Symposium Organizers:
Yue Zhang, University of Science and Technology Beijing, China; Zhiyong Tang, National Center for Nanoscience and Technology, China; Nobuhiro Tsuji, Kyoto University, Japan; Jae-il Jang, Hanyang University, Korea; Kenong Xia, University of Melbourne, Australia; Nathan Mara, University of Minnesota, USA

Monday PM  | August 19, 2019  | Room: Room 309(3rd Floor)
August 19, 2019  | Symposium: K

Chairs:

13:30-14:00 Keynote (1230344)
Effect of Grain Refinement Due to Severe Plastic Deformation on the Brittle-to-Ductile Transition in a Low Carbon Steel
Kenji Higashida, Kyushu University, now at National Institute of Technology, Sasebo College, Japan; Masaki Tanaka, Kyushu University, Japan; Tomotsugu Shimokawa, Kanazawa University, Japan

Brittle fracture of materials is a major concern for the safety in their structural application. Since dislocations in bcc crystals have rather high Peierls potential, plastic behaviors in those crystals highly depend on deformation temperatures. They fail in a brittle manner at low temperatures, but they tend to be ductile with increasing temperature. This is termed brittle-to-ductile transition (BDT). The mechanism behind this phenomenon has been investigated from the view point of the crack-tip shielding interaction. An explanation for the transition is made by the crack-tip shielding due to dislocations, where the internal stress field of the dislocations emitted around the crack-tip shields the crack-tip stress intensity. This means that the stress intensity due to the dislocations generated around the crack-tip relieves the external applied stress concentration around the crack-tip. The stress relieving process due to crack-tip shielding depends on dislocation mobility as well as dislocation source densities. Grain refinement due to severe plastic deformation (SPD) has attracted much attention because materials with ultra-fine grained structures demonstrate remarkable increase not only in yield strength but also in fracture toughness at low temperatures. However, the fundamental mechanism behind the both enhancements of strength and ductility in fine-grained structures has been still ambiguous. In the present study, it was attempted to reveal the mechanism of the enhancement of toughness at low temperatures after the SPD process. The dependence of the BDT on the grain size will be discussed, basing on the crack-tip shielding theory.

14:00-14:25 Invited (1270649)
Accumulated Roll Bonding of High Cr Steel at Elevated Temperatures
Jin-Yoo Suh, ae-Hyeok Shim, Korea Institute of Science and Technology, Korea; Min Seong Kim, Shih-Hoon Choi, Sunchon National University, Korea

Martensitic stainless steels with high carbon and chromium contents are characterized by the combination of superior strength and corrosion resistance. The inherent brittleness of the steel could be alleviated by tempering process but the unavoidably accompanying loss in strength might be unacceptable for some applications. In such a context, there have been some efforts to create a multilayered structure of a martensitic steel and a ductile metal to secure the maximum strength and reasonable ductility simultaneously. However, in this study, we studied the multilayered structure composed of only one martensitic steel roll-bonded at elevated temperature to check the feasibility of the microstructural and mechanical optimization of the multilayered steel plate. Multilayered 0.8C-15Cr martensitic steel plate was produced by utilizing the concept of accumulated roll bonding at elevated temperature up to 3 times to result in 8 layers. Because of the rolling temperature as high as the austenite region, highly accumulated strain together with the repeated phase transformation produced a fine-grained structure. Also, the decarburization of surfaces taking place right before the roll-bonding process resulted in carbon-depleted layers with lower hardness in between the layers of the initial steel composition. To further tailor the microstructure and mechanical behavior, heat treatment at different temperatures, 1073, 1123, 1173, and 1223K was carried out. Microstructure and mechanical behavior characterized by SEM-EBSD and microhardness-tensile test, respectively, will be discussed in the presentation to further present the properties of the high-strength multilayered steel.

14:25-14:50 Invited (1220367)
Strength–Ductility Balance in an Ultrafine-Grained Non-Equiatomic Fe50(CoCrMnNi)50 Medium-Entropy Alloy with a Fully Recrystallized Microstructure
Nokeun Park, Minku Choi, Ibrahim Ondicho, Yeungnam University, Korea; Nobuhiro Tsuji, Kyoto University, Japan

High-entropy alloys (HEAs) contain five or more principal elements in a concentration range of 5~35at.%. Recent studies have revealed CoCrFeMnNi has a single FCC phase when annealed at temperatures above 800°C, but second-phase precipitates are formed...
when it is annealed below this temperature. In this study, we explored the phase stability and mechanical properties of an ultrafine-grained non-equiatomic Fe50(0.3mass% C steel, via a powder metallurgy route consisting of controlled severe plastic deformation and subsequent heat treatments of the compacts resulted in a microstructure with tempered martensite Shell-network and ferrite/tempered martensite Core. Those sintered compacts with appropriate heat treatments showed superior mechanical properties such as high strength with ductility compared to the homogeneous microstructure counterparts. This behavior was essentially related to the ability of the HS to promote the uniform distribution of strain during plastic deformation, leading to improved mechanical properties by avoiding or delaying localized plastic instability. In other words, the characteristic "micro-scale stress concentration" and "macro-scale stress dispersion" by the Shell-network structure are assumed to lead a large work hardening and a constraint of the deformation localization.

This work was supported by JSPS KAKENHI Grant Number JP18H05256 and the JFE 21st Century Foundation. These supports are gratefully acknowledged.

15:10-15:30(1222595)
Effect of Grain Size on Hydrogen Embrittlement of Ultrafine-Grained Iron Processed by High-Pressure Torsion
Hideaki Iwaoka, Shuhei Morimoto, Shoichi Hirosawa, Yokohama National University, Japan

Hydrogen embrittlement is a phenomenon in which hydrogen atoms intrude into a metal, thereby the mechanical properties of the metal is decreased and the metal becomes brittle. It is considered that the mechanism of hydrogen embrittlement is closely related to lattice defects. In the case of dislocations, it is reported that hydrogen atoms promote movement of dislocations and enhance plastic deformation at vicinity of a crack tip by localization of shear strain. On the other hand, as for grain boundaries which also control deformation and fracture of metals, detailed research of effect on hydrogen embrittlement has not been carried out sufficiently. Therefore, we conducted tensile tests while hydrogen charging for an ultrafine-grained iron processed by high-pressure torsion (HPT) and investigated the effect of grain boundaries on hydrogen embrittlement. Pure iron (99.99% purity) discs were annealed at 1173K for 3h and processed by HPT under a pressure of 1.5GPa for 5 turns. The HPT samples were heat-treated at various temperatures from 225 to 350°C to change the grain size. Tensile tests were conducted for these samples while cathodic hydrogen charging in NaOH electrolytic solution. The fracture surface and fracture part of tensile specimen were observed by scanning electron microscope (SEM) and laser microscopy, respectively.
The tensile strength of HPT sample without hydrogen charging is significantly improved by grain refinement and the elongation is decreased. The grain coarsening is occurred by heat treatment after HPT processing, thereby the tensile strength degrades and the elongation recovers. The elongation of these samples is decreased by hydrogen charging, which indicates that hydrogen embrittlement was occurred. The reduction of area obtained from observation of fracture surface also shows degradation of ductility by hydrogen charging. Especially in the HPT samples heat-treated over 275℃, the fracture type changes and the reduction of area is rapidly decreased. It is revealed that hydrogen embrittlement of ultrafine-grained iron is affected by grain size.

