING PLASTIC DEFORMATION IN AN AMORPHOUS METAL**

Ryuichi Tami, Akio Ogura, Masayuki Shimojo, Kazuki Takashima, Yukihiko 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 longrange ordering. It is therefore expected to be used as 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. Monte Carlo simulation is one of the most powerful 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 a nanosized crystallization during plastic deformation in an amorphous metal. In molecular dynamics simulation, an amorphous metal is modeled from 125 Na atoms together with 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 3.4 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 face structure which had an orientation relationship, i.e. the shear direction and \( \{111\} \) 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 nanosized 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 Tami, Masayuki Shimojo, Kazuki Takashima, Yukihiko Higo, Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, JAPAN.

Amorphous alloys, which have no longrange 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. Crystallization during plastic deformation is considered to be different from that during heat treatment. However, the detail of the crystallization during plastic deformation has not 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 nanosized nickel and FeSi-B amorphous alloys. Micropillar and cleaver beam type specimens with dimensions 10 x 8 x 55 \( \mu \)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 nanosized 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 amorphous 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 nanosized crystals during plastic deformation. The formation mechanism of nanosized crystals during plastic deformation is also discussed.

**SESSION B2: MECHANICAL PROPERTIES AND DEFORMATION BEHAVIOR OF BULK MATERIALS**

Chair: Diana Perkins

Tuesday Afternoon, November 28, 2000

Hempstead (Sheraton)

**1:30 PM B2.1**

**CYCLIC DEFORMATION AND FATIGUE PROPERTIES OF ULTRAFINE-GRAIN SIZE MATERIALS: A REVIEW OF THE CURRENT STATUS**

Huels Mohrbr, Universität Erlangen-Nürnberg, Institut für Werkstoffwissenschaften, Erlangen, FEDERAL REPUBLIC OF GERMANY.

The study of the mechanical properties and the microstructure of ultratine grain (UGF) size metals and alloys has become an important research area. A few years ago, it has become possible to produce ultrafine-grain, 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 ultrahigh 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 microstructure under conditions of cyclic deformation. Thus, is pressive improvements of the fatigue strength have so far been limited mainly to stress-controlled fatigue (equivalent S-N life, wear tests) which, in strain-controlled fatigue (Manison-Coffin plots) tend to reduce the fatigue strength. The 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 UGF 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 microstructural and metallographic studies on different types of materials. The current stress 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, Hyoung Seop Kim, Mark Bush, Department of Mechanical and Materials Engineering, University of Western Australia, Nedlands, WESTERN AUSTRALIA.

A constitutive model describing deformation behaviour of ductile metallic alloys will be presented. The model is based on a phase mixture rule implying that the interior of the grains and the grain boundaries can be considered as distinct 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 combination of ultra-fine powders, will also be discussed.

**2:15 PM B2.3**

**DEFORMATION MECHANISMS IN NANO-CRYSTALLINE TiAl-x AND NAI1 INTERMETALLIC ALLOYS**


The deformation mechanisms in the nanocrystalline intermetallic alloys TiAl-x and NAI1 have been experimentally investigated by
mechanical testing (compression and tension) at different temperatures ranging from 300 to 873 K and observation in transmission electron microscopy. The samples were produced by mechanical milling in an inert atmosphere and consolidated by a spark plasma sintering technique. Single phase materials with ordered structures were obtained after sintering. Tial-x and nanocrystalline TiAl-Ni alloys have the B2 structure. The resulting average grain sizes (d) vary between 30 nm (Al/Ti) and 150 nm. The value of d depends strongly on the sintering temperature. In the case of Tial-x, the grain size was varied between 70 and 120 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 Tial-x, Alloys with lower 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 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 a characteristic of the deformation of these materials at higher testing temperatures. Tial-x 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 523 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 B 2.4
UNIQUE MECHANICAL BEHAVIOUR OF SPD NANO-STRUCTURED ALLOY S. Ruslan Vasiliev, Aviation Technical University of Physics of Advanced Alloys, 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 dispersed particles of secondary 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 characterisation by TEM/HEM and X-ray, including SAXS. An especial attention is focussed on tensile mechanical behaviour of these SPD 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 #B 2.5
MECHANICAL PROPERTIES OF ELECTRODEPOSITED NANOCRYSTALLINE COPPER, L. Lu, M. L. Su, S. X. Lu, and K. Lu, State Key Laboratory 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 prepare a bulk nc copper specimen (grain size of 30 nm) with a high purity and high 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 as room temperature upon rolling, creep and fracture behaviors. Compared with the conventional nc Cu made by means of consolidation of ultrafine particles or bulk-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 example of 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 characterisation 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 B 2.6
ELASTIC SOFTENING OF NANOCRYSTALLINE Pd-H DUE TO HYDROGEN REDISTRIBUTION AT GRAIN BOUNDARIES. P. Buzan, C. Lemier, J. Weismuller, Forschungszentrum Karlsruhe, Institute for Nuclear Activation Research, Karlsruhe, GERMANY, and Universität des Saarlandes, Technische Physik, Saarbrücken, GERMANY.

At thermodynamic equilibrium, grain boundaries in nanocrystalline (nc) Cu/Ni or Cu/Ag alloys are known to be enriched in hydrogen. These grains are produced by mechanical milling in an inert atmosphere and consolidated by a spark plasma sintering technique. Single phase materials with ordered structures are obtained after sintering. Tial-x and nanocrystalline TiAl-Ni alloys have the B2 structure. The resulting average grain sizes (d) vary between 30 nm (Al/Ti) and 150 nm. The value of d depends strongly on the sintering temperature. In the case of Tial-x, the grain size was varied between 70 and 120 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 Tial-x, Alloys with lower 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 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 a characteristic of the deformation of these materials at higher testing temperatures. Tial-x 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 523 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.