15:30-16:10 Tea Break

16:10-16:30(1222575)

Fabrication of Ultrafine Grained Fe-24Ni-0.3C without Severe Plastic Deformation

Wenqi Mao, Si Gao, Kyoto University, Japan; Gao, Myeong-Hoem Park, Yu Bai, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Elements Strategy Initiative for Structural Materials, Japan

It has been reported that metastable austenitic steels having ultrafine grained (UFG) microstructures can be fabricated simply by cold rolling (CR) and subsequent annealing without very high strains, using deformation induced martensitic transformation and its reverse transformation to austenite. However, the efficiency of grain refinement in this method greatly depends on the amount of martensite formed during CR. In a previous study by the present authors, the minimum grain size obtained by CR and subsequent annealing was 1.3μm in an Fe-24Ni-0.3C alloy, due to limited amount of martensite formed by CR. In order to fabricate UFG Fe-24Ni-0.3C having sub-micrometer grain sizes, an attempt has been made in the present study to accelerate the formation of deformation induced martensite during CR by increasing dislocation density in starting material via repetitive martensitic transformation cycles. Repetitive rolling and annealing processes were performed on the Fe-24Ni-0.3C alloy. The starting plate having a thickness of 20mm was firstly subjected to sub-zero quenching in liquid nitrogen to get thermal induced martensite, and then heated up to 600℃ for 30s in a salt bath followed by water quenching to get reversed austenite. Such transformation cycling was repeated for 5 times. Then the plate was cold rolled from 20mm to 10mm in thickness. In order to further reduce the grain size, the plate was subjected to another 5 times of the transformation cycling treatment and then cold rolled to 1 mm in thickness. The cold rolled sheet was finally annealed at 650℃for 30s using a salt bath followed by water quenching. UFG Fe-24Ni-0.3C with a mean austenite grain size of 0.5μm was successfully fabricated by the simple repetition of cold rolling and annealing. According to microstructure observations at different stages of the process, the dislocation density of the material was significantly increased by the repetition of thermal martensitic transformation and reverse transformation. As a result, the deformation induced martensitic transformation during CR was greatly accelerated, which made an important attribute to the formation of UFG austenite. The UFG specimen exhibited an excellent combination of high strength along with good ductility (ultimate tensile strength of 1.1GPa and uniform elongation of 70%).

16:30-16:50(222278)

\hspace{2cm} homogeneous Deformation of Ultra-Fine Grained 304 Stainless Steel during Tensile Test

Si Gao, Yu Bai, Wenqi Mao, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan; Ruixiao Zheng, Beihang University, China; Yanzhong Tian, Northeast University, China; Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan

Austenitic stainless steels (ASSs) exhibit excellent corrosion resistivity and deformability, but usually low yield strength around 300-400MPa due to their FCC structure. Previous studies have found that the ultrafine-grained (UFG) ASSs exhibit good combinations of strength and tensile ductility, as well as good fatigue properties, which make them prospective materials for future industrial applications. On the other hand, unusual tensile behaviors have been sometimes observed in such UFG ASSs. Long stress plateaus after the yield-drop are often observed on tensile stress-strain curves of some UFG ASSs, which seems classical Lüders deformation that typically occurs in carbon steels. However, detailed mechanisms of such an unusual tensile behavior have not yet been made clear. The present study aims to clarify the mechanism of the prominent Lüders-like deformation for the first time, using an austenitic stainless steel having UFG microstructures fabricated by cold rolling and annealing processes.

A commercial 304 stainless steel having ultra-fine grained (UFG) microstructures with the minimum average grain size of 0.3 micro-meter was successfully fabricated by simple cold rolling and subsequent annealing processes. Tensile test at room temperature revealed excellent combinations of tensile strength and uniform elongation in the UFG specimens in comparison with the coarse grained counterpart, which was attributed to the grain refinement strengthening in austenite and the occurrence of deformation induced martensitic transformation during the tensile test. On the other hand, it was noteworthy that the UFG specimens exhibited yield point phenomena with Lüders-band-
In the present study, Cu-10wt% microstructure have been rarely reported. Alloys with fully recrystallized ultrafine-grained (UFG) state. However, the mechanical properties of Cu-grained structures or nano-structures with deformed Cu alloys has been conventionally focused on coarse-grained alloys showing good balance of strength and ductility. On the other hand, some studies reported that known as a trade-off relationship between strength and tensile ductility. Grain refinement is an effective approach to enhance strength of metallic materials. However, tensile ductility usually decreases with increasing strength, which is known as a trade-off relationship between strength and ductility. Therefore, several studies reported that face-centered cubic (FCC) alloys with low stacking fault energy (SFE) showed good balance of strength and ductility. Since the SFE could be controlled by addition of alloying elements, Cu alloys are appropriate to understand the fundamental deformation mechanisms of low-SFE FCC alloys showing good balance of mechanical properties. The deformation mechanism in Cu alloys has been conventionally focused on coarse-grained structures or nano-structures with deformed state. However, the mechanical properties of Cu alloys with fully recrystallized ultrafine-grained (UFG) microstructure have been rarely reported.

In the present study, Cu-10wt%Zn and Cu-30wt%Zn alloys with various average grain sizes ranging from 0.23μm to 12.2μm were fabricated by high pressure torsion (HPT) and subsequent annealing. Mechanical properties were evaluated by a uniaxial tensile test at room temperature. It was found that yield strength and tensile strength increased with decreasing the grain size in both alloys. The Cu-30Zn alloy exhibited higher yield strength and tensile strength than those in the Cu-10Zn alloy with similar grain sizes. In addition, the discontinuous yielding with a yield drop phenomenon, which is a unique behavior of UFG materials, occurred when the grain size was smaller than 0.69 μm in the Cu-10Zn alloy, and the yield drop became sharper with decreasing the grain size. However, discontinuous yielding with a small yield drop was only observed in the Cu-30Zn alloy with the grain size of 0.23μm. In this presentation, the reason for the interesting mechanical behaviors in the Cu-Zn alloys, including difference in the yield drop in the UFG specimens will be discussed.

16:50-17:10(1221474)
Mechanical Properties of Cu-Zn Binary Alloys with Various Grain Sizes
Zhangfan Deng, Yu Bai, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan; Yu Bai, Myeong-heom Park, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan

Grain refinement is an effective approach to enhance strength of metallic materials. However, tensile ductility usually decreases with increasing strength, which is known as a trade-off relationship between strength and ductility. The prominent Lüders deformation was explained in terms of the grain size effect on early plastic instability (necking) of austenite and the enhanced strain hardening by the deformation induced martensitic transformation, which could be revealed by comparing the tensile behavior of the UFG specimen at room temperature and at elevated temperatures.

17:10-17:30 (1220836)
Microstructure Evolution in 99.9% Pure Copper and Cu-0.1Fe-0.03P Deformed by High-Strain Torsion at Various Temperatures
Reza Gholizadeh, Yu Bai, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan; Yu Bai, Akinobu Shibata Nobuhiro Tsuji, Kyoto University, Japan

A pure copper with 99.9wt.% purity and a precipitate-containing copper alloy (Cu-0.1Fe-0.03P) were continuously deformed to high strains (> 6) by torsion. The deformation was carried out at different temperatures ranging from room temperature to 650°C, and the flow stress-strain data were measured simultaneously. Torsion specimens were water-quenched immediately after the deformation, and microstructures under various deformation conditions were investigated by electron back-scattering diffraction (EBSD) method and transmission electron microscopy (TEM). The EBSD measurements were carried out in a field emission scanning electron microscope (JEOL JSM 7100F), and the TEM observation was conducted in a TEM microscope (JEOL JEM 2010F) operated at 200kV. The observation section on the gage part of the round-bar specimen was normal to the radial direction at a radial distance of 0.9R from the center. The results showed that increase in the deformation temperature resulted in a gradual transition in microstructures. Firstly, the deformation at low temperatures led to the formation of the ultrafine lamellar structures (< 1μm) which were caused by so-called grain subdivision mechanism. As the deformation temperature increased, however, the grain morphology changed to relatively equiaxed ones, and simultaneously the grain size increased and reached to several micrometers in the Cu-0.1Fe-0.03P alloy and several ten micrometers in the 99.9% pure copper. The increase in the grain size with increasing the deformation temperature was accompanied with a decrease in the flow stress, indicating that softening mechanisms such as boundary migration and dynamic recrystallization (DRX) became dominant during the deformation at elevated temperatures. Additionally, it was found that fine precipitates in the Cu-0.1Fe-0.03P alloy remarkably hindered grain growth and therefore the flow-stress softening, compared to those in the pure copper. Different mechanisms of the microstructure evolution, e.g., the grain subdivision at low temperatures and the grain growth and DRX at elevated temperatures, in the heavily deformed coppers with and without precipitates will be discussed in details: G.H. Xiao, N.R. Tao, K. Lu, Scripta Materialia, 65 (2011) 119-122.
Symposium Organizers:
Yue Zhang, University of Science and Technology Beijing, China; Zhiyong Tang, National Center for Nanoscience and Technology, China; Nobuhiro Tsuchiya, Kyoto University, Japan; Jae-il Jang, Hanyang University, Korea; Kenong Xia, University of Melbourne, Australia; Nathan Mara, University of Minnesota, USA
Tuesday AM  Room: Room 309(3rd Floor)
August 20, 2019  Symposium: K

Chairs:
8:30-9:00 Keynote (1235143)
Nanostructural Design of Metallic Materials for Superior Properties
Ruslan Valiev, Saint Petersburg State University, Ufa State Aviation Technical University, Russia

Multiple studies in recent years have proved severe plastic deformation (SPD) techniques as a very efficient way to produce nanostructured metals and alloys with significantly improved mechanical and functional properties. At the same time advanced properties affected by several nanostructured parameters, including ultrafine grains and also the defect structure of boundaries in SPD-processed nanomaterials. This report presents the recent results of complex studies of the formation of different grain boundaries (low angle and high angle ones, special and random, equilibrium and non-equilibrium with strain-distorted structure as well as with the presence of grain boundary segregations and precipitations) in nanostructured materials processed using SPD with various regimes and routes. This entails the materials with superior multifunctional properties, i.e. the combination of high mechanical and functional properties (superplasticity, corrosion and radiation resistance, electrical conductivity, etc.) that are induced by grain boundaries. Particular emphasis is laid on the physical nature and the use of multifunctional nanomaterials for their innovative applications in medicine and engineering.

9:00-9:30 Keynote (1233244)
Harmonic Structure Design: Creation of Innovative High Performance Metallic Materials
Kei Ameyama, Ritsumeikan University, Japan

Over many years, fabrication of materials with superior combinations of high strength and high elongation remains a hot issue in material engineering. Ultra-fine grained (UFG) metals have been proved to be attractive structural materials because of superior strength, especially when compared to their coarse-grained (CG) counterparts. However, the downside of homogeneous UFG materials is typically in a low elongation because of the plastic instability in the early stage of deformation. The Harmonic Structure (HS) design can be a candidate materials design, which combines high strength with high ductility at the same time. As opposed to a “Homogeneous-UFG” material, “HS” material has a unique heterogeneous “Three-dimensionally (3D) Gradient Microstructure” wherein the UFG areas form an interconnected three-dimensional network surrounding CG regions, and CG and UFG areas are periodically arranged in all the directions. In the present research, the harmonic structure design has been applied to many kinds of pure metals and alloys via a severe plastic deformation powder metallurgy (SPD-PM) route and subsequent sintering. At a macro-scale, the harmonic structure materials exhibited significantly better combination of strength and ductility, as compared to their homogeneous microstructure counterparts. Since the area under the stress-strain curve is considered as a representation of the tensile toughness of a material, the HS materials also exhibited improved tensile toughness. This behavior was essentially related to the ability of the harmonic structure to promote the uniform distribution of strain during plastic deformation, leading to improved mechanical properties by avoiding or delaying localized plastic instability. In other words, the characteristic “micro-scale stress concentration” and “macro-scale stress dispersion” by the Shell-network structure are assumed to lead a large work hardening and a constraint of the deformation localization. In addition, such a stress concentration in the Shell region leads to high density of dislocation generation and hence it results in a preferential recrystallization in the Shell. And the well-developed Shell structure by the preferential recrystallization provides further improvement of the mechanical properties.

9:30-9:55 Invited (1235849)
Interplay between Thermal Stability and Environmental Tolerance in Nanocrystalline Alloys
Jessica Krogstad, Pralav Shetty, Caelin Muir, Megan Emigh, University of Illinois, Urbana-Champaign, America

Enhanced thermal stability of nanocrystalline metallic alloys is necessary for improving processing routes, preserving desirable mechanical properties that arise from the ultra-fine microstructure, and expanding available applications spaces. Segregation of solute atoms to the grain boundaries in nanocrystalline metals has been explored as a potential route for microstructural stabilization. Decoration of grain boundaries either with distinct precipitates or more subtle concentration...
enhancements not only changes the mobility of the grain boundaries, but also of diffusing species along the grain boundary. As a result, we will show that microstructural thermal stability is also central to promoting the environmental tolerance of these materials. A series of DC magnetron sputtered thin film Ni-based alloys have been subjected to both in situ and ex situ heating and oxidation. Characterization via transmission electron microscopy and atom probe tomography has confirmed that those alloys, wherein the onset of recrystallization and rapid grain growth is shifted moderately (~100°C), exhibit improved passivating oxide quality (density, adherence and continuity). These observations are related to clustering of minor alloying additions in the vicinity of the original grain boundaries, which both reduce grain boundary mobility and also modify the grain boundary diffusion kinetics of desirable oxide forming alloying elements (e.g. Cr and Al).

9:55-10:15(1221699)

Zn single Crystals Deformed by a Single Pass of ECAP
Hiromoto Kitahara, Yuta Matsuo, Yuki Oda, Masayuki Tsushima, Shinji Ando Kumamoto University, Japan

A single pass of equal channel angular pressing (ECAP) at 223K was applied to six kinds of Zn bulk single crystals with different crystal orientations, and the deformation behavior was investigated, such as grain refinement and texture development. Six types of Zn single crystals are prepared in this study. Here, in the early stage of ECAP process, compression in the normal direction (ND) is simply loaded to a sample, as well uniaxial compression. Therefore, the deformation behavior in the early stage was expected, and the six types of Zn single crystals were employed and categorized into three groups. Twinning and basal slips were expected to occur in ND compression in Sample 1 and 2, with basal planes parallel and perpendicular to the theoretical shear plane. Twinning was expected to occur in ND compression in Sample 3 and 4, with c-axes are parallel to ND. In Sample 5 and 6 whose c-axes are parallel to the transverse direction (TD) and extrusion direction (ED), twinning and basal slips never occur in ND comp. However, the results depended on the group. The initial orientation of Sample 3 rotated due to (10-12) twinning above the shear plane in the early stage, and it corresponded to the initial orientation of Sample 6. Sample 3 and 6 fractured in the early stage of ECAP. In samples 1 and 2, twinning and double twinning occurs above the shear plane in the early stage. Also, recrystallization occurred and equiaxed grains were observed above the shear plane. Beneath the shear plane in the final stage, many grain were observed. That is, a large grain with several millimeters can be divided into a number of grains with several tens micron meters by a single pass of ECAP in Samples 1 and 2. On the other hand, in Samples 4 and 5, many (10-12) twins were observed above the shear plane, but recrystallization did not occur. Recrystallization above the shear plane is a key for the grain refinement in Zn. Increase in twin boundaries, due to twinning and activation of basal slips within the twins, are required for occurrences of the recrystallization.