4:00 PM B 2.7
EFFECT OF GRAIN SIZE DISTRIBUTION ON TENSILE PROPERTIES OF ELECTRODEPOSITED NANOCRYSTALLINE NICKEL, P. Buzan, C. Lemier, and K. L. Morgan, MSE 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 10mm 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 measured at 23 degree temperature. The grain size distribution was measured using transmission electron microscopy techniques. The grain size varied from a few nanometers to over 100 nm with average grain diameters in the range of 25 to 50 nm. 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 B 2.8
MECHANICAL PROPERTIES OF NANOCRYSTALLINE METALS IN RELATION TO THEIR MICROSTRUCTURE. F. Dalles Torsø, H. Van Svenningen, R. Schneebé, M. Victora, W. Wagner, Paul Scherrer Institute, SWITZERLAND, and J. Pell, Lusson, Dept. of Fusion Technology CRPP, SWITZERLAND.

Mechanical properties of nanocrystalline Cu, Ni, Pd and NiAl 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, 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 discuss microstructural evolution and to derive the mechanical properties of the materials. The grain size, its distribution and its evolution after deformation are studied using HREM for this purpose.

4:30 PM B 2.9
COMPUTER SIMULATION OF MISFIT DISLOCATION MOBILITY IN Cu/Ag AND Cu/Ag INTERFACES. Richard J. Kurz, Pacific Northwest National Laboratory, Richland, WA, Richard G. Herlitz, Los Alamos National Laboratory, Los Alamos, NM, Howard L. Heinrich, Pacific Northwest National Laboratory, Richland, WA.

Misfit dislocations exist in semicoherent Cu/Ni and Cu/Ag precipitates by co-deformation or physical vapor deposition. These dislocations display a novel theoretical tensile stresses and also substantial ductilities. Arrays of misfit dislocations exist in semicoherent interfaces to accommodate lattice parameter mismatch between layers. The mobility of misfit dislocations and their interaction with...
glide dislocations are reported here from the results of a molecular dynamics study of a sub-monolayered oriented Cu/Ag interface. The threshold shear stress to cause micro plasmons 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 interface on misfit mobility is examined and compared to the results of the sub-monolayer model. 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 up to 90% of the energy and a 10% probe beam, that is, by a purely 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. The dual frequency modulation technique is introduced in order to eliminate optical cross talk between the excitation pump pulse and the measuring probe pulse. Up to the 5th nonresonant echo in an aluminum layer on a glass substrate have been measured. Experimental results of further metallic thin film substrate combinations are compared with numerical simulations obtained by an elastic finite element integration technique. Several approaches describing the opto-thermo-mechanical interaction are compared and discussed. Further directions of the ongoing 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 solution. A slight increase in the milling temperature however results in a two- or even three-phase coexistence. The coexistence takes place in 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), microstructures of the ball-milled powders are investigated. Local 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 small strong scale (\( \approx 5 \text{ nm} \)) distortions due to a large density of dislocations (\( \approx 10^{13} \text{ cm}^{-2} \)), 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 milling can also be used to induce nanoscale decomposition, but in this case both phases seem to be connected. The hardness of these nanocomposites is measured by nanoindentation. Hardness values close to the theoretical shear strength are recovered, i.e., up to 6 GPa in the Cu-Co system. It is also observed that when the solid solution obtained by low temperature ball-milling exhibits already a large nanohardness (4 GPa in the Cu-Co 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 ALLOYING. B. Zhou, Jongsong Lee, Eric J. Lavernia, Univ. of California at Irvine, Dept. of Chemical and Biochemical Engineering and Materials Science, Irvine, CA.

The grain growth in a nanocrystalline aluminum (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 20 nm was produced by mechanical milling of pure elemental Al blende at the cryogenic temperature of -190°C. Grain growth kinetics was analyzed using two different models of grain growth, one of which takes pinning forces on the grain boundaries into account. The time exponent \( n \) from \( D_t^{1/2} \) dependence for the first Al to be<br>0.04 to 0.38 in a range of 0.01 to 0.03 of melting temperature (\( T_m \)), exhibiting the same general trend as high purity conventional polycrystalline Al with \( n \) increasing toward 0.6 as annealing temperature increases. The average grain size in the sample remains to be less than 100 nm even after annealing to temperatures up to...
80 °C for relatively long times (e.g., 200 minutes). The activation energy for grain growth in the n-Al was determined to be about 79 KJ/mol at the lower temperature range (80 °C). At 112 KJ/mol at the high temperature range. Possible mechanisms for the high grain size stability was discussed.

**B.3.7 STRUCTURAL AND ELECTRONIC PROPERTIES OF GOLD NANOWIRES GENERATED BY MECHANICAL STRETCHING.** Varleli Rodrigues -2, Daniel Ugarte -1, **Laboratório Nacional de Luz Ultravioleta, Campinas, BRAZIL; Instituto 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 been allocated to the electrochemical characterization of ballistic nanowires, 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 ultrahigh-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 nanowires 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], [110] and [111]) lies approximately parallel to the elongation direction. Nanowires following [100] and [110] directions display a pyramidal shape and evolve gradually to form nano-atom-thick wires. On the other hand, nanowires along [111] ones show rod-like morphology and break abruptly when they are a few nanometers thick. We have developed a model for the three possible atomic configurations and, predicted their conductance evolution and occurrence probabilities. We developed this model with the UHV-MCBJ conductance measurements and obtained a remarkable agreement. This is an important result, because it allows the manipulation of nanowire structure with the conductance behavior.