10:15-10:35(1234087)

Nanograin Formation of Second Phase-Strengthened High-Entropy Alloy: Grain Refinement Versus Particle Strengthening
Jeong-Min Park, Dong-Hyun Lee, Jae-il Jang, Hanyang University, Korea; Zhaoping Lu, University of Science and Technology Beijing, China; Jin-Yoo Suh, Korea Institute of Science and Technology, Korea; Megumi Kawasaki, Oregon State University, America; Upadrasta Ramamurty, Nanyang Technological Unoverersity, Singapore

A face-centered cubic (fcc) (CoCrFeNi)94Ti2Al4 high-entropy alloy (HEA) with and without Ni3(Ti,Al) second phase particles was subjected to high-pressure torsion (HPT) to examine the possibility of deriving the benefits of particle and grain boundary (GB) strengthening simultaneously. A marked reduction in the average grain size, from ~57µm before HPT to nm-scale, upon HPT was noted. This, in turn, increases the hardness substantially. Transmission electron microscopy analysis with elemental mapping of the alloy subjected to HPT reveal that dissolution of a large amount of the second phase particles and structural transformation (from L12 to disordered fcc) of the remnant ones occur concomitantly with the grain refinement. This precludes the accrual of benefits of simultaneous strengthening of the examined HEA through grain refinement and particle strengthening. Strain rate sensitivity measurements indicate that the former mechanism not only enhances the strength, but may also increase the HEA's ability for accommodating plastic deformation. This appears to occur, as indicated by the estimates of the activation volume for deformation, due to a transition from lattice distortion controlled processes in the coarse grained HEA to grain boundary controlled ones in the nanocrystalline HEA. These observations provide key directions for the design of strong and ductile HEAs.*This work was supported by the National Research Foundation of Korea (NRF) grants funded by the Ministry of Science and ICT (No. 2015R1A5A1037627 and No. 2017R1A2B4012255).

10:30-10:45 Tea Break
11:45-11:10 Invited (1235997)

Utilizing Solute Solution Drag of Nb in Ni for Fabricating Multilayered Structures in a Sheet Metal
Zakaria Quadir, Curtin University, Australia

Solute solution strengthening has been a subject in many metallurgical phenomena. The required level of concentration for creating an effective drag has been a key question for many years. In this investigation, it was found that the solute drag effect of Nb atoms in Ni slows down the thermal restoration process. This was reflected from the changes in deformation structures during annealing. This phenomenon was observed in a Ni sheet containing 0.5 atomic percentage of Nb in the form of a homogeneous solid solution. The sheet was roll bonded with commercial purity of Ni to a high strain by a severe plastic deformation (SPD) process known as accumulative roll bonding (ARB). ARB involves successive bonding of sheets for many iterations while the thickness of the sheet remains constant. This investigation shows the change in crystallographic textures and microstructures with ARB processing and subsequent annealing heat treatments. This investigation incorporates a range of advanced characterization techniques that include scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), transmission Kikuchi diffraction (TKD), transmission electron microscopy (TEM) and atom probe tomography (ATP). Discoveries are made on the maximum level of refinements by rolling, and concentration of solute for preventing coarsening in ARB formed microstructures.

11:10-11:35 Invited (1222729)

Analysis on Temperature- and Rate-Dependent Nanomechanical behavior of Nanocrystalline High-Entropy Alloys Through High-Temperature Nanoindentation
In-Chul Choi, Kumoh National Institute of Technology, Korea; Dong-Hyun Lee, Max Planck Institute for Iron Research, Korea; Jae-il Jang, Hanyang University, Korea; Ruth Schwaiger, Karlsruhe Institute of Technology, Germany

High-entropy alloys (HEAs) which are multicomponent alloys containing five or more elements in (near-)equal atomic percent, have attracted significant research interest over the past decade due to their simple microstructures with interesting mechanical properties. Recently, it has been reported that the mechanical performance of HEAs can be further enhanced by reducing the grain size to the nanocrystalline (nc) regime. One of the ways to obtain a nc HEA is through the high-pressure torsion (HPT) process. The mechanical behavior of such nc HEAs is also reasonably well understood; this was mostly accomplished by employing small-volume mechanical testing techniques such as a nanoindentation and a micro-pillar compression due to the due to limited volume of material available. However, most of these studies are confined to room temperature (RT). In this study, high-temperature nanoindentation experiments in the temperature (T) range of 298 and 573K were performed to determine the hardness, strain-rate sensitivity (m), activation volume (V*) and activation energy (Q) for the plastic flow in a nc CoCrFeMnNi HEA, which was synthesized using HPT process. The operating deformation mechanisms are interpreted on the basis the data obtained. First of all, the value of m remains almost constant (~0.015) until 423K and increases from 473K onwards with T. In contrast, V* a peak at 423K. Although these results do indicate a possible change in the deformation mechanism, they are insufficient to confirm a mechanistic transition whereas, Q, being a thermodynamic parameter for the thermally-activated plastic flow can provide such confirmation. Especially, the activation energy of the nc CoCrFeMnNi HEA suddenly increases from ~0.5 to ~1.8eV at a T of 473K. It was revealed that this increase is not by the microstructural instability but by the change in deformation mechanism, i.e., enhanced plasticity due to GB diffusion at high T.

11:45-12:10 Invited (1234977)

Multiscale Simulation Based on Macroscopic Deformation Model Predicted by Microstructure Information of Ultrafine-Grained Metals
Yoshiteru Aoyagi, Atsushi Sagara, Tohoku University, Japan; Chiihiro Watanabe, Kanazawa University, Japan; Masakazu Kobayashi, Yoshikazu Todaka, Hiromi Miuragnes, Toyohashi University of Technology, Japan

A uniaxial tensile test generally determines mechanical properties of materials by neglecting a directional dependency of the materials. Advanced materials such as ultrafine-grained metals (UFGMs) having peculiar mechanical properties that are not found in conventional materials have attracted a great deal of attention due to an increase in industrial demand for materials. It is reported that UFGMs improve the tensile strength that is about four times stronger than the usual materials. UFGMs produced by severe plastic deformation such as accumulative roll-bonding (ARB) processing show mechanical anisotropy depending on strong rolling texture. Therefore, it is difficult to determine the mechanical properties of UFGMs by an only uniaxial tensile test. It is necessary to take the information of microstructure into account for accurate comprehension of the mechanical properties of advanced materials. In this study, yield functions for severely rolled pure aluminum, A1050, was constructed in order to investigate the effects of microstructures on mechanical properties.
The yield function is estimated from the contours of plastic work obtained by uniaxial and biaxial tensile tests with yield function of Yld-2000-2D. Moreover, the yield function is predicted by crystal plasticity analyses based on the microstructure observed using EBSD method and the curve fitting with a stress-strain curve obtained by a uniaxial tensile test. Comparison of both results verifies the validity of the numerical prediction of the yield function. The practical CAE system based on microstructures of materials is constructed by introducing the yield function predicted by the crystal plasticity simulation into a general-purpose CAE software.

12:10-12:30(1234279)

In Situ Atomistic Observation of Disconnection-Mediated grain Boundary Migration
Qi Zhu, Guang Cao, Jiangwei Wang, Zhejiang University, China

Shear-coupled grain boundary (GB) migration is of general significance in the deformation of nanocrystalline and polycrystalline materials, but comprehensive understanding of the migration mechanism at the atomic scale remains largely lacking. Here, we systematically investigate the atomistic migration of $\Sigma 11(113)$ coherent GBs in gold bicrystals using a state-of-art in situ nanofabrication and shear testing technique inside aberration-corrected transmission electron microscope, combined with molecular dynamic simulations. We show that shear-coupled GB migration can be realized by the consecutive nucleation and lateral motion of GB disconnections, where both single-layer ($b=1/22<471>$) and double-layer ($b=1/22<332>$) disconnections have important contributions to the GB migration through their frequent (energy driven) composition and (GB kinetics induced) decomposition. Such disconnection-mediated GB migration is also confirmed in other high angle GB structures in our experiments, which should represent a general deformation phenomenon in the GB dominated plasticity; in the meantime, the disconnection-mediated GB migration mechanism is weakly influenced by the interaction between GBs and lattice defects, including dislocations and stacking faults. We further reveal the consecutive nucleation of GB disconnections from the triple junction, which is a prevalent structure in engineering polycrystalline and nanocrystalline materials. Moreover, we demonstrate that the disconnection-mediated GB migration is fully reversible in shear loading cycles without the emission of lattice defects, which enhances the deformability of nanostructured materials, inspiring the tuning of mechanical properties through proper design of GB structures. This talk is based on our recent publication in Nature communications (Nature Communications, 2019, 10, 156)
Symposium Organizers:
Yue Zhang, University of Science and Technology Beijing, China; Zhiyong Tang, National Center for Nanoscience and Technology, China; Nobuhiro Tsuji, Kyoto University, Japan; Jae-il Jang, Hanyang University, Korea; Kenong Xia, University of Melbourne, Australia; Nathan Mara, University of Minnesota, USA

Tuesday PM Room: Room 309(3rd Floor) August 20, 2019 Symposium: K

Chairs:

13:30-14:00 Keynote (1234924)
Probing Electron Transport in Atomically Thin Semiconducting Transition Metal Dichalcogenides
Ning Wang, Hong Kong University of Science and Technology, Hong Kong, China

Atomically thin semiconducting transition metal dichalcogenides (TMDCs) such as MoS\textsubscript{2}, MoSe\textsubscript{2}, WS\textsubscript{2}, and WSe\textsubscript{2} form a family of layered two-dimensional materials exhibiting novel electronic and optical properties. Probing the quantum transport in these TMDCs has been a longstanding challenge due to the low carrier mobility and the large contact resistance in their field-effect devices prepared by the exfoliation method. In this talk, I demonstrate our recent experimental study on quantum transport of few-layer MoS\textsubscript{2} and WSe\textsubscript{2}, and their unconventional Landau levels (LLs) with strong interaction effects. We fabricate high-quality n-type MoS\textsubscript{2} and p-type WSe\textsubscript{2} devices by encapsulating these TMDCs in ultra-clean hexagonal boron nitride sheets which effectively eliminate impurity scattering and provide clean interfaces for making high-quality low-temperature ohmic contacts to these semiconducting TMDCs. Few-layer MoS\textsubscript{2} and WSe\textsubscript{2} field-effect devices with mobilities up to 30,000 cm\textsuperscript{2}/V s have been achieved at cryogenic temperatures. We observe interesting quantum Hall (QH) phenomena involving the Q valley, $\Gamma$ valley and K valley, such as the Q valley Zeeman effect in all odd-layer MoS\textsubscript{2} devices and the spin Zeeman effect in all even-layer MoS\textsubscript{2} devices and highly density-dependent QH states of $\Gamma$ valley holes in WSe\textsubscript{2}. The predominant sequences of the QH states of $\Gamma$ valley holes in few-layer WSe\textsubscript{2} alternate between odd- and even-integers with reducing the density. By tilting the magnetic field to induce Landau level crossings, we show that the strong Coulomb interaction enhances the ratio of Zeeman-to-cyclotron energy, giving rise to the even-odd alternation of the predominant sequences.

For n-type MoS\textsubscript{2}, we have studied the valley-resolved SdH oscillations relevant to the spin-valley locked massive Dirac electron LLs. With decreasing the carrier density in the conductance band (K valley), we observe LL crossings induced valley ferrimagnet-to-ferromagnet transitions and the enhancement of the valley Zeeman effect by Coulomb interactions. In n-type monolayer and trilayer MoS\textsubscript{2}, we first detect the intrinsic valley Hall transport without any extrinsic symmetry breaking through measuring the nonlocal resistance that scales cubically with the local resistance. Such a phenomenon survives at room temperature with a valley diffusion length at the micron scale. We believe that the large intrinsic bandgap in MoS\textsubscript{2} contributes to maintaining a large amplitude of the Berry curvature, allowing to observe the valley Hall effects even at room temperature in monolayer MoS\textsubscript{2}.

14:00-14:25 Invited (1225047)
Direct Observation of the Dislocation Interaction with Grain Boundary in Ultrafine-Grained IF Steels by in-Situ TEM Technique
Seiichiro Ii, Hongxing Li, Takahito Ohmura, National Institute for Materials Science, Japan; Takahito Ohmura, Kyoto University / Kyushu University, Japan; Nobuhiro Tsuji, Kyoto University, Japan

Mechanical properties of polycrystalline materials are often governed by their grain boundaries, which is well-known as Hall-Petch relation. In the Hall-Petch relation, the yield strength is linearly related to the inverse square root of the mean grain size and the slope of the relation is termed $k$, based on the concept of the interaction between dislocations and grain boundaries. For an improvement of the mechanical properties in the polycrystalline materials, the grain refinement is one of the effective ways. Ultrafine-Grained (UFG) materials, which are consisted of the fine grains of less than 1\textmu m, exhibit high strength. Moreover, $k$ value also becomes larger than that of coarse-grained one (>1\textmu m). Even in the UFG metals, many researchers have discussed the plastic deformation mechanisms from the experimental and computational techniques. On the other hand, in-situ transmission electron microscopy (TEM) is known as one of the powerful techniques to directly clarify the microstructure change under the external field. Especially, recent in-situ straining TEM holder can obtain the mechanical data as well as microstructure change simultaneously. In this study, we investigated the dislocation interaction with grain boundary in the UFG IF steel fabricated by an accumulative roll-bonding process by the in-situ straining TEM technique. Micropillar specimens for in-situ observations were prepared by focused ion beam (FIB). The in-situ straining TEM was performed using an indentation TEM holder under compression with the displacement-controlled mode. Microstructure change during compression was
recorded as a video using a CCD camera at 30 fps. We could directly observe that pre-existed dislocations in grain interior were moved during loading and those in the specific grain are absorbed into the grain boundary without piling up.

14:25-14:50(Invited)

Atomistic Structural Characteristics of Amorphous B-Si-C-N based Coatings at Temperatures >1000°C

Jiechao Jiang, Efstathios I. University of Texas at Arlington, USA; J. Vlček, Physics Department, University of West Bohemia, Czech Republic

We present, in this talk, atomistic structure of B-Si-C-N and Hf-B-Si-C-N coatings at temperatures above 1000°C and the high temperatures oxidation mechanism. The coatings were deposited by magnetron sputtering and are hard with an amorphous structure and exhibit superior high temperature oxidation resistance up to 1100°C without any atomic structure changes. Annealing in air at temperatures from 1100°C to 1500°C transforms the amorphous coating structure into a two-layered structure comprising of an oxidized layer on the top followed by a non-oxidized bottom layer. The top oxidized layer possesses either a pure amorphous SiOx, or nanocomposite of HfO2 nanoparticles dispersed in amorphous SiO2 matrix structure. The thickness of the oxide layer varies with the annealing temperature and coating composition. The non-oxidized bottom layer structure varies from amorphous to nanocomposite of HfB2 and HfN nanoparticles separated by h-Si3N4 and h-BN boundaries depending on the annealing temperature. The oxide/bottom layer interfaces possess high population of either fine BN nanoparticles or fine HfO2 nanoparticles surrounded by SiO2 boundaries. The high-temperature oxidation resistance evolves either nanocomposite of BN nanoparticles and SiOx, or HfO2 nanoparticles within a dense SiOx-based matrix and quartz SiO2 in front of the bottom layer acting as a barrier for heat and O diffusion. This work was supported by the U.S. National Science Foundation under Award NSF/CMMI DMREF-1335502.