**B.3.8 SIMULATION OF POSITRON CHARACTERISTICS IN NANOCRYSTALLINE MATERIALS.** Jan Krupka, Charles Univ, Dept of Low Temperature Physics, Prague, CZECH REPUBLIC; Steven Van Petegem, Danny Segers, Charles Daudey, Ghent Univ, Dept of Subatomic and Radiation Physics, Ghent, BELGIUM; Pierre Hoss, 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 electronic (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 we report on the extension in a computational technique to determine extraction properties of nanocrystalline materials. It is presented in such calculations breakdowns of materials obtained using hybrid theoretical methods. 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 approximate '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-wires, and bulk-like regions. We study the virtual samples of n-C, n-N, n-NiAl, 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.

**B.3.9 POSITRON LIFETIME MEASUREMENTS IN NANO-STRUCTURED Ni-Al SAMPLES.** Steven Van Petegem, Danny Segers, Charles Daudey, Ghent Univ, Dept of Subatomic and Radiation Physics, Ghent, BELGIUM; Florain Dalha Terre, Helena Van Swygenhoven, Paul Scherrer Inst, Villigen, SWITZERLAND; Mohammad Yedim, Dominique Schreyers, Gustav Van Tendek, Univ of Antwerp, Centre for Electron Microscopy and Materials Science, Antwerp, BELGIUM, Jan Krupka, Charles Univ, Dept of Low Temperature Physics, Prague, CZECH REPUBLIC; Pierre Hoss, 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 nanocrystalline ones. The size of free volumes associated with defects can be estimated using the lifetime components found in the positron annihilation spectrum. The experiments are performed on nanocrystalline Ni-Al, (~2.75 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 intersections of different phases. The third lifetime 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.

**B.3.10 LOGARITHMIC RELAXATION OF ELECTRICAL RESISTANCE IN PLASTICALLY DEFORMED Au4Fe2, at ROOM TEMPERATURE.** Paolo Allini, Marco Coisson, Vincenzo Staggini, DIII-DISPE, Politecnico di Torino and INFIM, Torino, ITALY; Paolo Tintirotto, Franco Vinai, IEN Galileo Ferraris and INFIM, Torino, ITALY; Marcello Biondo, Enrica Bocco, Diego Palesti, Dip. IFIM, Universita di Torino and INFIM, Torino, ITALY.

Rigidly solidified Au4Fe2 ribbons (thickness 60 μm, width 2 mm) were prepared from a homogenized master alloy by plasma flow casting in controlled atmosphere at 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, meaning 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 μΩcm 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 a 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 Fe or FeC. On the other hand, no relevant deformation-induced grain boundary re-arrangements are expected to occur, because these ribbons are characterized by the presence of large, stable grains. The electrical resistivity of Au4Fe2 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.

**B.3.11 EFFECT OF DEPOSITION PARAMETERS ON TENSILE PROPERTIES OF PULSE-PLATED NANOCRYSTALLINE NICKEL.** KL. Morgan, Z. Ahmed and F. Ebrahimi, MS&E Dept., University of Florida, Gainesville, FL.

Electrodeposition is a viable method for fabricating nanocrystalline materials. 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 0.3 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 gauge 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.

**B.3.12 DEFORMATION OF NANOCRYSTALLINE Ni INVESTIGATED BY TEM SIMULATION COUPLED TO MD SIMULATION.** R. Schunklin, Fusion Technology + Materials, Centre de Recherches en Physique des Plasmas, Ecole Polytechnique Federale de Lausanne, Villigen PSI, SWITZERLAND; H. Van Swygenhoven, Paul Scherrer Institute, Villigen PSI, SWITZERLAND.

EM images simulations are used together with molecular dynamic
assessments of capsule mechanical properties is now available using this testing technique which will facilitate the design of spherical capsules as a protective material. Various capsule geometries were fabricated using materials from different seeds with random locations and orientations. TEM images are simulated using the multislice method. The deformation behavior is studied by accessing to high-resolution TEM image, diffraction contrast image and the strain 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.

D3.13
EXTENSION OF HIGH CYCLE FATIGUE LIFE BY THE
FORMATION OF NANO-SIZED MARTENSITE PARTICLES AT INTERSECTIONS OF DISLOCATIONS IN AN AUSTENITIC STAINLESS STEEL
Naotaka Imamura, Masakazu Shimojo, Kuniki Takashima, Yokiichi 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 is expected to be hampered when the accumulated density of available mobile 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 when dislocation slip occurs in austenitic stainless steels at 680°C temperature during cooling. According to Bogers and Burgers, two (111) < 112 > shear transform fcc-rolling into bcc-rolling. Thus, it is suggested that bcc-like stacking regions exist at intersections of partial dislocations and may transform easily into fine alpha- martensite particles at a certain cryogenic temperature above 680°C 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-1In-0.5Mn) was cryogenically treated above 680°C 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.

D3.14
MONTE CARLO SIMULATION OF GRAIN BOUNDARY SLIDING AND MIGRATION EFFECT OF VACANcies Peter Balli,
Department of Physics, Slovak Technical University, Bratislava, SLOVAK REPUBLIC; Nikolaus Kaczus and Geng Liu, 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 Σ5 [001][210] tilt grain boundary (GB) in aluminum and the effect of vacancies on the sliding properties. We find that the simultaneous annealing allows the system to gradually unravel to a globular-aluminum 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 are from that 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.

D3.15
BERYLLIUM DENSIFICATION AND STRENGTH ENHANCEMENT BY BIASED DEPOSITION. Alan Janczakowski,
Lawrence Livermore National Laboratory, Livermore, CA.