14:50-15:10(1228092)

Designing Heterogeneous Ultrafine-Grained Metals and High-Entropy Alloys to Enable High Strength and High Ductility Simultaneously

Evan Ma, Johns Hopkins University, America

In a previous review, Materials Today 20 (2017) 323-331, we presented our perspective that heterogeneities intentionally introduced into a metal, such as a grain size distribution/gradient, or hierarchical defect structures, promote strain hardening and hence uniform tensile ductility. The result is a strength-ductility combination noticeably better than that known for conventional cold-worked or ultrafine-grained counterpart.

In this talk, we demonstrate that the properties achieved above still remain a trade-off between strength and ductility, albeit at a level better than that normally seen in conventional metals. This is true even for the most recent reports in 2018. In other words, there is plenty of room in the strength-ductility space yet to be reached. Very recent attempts, including in situ nanostructuring and hierarchical microstructures in multi-component multiphase alloys, have opened new opportunities that are not previously possible in those simple metals above.

We will discuss FCC high-entropy alloys and BCC high-entropy alloys to review what have been found over the past several years. These HEAs are strengthened via "multiple principal elements", together with easy storage of defects during tensile deformation due to their low stacking fault energy. The result is an added propensity for heterogeneous microstructure that promote strain gradient hardening. The most recent successes typically have a combination of gigapascal yield strength with >25% tensile strain, see, e.g., M. Yang et al., PNAS 2018, and Z. Lu et al., Nature in October 2018. The latter example used oxygen-(Ti,Zr) complexes (ordered local entities and not yet second phase) as heterogeneities to help increase the ductility of the base BCC high-entropy alloy. Such chemical heterogeneities go beyond the structural heterogeneities in single-element metals. An exciting case, reported last month in Science by C.T. Liu’s group, used a carefully crafted high-entropy intermetallic as the second phase. They achieved 50% uniform elongation, making the complex alloy as ductile as any single-element un-strengthened metal, at a yield strength above 1.5GPa (an order of magnitude higher than any single-element, un-strengthened metal). This extends the strength-ductility range to unprecedented territory. Note that previously, strength-ductility trade-off was claimed to have been "overcome" only by comparing with an already-strengthened low-ductility metal/alloy, or with alloys that have yield strength well below 1GPa.

Multi-component alloys also allowed the exploitation of in situ phase transformations, similar to the TRIP effect in steels. The Nature (2016) paper by Z. Li and D. Raabe for FCC HEAs, and the Advanced Mater. (2018) paper by Y. Wu et al. for BCC HEAs, followed this route. They have made use of martensitic transformation during the tensile deformation to elevate strain hardening, sustaining uniform elongation while strengthening the high-entropy alloy. This TRIP route towards strength-ductility synergy is interesting, as it adds a new twist to the known TRIP steels and takes advantage of the metastable nature of high-entropy alloys.

We advocate that multi-component, multiphase alloys are more effective in pushing the strength-ductility envelope, and constitute a fertile playground
to be explored, especially with the advent of complex concentrated "high-entropy" alloys, a field that is emerging and attracting widespread attention recently.

15:10-15:30 (1416060)

Microstructural Characteristics and Dynamic Behaviour of Dislocations in Metal Nanowire under Tensile Process
ZhaoYang Hou, Xiao Qixin, Liu Rangsu, Chang'an University, China; Rangsu, Hunan University, China

The dislocation is an important microstructural configuration during the deformation process of nanocrystalline metals. The dynamic behaviour of dislocations play a crucial role in the mechanical properties and deformation mechanism of nanocrystalline metals. The dislocation atoms are usually regarded as disordered atoms, but its detailed microstructural characteristics are not completely understood. In this work, the microstructure characteristics and dynamic behaviour of dislocations in Au nanowire under the tensile process are investigated by molecular dynamic simulation. It is found the plastic deformation mechanism in Au nanowire is the emission, propagation and annihilation of Shockley partial dislocations, which is agree with the experimental results obtained by in situ high-resolution transmission electron microscopy. The microstructural configurations of the dislocation atoms are analysed by means of the microstructural analysis method--cluster-type index method (CTIM) proposed by us. It is found the dominating microstructural configurations of the dislocation atoms are the defective FCC clusters represented by the CTIM indexes (12, 2/1311, 1/1411, 9/1421), (11, 4/1311, 7/1421), (12, 8/1421, 2/1431, 2/1541), (13, 8/1421, 1/1441, 4/1541), and the defective BCC clusters represented by the CTIM indexes (14, 4/1441, 4/1551, 6/1661). During the propagation process of Shockley partial dislocations, a stacking fault with HCP atoms follows the dislocation. There are two different paths during the FCC→HCP transformation in term of tracing the evolutions of cluster structures.

15:30-16:10 Tea Break

16:10-16:35 Invited (1235391)

Investigating Deformation in Nanocrystalline Materials Using in-Situ Transmission Kikuchi Diffraction
Glenn Sneddon, Vijay Bhatia, Julie Cairney, University of Sydney, Australia; Patrick Trimby, Oxford Instruments Nanoanalysis, England

Observations of strained nanocrystalline materials have shown that discontinuous grain growth can occur at room temperature with as little as 2% strain. Understanding such changes in nanocrystalline material properties at room temperature is vitally important and has, until now, required detailed TEM observations of the resulting microstructure and inferences regarding the processes involved. Transmission Kikuchi diffraction (TKD) in the scanning electron microscope (SEM) has advantages in resolution over conventional electron backscatter diffraction that have resulted in a wide range of applications of TKD across the materials and earth sciences. However, to date TKD has not been applied to the study of dynamic processes, utilizing in-situ deformation. Here we present results from a prototype in-situ tensile deformation stage that has been custom modified for use with TKD, enabling the investigation of discontinuous processes on the nanoscale, such as grain boundary sliding, twinning and stress-assisted grain growth. The experiments were carried out using a Zeiss Ultra Plus field emission SEM equipped with an Oxford Instruments AZtec EBSD and TKD system, acquiring TKD data at discrete load steps for each sample. A variety of materials were analysed, including nanocrystalline copper films, surface treated stainless steels and steels undergoing stress-induced phase transformation. We believe that this in-situ TKD technique can become an effective tool for understanding the complex nature of discontinuous stress-assisted grain growth, allowing us to identify the exact nature of the grains and boundaries that are most affected by this mechanism.

16:35-16:55(1233786)

Significantly Enhanced Wear Resistance of an Ultra-fine Grained Edium Entropy Alloy CrFeNi at Elevated Temperatures
Fuzeng Ren, Dingshan Liang, Cancan Zhao, Weiwei Zhu, Pengbo Wei, Feilong Jiang, Ywen Zhang, Southern University of Science and Technology, China

Metallic alloys for mechanical components involving sliding contacts in relative motion, particularly at the elevated temperatures, require not only good combination of strength and ductility, but also high sliding wear resistance. Thus, the design of novel metallic alloys with exceptional damage-tolerance for harsh environment is in great demand. In recent few years, new alloy design strategy with multi-principal elements have been developed, referring to as high/medium entropy alloys (HEAs or MEAs). Herein, we report a novel cost-effective equiatomic medium entropy alloy (MEA) CrFeNi consisting of an ultrafine-grained (UFG) face-centered cubic (fcc) matrix with a high density of nanoscale body-centered cubic (bcc) Cr-rich precipitates. Such a hierarchical structure offers an excellent combination of high tensile strength of 826 MPa and elongation of 26%. Remarkably, the alloy
The strong desire for materials with high strength and good ductility in industrial applications is driving the development of new concepts in alloy design. In recent years, a new type of equiatomic or near equiatomic multicomponent alloys named high-entropy alloys (HEAs) intrigue a new strategy in developing alloys. Among the HEA systems, the HEAs with face-centered cubic (fcc) structure were widely studied because of the attractive properties, such as dynamic properties, fracture toughness and corrosion resistance. However, the yield strength of HEAs with FCC structure is insufficiently strong. In order to increase the yield strength of the HEAs with FCC structure, many efforts were conducted, such as grain refinement, precipitation and heterogeneous structure. Here, we introduce a new strategy to fabricate FCC HEA with superior strength and ductility. Bulk CoCrFeMnNi HEAs were prepared by magnetic levitation melting technique. The cast got was firstly hot forged into rods and further cold rolled into sheets 1 mm thick with a nominal reduction in thickness of 95%. Ultrafine-grained (UFG) specimens with fully recrystallized microstructures were fabricated by cold rolling and subsequent annealing treatments. This novel UFG HEA is characterized with fine grains, high-fraction of high-angle grain boundaries and low dislocation density. Tensile properties were tested at room and cryogenic temperatures. Transmission electron microscope (TEM) and electron backsattered diffraction (EBSD) were used to characterize the microstructures. With decreasing the temperature, the yield strength, uniform elongation and strain-hardening capability were enhanced significantly. The enhanced yield strength is related strongly to the small grain size, and is originated from thermal and athermal contributions. The ductility and strain-hardening rate are enhanced by inhibiting the dislocation recovery and facilitating the deformation twins by lowering the temperature. Synchronous increase in strength and ductility is achieved in the present UFG HEA by decreasing temperature, which provide a strategy to design HEAs with adaptive mechanical properties.