A mechanical testing technique is developed to load thin-walled spherical capsules in the uniaxial 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 capsule is prepared by magnesium sputter deposition of Be and Be alloys onto spherical mandrels. The collection 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
situ TEM straining experiments. These results are correlated with hardness measurements from nanoindentation testing.

9:30 AM B4.4
SUPERRHEOLOGICAL DEFORMATION OF ADAPTIVE NANOCOMPOSITES. Julia Mauzerall, Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, MD; Alexander L. Roytburd, Dept of Materials Science and Engineering, Montana State University, College Park, MD.

We model the superrheological deformation of multilayer nanocomposites consisting of thin adhesive layers of a shape memory alloy separated by the thin layers of a passive metal. It is shown that the martensitic transformation in the constrained nanolayers proceeds through controlled evolution of polydomain microstructure resulting in superrheological 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 with respect to stress. The 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. Howland, H. Kang, A. Mora, Los Alamos National Laboratory, Los Alamos, NM; J.D. Embury, McMaster University, Hamilton, Ontario, CANADA.

The strength of metallic nanolayered compositions, even when constructed of combinations of relatively soft for 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 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 cubic and semi-coherent interfaces between different for 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 semi-coherent interfaces, while serving to relax k-space coherence 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 STRESSES IN Ag-Ni [11] 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 reconstruction of the lattice normal to the interface. In Ag-Ni multilayered thin films, the in-plane strain relaxation due to interdiffusion is a consequence of the relaxation of the interfacial strain. Interdiffusion and interfacial strain relaxation have been shown to affect the stoichiometry of the thin films. In this study, the stress-strain response of Ag-Ni multilayered thin films was studied using x-ray diffraction and/or x-ray stress analysis. The results indicate that the stress-strain response of Ag-Ni multilayered thin films is sensitive to the interdiffusion of Ag-Ni and that the interdiffusion process can affect the stress-strain response of Ag-Ni multilayered thin films. The results suggest that the interdiffusion of Ag-Ni may affect the stress-strain response of Ag-Ni multilayered thin films and that the stress-strain response of Ag-Ni multilayered thin films is sensitive to the interdiffusion of Ag-Ni.
COHERENCY STRAIN AND A NEW YIELD CRITERION. N.B. Jangwener, 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. Busby, 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 J. P. Delhaes, 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-constrained-layer superlattices, using nanodiffraction 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 changing the strain, superlattices with a micron 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 solution compared to the usual inverse Hall-Petch behaviour, 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 the entire length of the crystal across. The result has implications for finite-element modeling of point contact, and for the design and understanding of materials with coherency-strained microstructure.

SESSION B5/W4: JOINT SESSION
Chair: Deryl C. Chorom and Richard G. Hoggland
Wednesday, Afternoon, November 29, 2000
Room 310 (Hynes)

1:30 P.M. *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 Choobshi 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 nanocrystalline regime. However, it was subsequently pointed out that for most of the experiments performed, the inverse Hall-Petch behavior was not observed, the observed size change was by recrystallization of the initially finest grain size specimens to grow the grains. It was suggested that thermally treating nanocrystalline samples may cause changes in the stress-induced grain growth in both grain size (e.g., from a microstructure, 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 the occurrence. Molecular dynamics simulations have also predicted the inverse Hall-Petch effect. Since conventional dislocation-induced deformation mechanisms are unlikely at the finest nanocrystalline 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.


Support from NSF under grant no. DMR-9871810.

2:00 P.M. B5.2/W4.2
LARGE TENSILE DUCTILITIES AND ULTRAHIGH YIELD STRENGTHS AT ROOM TEMPERATURE IN NANOCRYSTALS.
TALLINE Fe-Co ALLOY.
Chan Il Chang, C. Van Heeringen, H. C. Cammarata, The Johns Hopkins University, Department of MS&E, MD; C. Y. Chen, 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 conventional 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 Fe-Co alloys that were fabricated using bulk processing techniques. The microstructural observations showed very attractive mechanical properties at room temperature with yield strength ranging from 2 GPa to 3 GPa and tensile ductilities ranging from 3% to 4%. Both tensile strengths and hardnesses follow Hall-Petch relations, and the fracture surfaces show ductile facets. Results from TEM, SEM, and X-ray diffraction investigations will be used to describe the microstructure of these flow-free, nanocrystalline materials.

2:15 P.M. B5.3/W4.3
DEFORMATION BEHAVIOR AND FAILURE MODE OF CONSOLIDATED FULL-DENSITY NANOFASE Fe AT QUASI-STATIC AND HIGH STRAIN RATES.
Y. M. Wang, K. T. Ramesh, and E. Mas, Department of Mechanical Engineering, Department 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, has 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 of deformation in our consolidated materials. Little strain rate sensitivity of the flow stress is observed over a wide strain rate range (up to 500/s). These materials are characterized by 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 bar alloys in military kinetic energy penetrators where the shear banding mode and self-sharpening capability during high rate deformation are desired.

2:30 P.M. *B5.4/W4.4
THE LIMIT OF GRAIN REFINING STRENGTHENING IN IRON.
Satoshi Takashi, Kenji Kawasumi, Kyushu Univ, Dept of MS&E, Fukuoka, JAPAN; Yoko Inoue, National Research Institute for Metals, Baraki, JAPAN.

Mechanical milling [MM] using a high energy ball mill was applied to iron powder to obtain ultrafine-grained iron. After treatment, the iron powder with the hardness HV0.3 GPa was about 25 GPa. In the Hall-Petch plot of such ultrafine-grained iron, a linear Hall-Petch relationship was confirmed to around 10 mm but the plots below 10 mm 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 10 mm but the hardness was also much smaller than the value expected by the extended Hall-Petch line and showed a tendency of leveling-off at HV0.3 GPa. As a result, the grain size of iron is seemed to be refined to around 10 mm by severe deformation and the hardness achieved by grain refining is thought to be about HV0.3 GPa (expected yield strength: 3.6 GPa).

3:30 P.M. *B6.5/W4.5
ATOMIC STUDIES OF PLASTICITY IN NANOFASE METALS. A. C. Coro, Centro Atomico Bariloche, Bariloche, ARGENTINA; T. Y. Van Ingenhoos, P. Derlet, Paul Scherrer Institute, Villigen, SWITZERLAND; D. Farkas, Dept. of MS&E, Virginia Polytechnic Inst. & State Univ., Blacksburg, VA; M. J. Curti and T. Diaz de la Rubia, Lawrence Livermore National Lab., Livermore, CA.

We have studied structural, energetic, elastic, and plastic properties of a family of computer generated nanophase samples of Cu and Ni containing 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 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 the phonon structure as the grain size decreases, with the phonon density of states becoming higher at smaller nanocrystallite sizes. This observation is of primary importance in the interpretation of the inverse Hall-Petch relation. Experimentally, it was found that the phonon density of states becomes higher at larger nanocrystallite sizes, which supports the idea that the phonon density of states is inversely proportional to the grain size. Additionally, computer simulations provide access to a microscopic view of the deformation processes. In this way, we obtained 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 extracted 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 1-9s), our input microstructures were tailored (i) to a uniform grain size of about 10 nm and (ii) contain only high-energy grain boundaries, which are known to exhibit rapid plastic deformation. Our simulations reveal a high level of dislocation density, and a grain size of 10 nm, with a strain rate of 10^-4 s^-1, that agrees quantitatively with that given by the Cowper-Tabor model. The extension of these results to larger temperatures, with emphasis on the crossover in the Hall-Petch effect, is discussed.


4:30 PM B5.7/W4.7
GRAIN MICROSTRUCTURE EVOLUTION IN NANOSCALE MATERIALS: RECENT SIMULATION RESULTS.
Monirul Ummayyan1,2,3 and David J. Srolovitz1,2
Princeton Materials Institute, Princeton, Princeton University, New Jersey. 1Dept. of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ. 2Dept. of MSE, University of Michigan, Ann Arbor, MI.

The mechanical properties of polycrystalline materials vary 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 the Coble creep. Such effects are especially important on the nano-scale. In this study, we examine several issues associated with the evolution of the microstructure. In particular, we report on some important issues in grain size evolution that have become important when the grain size is very small. These studies were performed using molecular dynamics simulation in two-, three-dimensions. We examine the range of grain sizes over which the classical concepts of grain boundary mobility and curvature drive 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 explicitly excluded grain boundary triple junction mobilities for a large number of grain orientations. We find that while triple junctions provide a driving force at very small grain sizes, the grain size dependence is not strongly consistent with expectations based on the misorientation dependence of the boundary energy and does not require the motion of intrinsic dislocations.

4:45 PM B5.8/W4.8
THE HALL-PETCH RELATION IN NANocrystALLINE METALS
J. Schütz, 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 the possible Hall-Petch effect, 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 nanoscale simulations to demonstrate that copper and palladium with grain sizes below 15 nm exhibit a reverse Hall-Petch effect, caused by sliding in the grain boundaries [Schröder et al., Nature Materials 2004; 361]. Using these simulations, a model is presented that gives 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 grain boundaries, and grain boundary sliding.

SESSION D6: POSTER SESSION.
Wednesday Evening, November 29, 2000
8:00 PM
Exhibition Hall A (Hynes)

B6.1 NOVEL TUNGSTEN CARBIDE NANOCRYSTALLINE COMPOSITES BY PULSED LASER DEPOSITION

We have developed a novel processing technique to fabricate artificial free Tungsten Carbide nanocomposites. In this method, a modified pulsed laser deposition of WC in conjunction with a very small amount 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 aluminate deposited in each pulse. The role of NiAl is to enhance the nucleation sites of WC and ensure the three-dimensional nucleation of Tungsten Carbide grains. It has a high surface energy and is also insulative in Tungsten Carbide. Using this approach, we 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 size and hardness values obtained by nanoindentation techniques will also be discussed.

B6.2 CERAMIC NANOALLOYS FROM EUTECTIC MELTS: THE SYSTEM CaO-ZrO2-Y2O3-ZrO2-Y2O3-Y2O3-Y2O3
Hiroshi 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 aluminoyttriamosaic. The composite was self-assembled in one quick step by rapid quenching of the multioxide aluminum-Y2O3 system melt in a molar furnace. The eutectic composite has a nanosized structure of eutectic lamellas, with lamellas 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 nanocomposites – will have excellent thermomechanical stability at high temperature compared to any sintered ceramics, and excellent prospects for applications in insulating and high temperature components. 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 nanosized self-assembled eutectics.

B6.3 GROWTHS WITH THE RUTILE STRUCTURE: A TIGHT-BINDING CALCULATION OF THE ELECTRONIC STRUCTURE
A.M. Marchesi, 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, films and thin layers which have a structure with a grain size of 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 microcrystalline films from the properties of the basic building block, that is, a real crystalline grain which is described as quantum mechanical level. This approach has been adopted also in this contribution. In this work, we present the electronic properties of crystalline grains of TiO$_2$ and SnO$_2$ 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 Mirovskii, 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]$_{1-x}$[B$_x$C$_x$]$_x$ superlattice (SL) with ultra-short period. It is based on the idea that under such SL, a composite 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$_x$B$_{1-x}$C$_x$ crystal from the properties of its basic building block. 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. Mischel, T. Hinklin, R.M. Laine, Dept. of Materials Science and Engineering and Chemistry, University of Michigan, Ann Arbor, MI.