**Formation of Nanostructures in a γ' Precipitate Strengthened Ni-based Superalloy Processed by Surface Mechanical Rolling Treatment**

**Yong Zhang, Hao Wu, Shengyun Yuan, Nanjing University of Science and Technology, China**

Nanostructured materials have been widely investigated over the past twenty years due to its high strength, superior fatigue properties and so on. However, the fabrication of nanostructured Nickel-based superalloy has been few investigated before. In this study, a Nickel-based superalloy with high density of L12 structured γ' precipitates was processed by surface mechanical rolling treatment (SMRT). A gradient microstructure was formed along the depth from the treated surface, including nano-grained structure, nano-twins and stacking faults (SFs). The formation mechanisms of nanostructure in the topmost surface (<100mm) were systematically investigated by means of transmission electron microscopy (TEM), Super-X energy dispersive spectroscopy (EDS) and three-dimensional atom probe technology (3DAPT). TEM observations on the topmost surface indicate that nano-grained structure has the face centered cubic (fcc) structure with an average size of ~15nm. However, the EDS scan perpendicular to the grain boundaries demonstrate that neighboring nano-grains exhibits similar elements partitioning tendency to the γ/γ′ phase structure. The Ni, Ti and Al atoms tend to partition into one grain, while Co/Cr atoms into the neighboring grain, suggesting that the composition distribution in the nano-grains inherits from γ′ phase structure. TEM observations indicate that the deformation microstructure in the subsurface of SMRT processed sample are characterized by high density of nano-twins and stacking faults on two (111) planes. This suggested that the nanostructure on the topmost surface can be ascribed to the intersection of high density of nano-twins. The EDS analysis also indicate that the elemental partitioning tendency in the neighboring twin/matrix is quite similar to those in neighboring nano-grains, i.e., the neighboring twin/matrix exhibits enrichment of Ni, Ti and Al atoms and Co/Cr atoms alternatively. Therefore, the formation of the fcc structured nano-grains in the topmost surface can be attributed to shearing deformation exerted by the deformation twins on the γ′ phase structure, which leads to the fragmentation of γ′ phase while preserving the composition.
Symposium Organizers:
Yue Zhang, University of Science and Technology Beijing, China; Zhiyong Tang, National Center for Nanoscience and Technology, China; Nobuhiro Tsuji, Kyoto University, Japan; Jae-il Jang, Hanyang University, Korea; Kenong Xia, University of Melbourne, Australia; Nathan Mara, University of Minnesota, USA

Wednesday AM Room: 309(3rd Floor)
August 21, 2019 Symposium: K

Chairs:
Ye Pan, Southeast University, China
Lin Liu, Huazhong University of Science and Technology, China

8:30-9:00 Keynote (1232021)
Phase Engineering of Novel Aanomaterials
Hua Zhang, City University of Hong Kong, Hong Kong, China; Nanyang Technological University, Singapore

In this talk, I will summarize the recent research on the phase engineering of nanomaterials in my group. It includes the first-time synthesis of hexagonal-close packed (hcp) Au nanosheets (AuSSs) on graphene oxide (GO), the first-time synthesis of 4H hexagonal phase Au nanoribbons (NRBs), the synthesis of crystal-phase heterostructured 4H/fcc Au nanorods, the epitaxial growth of metal nanostructures with novel crystal phases on the aforementioned Au nanostructures, and the synthesis of amorphous crystal lattice hetero-phase Pd and PdCu nanosheets. In addition, the first-time synthesis of 1T-MoS₂ and 1T-MoS₆ nanocrystals have been achieved. Moreover, the phase transformation of transition metal dichalcogenide (TMD) nanomaterials during our developed electrochemical Li-intercalation method will also be introduced. Interestingly, the lithiation-induced amorphization of Pd₃P₂S₈ is also achieved. Currently, my group focuses on the (crystal) phase-based nanomaterial syntheses, property studies, and applications in catalysis, surface enhanced Raman scattering, waveguide, photothermal therapy, chemical and biosensing, clean energy etc., which we believe are quite unique and very important not only in fundamental studies, but also in practical applications. Importantly, the concepts of crystal-phase heterostructures and hetero-phase nanomaterials are proposed. We believe the phase engineering of novel materials will become one of the hottest research topics in materials science and chemistry in the near future.

9:00-9:30 Keynote (1230709)
Peculiar Electron Phonon Coupling in Van Der Waals Two-Dimensional Heterostructures
Pingheng Tan, Institute of Semiconductors, Chinese Academy of Sciences, China

Two-dimensional materials (2DMs) have been under intensive investigation. The rapid progress of research on graphene is now stimulating the exploration of different types of 2DMs, such as monolayer and multilayer 2DMs, 2D alloy, 1D nanoribbon, 0D nanocrystal and van der Waals heterostructures (vdWHs). Raman spectroscopy can reveal information on the crystal structure, electronic structure, lattice vibrations and flake thickness of 2DMs, and can be used to probe the strain, stability, charger transfer, stoichiometry, and stacking orders of 2DMs. In this talk, we will discuss that Raman spectroscopy of vdWHs can be modulated by the excitonic resonance effects, which demonstrate peculiar electron phonon coupling. Due to this peculiar electron phonon coupling, the intensity of some Raman modes is significantly enhanced when the excitation energy matches the energy of excitonic absorption peaks. These excitonic resonance effects in Raman spectra of vdWHs make many interesting results be revealed.

9:30-10:00 Keynote (1235377)
Nanostructured Semiconductor Photoelectrodes for Solar Energy Conversion
Lianzhou Wang, The University of Queensland, Australia

Semiconducting materials hold the key for efficient photocatalytic and photoelectrochemical water splitting. In this talk, we will give a brief overview of our recent progresses in designing semiconductor metal oxides materials for photoelectrochemical energy conversion including photocatalytic solar fuel generation. In more details, we have been focusing the following a few aspects: i) band-gap engineering of layered semiconductor compounds including layered titanate, tantalate and niobate-based metal oxide compounds for visible light photocatalysis, and ii) facet-controlled TiO₂, Fe₂O₃, WO₃, BiVO₄ as building blocks for new photoelectrode design, and iii) the combination of a high performance photoelectrode BiVO₄ with perovskite solar cells can lead to unassisted solar driven water splitting process with unassisted solar-to-hydrogen conversion efficiency of >6.5% (Fig.1); The resultant material systems exhibited efficient photocatalytic performance and improved power conversion efficiency in solar cells with record high efficiency, which underpin important solar-energy conversion applications including solar fuel and solar electricity generation.
Asymmetric Cyclic Response of Pretensioned Cu with Highly Oriented Nanoscale Twins
Qingsong Pan, Qiu Hong Lu, Lei Lu, Institute of Metal Research, Chinese Academy of Sciences, China; Hao Fei Zhou, Zhejiang University, China; Huajian Gao, Hao Fei Zhou, Brown University, USA