Nano-sized YAG powders can be produced by flame spray pyrolysis (FSP) from very small yttria and alumina precursors, that can be doped with various metallic. This scalable synthesis route produces single crystal nanoparticles at > 100 g/h with surface areas of 50-100 m$^2$/g. Typically, the as-sprayed particles consist of unaggregated partially amorphous powders, with sizes 2800 nm diameter. As-shot powders are not crystalline YAG, but are converted to monocryalline unaggregated YAG powder after low temperature heat treatment. These powders can be used to make YAG monoliths, fibers, and novel photonic material. 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. Mischel 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 oxide and silicate complexes as well as other soluble metal complexes. This scalable synthesis route produces reproducible, high purity nanosized powders. We have previously studied single crystal TiO$_2$. CeO$_2$, crystalline mullite (3Al$_2$O$_3$ - 2SiO$_2$) alumina and nepheline syenite aluminoc phase (3Al$_2$O$_3$ - 2SiO$_2$) powders with controllable average particle sizes of 3-200 nm and surface area > 45-100 m$^2$/g. The powders are characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The powders are also characterized using X-ray photoelectron spectroscopy (XPS) and infrared spectroscopy (IR).

B6.7 PROCESSING AND CHARACTERIZATION OF Co$_3$Al$_2$O$_4$ NANO Structured Composites Synthesized by In-Situ Reduction. M.S. Matta, E.A. Brochi, P.R. Jens and I.G. Schirran, Catholic Univ. Rio de Janeiro, Dept. of Materials Science and Metallurgy, BRAZIL.

The copper-alumina system is reported to have excellent combination of properties that are essential dependent on their microstructure, which, in turn, will vary according to the preparation method. In this work, the synthesis of the Co$_3$Al$_2$O$_4$ nanomaterial was carried out by in-situ reduction of a powder mixture consisting of CuO and Al$_2$O$_3$, previously obtained through a chemical route. The procedure involves the reduction of the product special features in terms of microstructure. FEG SEM observations have suggested the formation of a composite with monoclinic 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 Cu-O clusterization in some regions. TEM bright field and centered dark field imaging have shown well-developed copper crystals surrounded by Al$_2$O$_3$, 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. Shigeo Kuroyanagi and Noriko Yamada, Nippon Steel Corporation, Advanced Technology Research Labs, Futsu, JAPAN.

Inorganic/organic hybrids prepared from polydimethylsiloxane (PDMS) and inorganic colloids (TEOS) have flexible and rubbery properties. However, they have only an inorganic component of the silicone network derived from TEOS. We successfully fabricated flexible sheets of PDMS-doped hybrids from PDMS and chemically modified metal alkoxides in 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 XEAFS, 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 3.0 MPa. When the storage modulus of 10$^{-2}$ Pa in rubbery region was approximately constant at temperatures of 200°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, MFT, 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.

Nano fabrication technology has recently been opening up new opportunities for innovative magnetic materials and devices. It is anticipated that novel magnetic materials required to meet the demands of high density recording are characterized by such techniques can be useful for recording media for future high-density recording and can increase the recording density beyond 100 Gbit/in$^2$. For future application, structures e.g. nanowires are believed to be the most promising recording media. We report the fabrication of nanostructured network of an important recording media, gamma iron oxide (+Fe$_3$O$_4$) by rf magnetron sputtering and reactive magnetron sputtering of an iron target with a capping 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 nanowires deposited at lower substrate temperatures and oxygen pressures are found to be under tensile stress due to the porous nature of the film. The gamma iron oxide nanowires deposited at higher substrate temperatures and oxygen pressures are found to be compressively strained indicating an in-channel annealing of the films. The surface topography of such nanowires by Atomic Force Microscopy (AFM) shows relatively larger topography 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 Kwarsmo, EG&G Inc.,
Groton, CT; Roger Richards, Naval Undersea Warfare Center Division, Newport, Newport, RI.

Lead magnesium niobate (PMN) ceramic is a reduced 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 related 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 substitutions such as strontium and barium.

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

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


Development of hybrid materials from cubic silsesquioxanes [([RSiO]₃ · n)ₙ] 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]₃ · n)ₙ 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 form 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 or at length scales typical of interphase materials and the composites should be wholly interphase.

6.13 SYNTHESIS AND CHARACTERIZATION OF PERIODIC NANOSTRUCTURED ORGANOSILICATES BY USING BLOCK COPOLYMER TEMPLEATES. Hyun-Bum Cho, Dae-Hwan Kim, Sung-Man Cho, Kookheon Cho, Seoul National University, School of Chemical Engineering, Seoul, KOREA.

Nanostructured organic-inorganic hybrids and nanocomposites can be achieved by solgel 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. Nanostructured organosilicates showing well-defined channels can be applied to a wide range than materials obtained only by TEOS. We have tried to prepare more mechanically stable porous materials by using organosilicates such as methyltriethoxysilane (MTEIS), dimethoxydimethylsilane (DMDS), and 1,3-bis(trimethylsilyl)ethane (BTMS). Methyl methacrylate (MMA) of MTEIS, DMDS, and BTMS as monomers and TEOS, MTEIS, DMDS, and BTMS as inorganic precursors. We have investigated the reaction mechanism between the block copolymers and the organosilicates, and obtained nanocomposite films as well as nonporous organosilicate powders by varying experimental conditions. We have also studied the pore shape and packing structure of the nanomeric materials using small angle X-ray scattering (SANS), BET isotherm, transmission electron microscopy (TEM), and nuclear magnetic resonance (NMR).

6.14 PROPERTIES OF COLLOID-LIQUID CRYSTAL COMPOSITES. Jason Craig, R.J. Lavery, S.P. Mcker, W.C. 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 magnetic transition the colloidal particles are expelled from the expelled from the field of increasing confined to a nematic phase and become localized in 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 wedge, is 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 10% volume fraction. Such composites are mechanically rigid and self-supporting. A rigorous theoretical description of the mechanical properties of this class of material 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 optical 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 in view of potential applications in liquid crystal display devices.


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 applied to a wide range of 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 solution is brought to reflux using a heating mantle as the heat source. In this study, a millimeter-wave beam provided by a Gycron Ltd., 83 GHz CW gyrotron [operated at less than 280W 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 difference 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: Merrilee J. Mayo
Thursday Morning, November 30, 2008
Hampton (Sheraton)

8:30 AM PDT
BREAKING THE NANOGRAIN BARRIER IN SINTERED
9:00 AM B7.2
PROCESSING AND MECHANICAL PROPERTIES OF NANO-SCALE COMPOSITES FROM POLYMERIC PRECURSOR, HIGH-PRESSURE AND SPARK PLASMA SINTERING ROUTES. Amiya K. Maldaverje, Julin Wain, Matt J. Gerash, Joshua D. Kuntz, University of California, Dept. of Chemical Engineering & Materials Science, Davis, CA.

The engineering of 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/alumina carbide ceramic composites. The structural evolution from polymer to nano-ceramic, two processing routes have been developed to produce silicon nitride/alumina 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/alumina carbide composite with nanometric grains. Method 2 involves the spark plasma sintering of pyrolysis-derived porous powder with ceramic additives. Results, so far, indicate remarkable flexibility in microstructural design and possibly improved high temperature properties of nanocomposites produced from polymer precursors.

9:15 AM B7.3
MULTIMODAL POWDERS AND COATINGS: A NEW APPROACH TO THERMAL SPRAYING. Gohsh Sk Pang, Rave Yiv, Nanopower Enterprises Inc, Piscataway, NJ, Bernard H. Ker, Rutgers-The State University of New Jersey, Piscataway, NJ, Yunfei Qiao, Lucy Liu and Trongdai 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 nanomaterial 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 nano-constituent fills the available pore spaces between the heated and softened aggregates, providing a strong and tough coating. The deposited composite, in conjunction with the composite 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. Characterization of these multimodal coatings with the structure and properties of the resulting coatings, will be discussed.
11:00 AM B7.7
STRUCTURAL DISORDER IN THE ANION LATTICE OF NANOCRYSTALLINE ZIRCONIA AND HAFNIA PARTICLES.
Dietz Vollath, D. Vinh Sanh, Michael Hageleisen,
Forschungszentrum Karlsruhe, Karlsruhe, GERMANY; Manfred
Ferker, University Bonn, Bonn, GERMANY.

Nanoparticles often crystallize in different structures than bulk materials. Typical examples are zirconia, 
ZrO2 and hafnia, HfO2, crystallized from the melt. Yet, bulk materials which contain these
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 possible cubic lattice for both materials.
The comparison of the magnitude of the Fourier transforms of 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 case a broadening of peaks, 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
corner should its shoulder was neither observed on 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 CHARAC,
TERIZATION OF CERAMIC/ CERAMIC/ THERMAL NANOCOMPOSITE.
THIN FILMS BY XANES, EXAFS, SAXS, HRTEM, AND
NANOINDENTATION, W.J. Meng, D.E. Alexander1, D.M. Cao, B.
Feng, R.C. Tittsworth 2, Mechanical Engineering Department,
Louisiana State University. 1 Materials Science Division, Argonne
National Laboratory Center for Advanced Microstructures and
Devices, Louisiana State University, LA.

Using Ti-containing hydrocarbon (Ti-C-H) thin films deposited by plasma assisted CVD (PA-CVD) technology as an example, we
demonstrate a general experimental methodology for in-depth characterisation 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 TiOx-H and CxOy-H nanostructures 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 nanostructures 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 mechanical properties are controlled by TiC/a-C:H nanocomposites and
conform well to micromechanical bounds for two-phase materials. Further results on TiC/a-C:H and TiC/a-C:H thin films serve as a basis with which data on other nanocomposite thin films are compared.

11:30 AM B7.9
NANO-MECHANICAL PROPERTIES OF NACRE: A BIOMIMETIC MODEL FOR LAYERED COMPOSITES. EXPERIMENTS AND
MODELING, Jeffrey M. Syps, William N. Mercer, Hanson Fang,
and Nellesen Sanjaya, Materials Science and Engineering, University of
Washington, Seattle 98195, USA; K. Neoh, University of Cambridge, 

Organisms produce biocomposites of inorganic and biomolecules,
intricately architected hierarchically, at all dimensional scales
exhibiting physical properties superior to synthetic materials with
similar phase compositions. These include bone, dental tissues, 
muscle, skin, hair, hair, scales, scales, and feathers. These
biocomposites are excellent models for nanocomposite engineering
materials. A truly biomimetic design requires a knowledge of detailed
inter and interface structures and local properties of individual
biomacromolecules. Using a nanomechanical system at atomic
force microscope, we determined mechanical properties of both
the nacre 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 a 3D finite
element analysis model that analyzed detailed nacre structures. These results
revealed hitherto unknown properties, potentially the basis for
biomimetic design of layered hybrid composites. Here, a sharp
microindentor (diamond Berkovich tip) was used to determine local
hardness and elastic modulus of the biomineral aragonite crystals
and these are compared with those of large single crystal geological
materials over a range of length-scales. Similarly, the properties of
the organic matrix are determined using specially-engineered blunt,
semihedral (metallic) tips at very small stresses on cleaned 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 of the nacre (e.g., elastic modulus) of individual
components. Based on these experimental results, we constructed a
nanoscale three-dimensional model nacre using a non-linear finite
element method (FEM) to simulate strain-state effects and large
deformations in organic phase. We incorporated interface mechanics
to 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. Vas Vleihan,
S. Suresh, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA; E.S. Ahn,
J.Y. Vleihan, 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, inhomogenieties, and impurities introduced
during conventional processing of this ceramic limit its mechanical
strength. Alternatively, nanostructure processing of HAP has achieved microstructural and chemical homogenity 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 nanostructure 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
micromanipulation. 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 nanoindentation technique scales very small volumes, we will also evaluate the extent to which our processing has achieved microstructural uniformity.

SESSION 88: CLUSTERS AND OTHER NANOSTRUCTURES
Chair: Julia R. Weinstet
Thursday afternoon, November 30, 2000
Hampton (Sheraton)

1:30 PM B8.1
MECHANICAL PROPERTIES OF NANOSTRUCTURED ALUMINA-TITANIA COATINGS, L. Sheng, D. Goberman, Y.H.
Sohn, E.H. Jordens, and M. Gei, Department of Metallurgy and
Materials Engineering, Department of Mechanical Engineering,
University of Connecticut, Storrs, CT.

Nanocoated coatings have potentials to provide significant
improvements over coarse-grained counterparts in load-bearing
applications owing to enhanced hardness and toughness. In this
paper, Al2O3 - 13 wt % TiO2 coatings formed via thermal spray
approach using reconstituted nanoscale Al2O3 and TiO2 powder
powders are described. Microhardness, indentation crack growth resistance,
adhesion strength, spallation resistance during bend and impact,
abrasive wear resistance, sliding wear resistance and single scratch
resistance of the coatings obtained from the nano-powder layers have been
characterized and compared to those obtained from commercial coating
counterparts. The properties of the coatings from the nano-powder layers are discussed and related to their microstructures and
thermal spray conditions.
The properties of elemental and bimetallic 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 nanomaterials is evaluated. The formation of molecular structures with high resolution transmission electron microscopy (HREM) allows to predict fine detail of the lattice relaxation of a truncated octahedral gold cluster deposited on MgO. Montecarlo Monte Carlo (MC) predicts that a lattice mismatch may contribute to disordering in deposited Cu-Au 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, clusters formed by condensates are obtained for both methods. The existence of a combination of Molecular Dynamics and MC. Whatever the nanomaterial investigated, the segregation properties of free clusters remain in the nanometer scale. The segregation is found to be a loss of formation of a metastable martensite phase as observed in bulk Ni-Al alloys. The prediction is consistent with HREM 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 HREM. On the other hand, their distribution and sizes are sensitive to the cluster morphology and sizes. 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.

Difficult structural configurations of nanoparticles have been investigated by advanced TEM techniques. For Au clusters, produced in a inert gas condensation and deposited on MgO and on amorphous carbon, the crystal structure interaction was investigated by high resolution transmission electron microscopy [HREM]. 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 dilution of the Au lattice is measured for clusters deposited on the crystalline surface of MgO. This dilution perfectly accommodates the mismatch with the MgO lattice. This behavior is modeled with classical molecular dynamics (MD) and the resulting structures are used as input for HREM simulations: good agreement between experiment and modeling is found. The same methods are used to produce and to characterize different Au-Al alloys in order to study the chemical ordering. Electron diffraction ring patterns obtained from Au-Al and Cu-Al clusters lying on amorphous carbon, are indexed as face centered cubic with a lattice parameter of 3.82 Å, and 3.76 Å, respectively. This indicates that clusters of Au-Al and Cu-Al 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 crystal structure, magnetic properties, and boundary structure are investigated. Non-stoichiometric Ni$_{72}$Al$_{28}$ samples contain B$_2$, L$_1_2$ as well as martensitic L$_1_0$ grains with slight composition differences whereas close to stoichiometric Ni$_{75}$Al$_{25}$ metallic content completely the L$_1_0$ structure. The core-shell model is used to correlate the grain morphology with the phase diagram. Some amorphous area merging around 5 nm in diameter as well as incipient and semi-coherent interface are observed in adjacent grains.

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 1000. The EAM potential developed by the present authors is adopted to express the interaction between atoms. The structural and thermal properties are formed by two methods. One is to optimize 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 and 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 pores, are formed 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.

The application of severe plastic deformation to metals provides a convenient procedure for achieving nanometric and submicrometric 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 workpiece. 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 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.

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 and the size dependence of the mean square relative displacement (MSRD) of the atoms near the surface of the particle. The theories also consider matrix-related 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 grain size. The mean-square relative displacement of the lead atom was determined by analysis of x-ray radial distribution functions. Additional measurements of the melting temperature and melting enthalpy by DSC experiments on the nanocrystalline samples allowed 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 experimental current results.
powder, some WC grains are seriously faulted in terms of the stacking sequence of the perfect structure for WC crystal. The dominate 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 Amm2. 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 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, Co, 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-Abreu, Miguel Jose Yacaman.

To reveal the coalescence processing and its role 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 face lattice or disordered arrangement. Actually, there are several different characteristics of isomers in gold, furthermore, small nanoclusters generally behave fusing, 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 role of transition and recombination 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 HREM STUDY OF ATOMICISTIC PROCESS IN SUSPENDED ONE-ATOM THICK GOLD ATOM CHAINS. Varlei Rodrigues1,2, Daniel Ugarte1, Laboratório Nacional de Luz Síntese, Campinas, BRAZIL; Instituto 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 (HREM) study of the atomicistic 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 (0.36-0.38 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 (i.e. substructures) in the quantum conductance response.