Pre-deformation of metallic materials generally induces high density dislocations and irreversible microstructural change, thus strongly influencing their subsequent cyclic behavior and causing obvious cyclic softening. In this study, to deepen the understanding of cyclic deformation in nanotwinned (NT) structures, small levels of pre-tension strains were applied on NT-Cu, followed by strain-controlled symmetric tension-compression cyclic tests. Distinct from the symmetric cyclic response of as-deposited NT-Cu, the magnitude of the maximum stress in tension is much larger than that of the minimum stress in compression, indicating that the cyclic response of pre-tensioned NT-Cu is highly asymmetric. The degree of its cyclic asymmetry gradually decays as the number of cycles or the plastic strain amplitude is increased. The pre-tensioned NT-Cu recovers to its symmetric cyclic response after cyclic deformation at sufficiently large plastic strain amplitude, analogous to that detected in as-deposited NT counterparts. Molecular dynamics simulations and microstructural observations revealed that the observed asymmetric cyclic response is mainly related to the activation and movement of threading dislocations with extended misfit dislocation tails lying on the twin boundaries (TBs) during pre-tension deformation. During cyclic deformation, threading dislocations in adjacent twin interiors tend to link their long tails with one another to form correlated necklace dislocations (CNDs) with a symmetric structure. The CNDs move back-and-forth along the twin boundaries without directional slip resistance, contributing to the transition from asymmetric to symmetric cyclic response of NT-Cu.

10:00-10:20 (1235085)
Asymmetric Cyclic Response of Pretensioned Cu with Highly Oriented Nanoscale Twins
Qingsong Pan, Qiu Hong Lu, Lei Lu, Institute of Metal Research, Chinese Academy of Sciences, China; Hao Fei Zhou, Zhejiang University, China; Huajian Gao, Hao Fei Zhou, Brown University, USA

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10:30-10:45 Tea Break

10:45-11:10 Invited (1235822)
Increasing Photoluminescence Quantum Yield by Nanophotonic Design of Quantum-Confined Halide Perovskite Nanowire Arrays
Zhiyong Fan, Daquan Zhang, Yuanjing Lin, Leilei Gu, Hong Kong University of Science and Technology, Hong Kong, China

Metal halide perovskite materials have demonstrated many appealing properties that can be harness for high performance optoelectronics. Among them, high photoluminescence quantum yield (PLQY) is required to reach optimal performance in solar cells, lasers and light-emitting diodes (LEDs). Typically, PLQY can be increased by improving the material quality to reduce the non-radiative recombination rate. It is in principle equally effective to improve the optical design by nanostructuring a material to increase light out-coupling efficiency and introduce quantum confinement, both of which can increase the radiative recombination rate. However, increased surface recombination typically minimizes nanostructure gains in PLQY. Here a template guided vapor phase growth of perovskite nanowire (NW) arrays with unprecedented control of NW diameter from the bulk (250nm) to the quantum-confined regime (5.7nm) is demonstrated, while simultaneously providing a low surface recombination velocity of 18cm s\(^{-1}\). This enables an obvious PLQY enhancement from 0.33% up to 42.6% for MAPbI\(_3\) quantum wires, and up to ~90% for MAPbBr\(_3\) quantum wires, exclusively using nanophotonic design, which will be promising for LEDs applications with high external quantum efficiency (EQE). The simple extension of this technique to a wide variety of semiconductors and the ultra-high density of vertical QWs may also provide interesting opportunities in quantum transport, electronics and memory devices in the future.

11:00-11:30 (1308397)
Atomically Thin Homogeneous MoS\(_2\) Logic Inverter via Solution-Processable Defect Engineering
Li Gao, University of Science and Technology, China

Ultrathin molybdenum disulfide (MoS\(_2\)) presents ideal properties for building next-generation atomically thin circuitry because of their atomic-level thickness, superior theoretical mobility, moderate band gap, and stable structure. However, it is difficult to construct a logic unit of monolayer MoS\(_2\) using traditional silicon-based doping schemes, such as atomic substitution and ion implantation, to cause lattice disruption and doping instability. Nevertheless, defects, which customarily act as negative components, may play a stable and positive role in modulating the electronic structure of MoS\(_2\) to build atomically thin logic devices. Here, a facile, feasible and novel electronic structure modulation strategy of a defect is proposed to construct an in-plane monolayer MoS\(_2\) logic inverter. By utilizing the energy-matched electron induction of the solution process, numerous pure monosulfur vacancies (Vmonos) with lattice stability are introduced to modulate the electronic structure of monolayer MoS\(_2\) via a trapping effect. The resulting modulation effectively reduces the electronic concentration of MoS\(_2\) by one hundred times and improves the work function by approximately 100 meV. Under modulation of Vmonos, an atomically thin homogenous monolayer MoS2 logic inverter with a voltage gain of 0.34 is successfully constructed. This research highlights a new and practical design route for...
Helping to reconstruct and restore tactile perception motivates the continuous development of functional materials, sensing structures, and manufacturing strategies. Functional materials including micro/nanoparticles, one- or two-dimensional materials, and their composites with the capabilities that provide the electromechanical signal conversion of exteroceptive stimulus play a considerable role in the reconstruction and restoration of tactile perception, and thus enable the dexterous manipulation of objects and the discerning interaction of apparatus. Efforts of sensing devices are mainly based on homo-contact microstructure (HoCM) that utilizes the microstructural contact between the same materials or within a single material as the sensing contact for the scientific and engineering researches of artificial tactile perception. However, the sensing mechanism of hetero-contact microstructure (HeCM) that bases on the microstructural contact between different types of materials is seldom reported where the influence on device’s sensitivity and sensing range has yet to be studied.

Here we propose the HeCM to fabricate tactile sensors inspired by the synergistically sensory mechanism of mammalian tactile mechanoreceptors. The synergistically perceiving of HeCM enables the mechanosensational range of the tactile sensor to be significantly enhanced by >100% compared to the one of the counterpart based on the HoCM. The as-designed HeCM tactile sensor characterizes fast, stable, and reproducible electromechanical properties to identify static and dynamic external pressure changes. Wearable three-dimensional tactile panel with auxiliary analog-to-digital conversion circuit demonstrates the fascinating application of the HeCM tactile sensor in the field of human-machine interactions. Noteworthy, the coding and use of the mechanotransduction signal from the HeCM tactile sensor do not need any denoise processing or signal amplification that significantly reduces the difficulty of circuit design specially for multiple units integrated system and makes the competitive advantage compared to other sensors. As a proof-of-concept, the HeCM tactile sensor is served as the functional sensing medium of a data glove to turn actual fingertip events into specified interactions of virtual reality system, where precise and free control of virtual navigation is realized.
Understanding Interface-Driven Mechanisms in Biphase Nanolaminates

Irene Beyerlein, University of California at Santa Barbara, America

Nanolayered materials are a material class gaining much attention due not only to their ten-fold enhancement in strengths higher over those of its constituents, but also the tunability of this exceptional strength with layer thickness and interface engineering. They have also demonstrated high radiation damage tolerance, shock resistance, and thermal stability. Approaches to fabricate them include physical vapor deposition (PVD), electrodeposition (ED) for small-scale laboratory size samples and accumulative roll bonding (ARB) for larger-scale bulk size samples. While the positive layer size scaling with decreasing layer thickness applies to many nanolayered material systems, such as nanotwinned materials and bimetallic nanolayered composites (MNCs), its origins are not well understood, hindering exploitation of this material class in broad application.

We present and apply a crystal plasticity based computational method to predict the plastic response and underlying deformation mechanisms of nanotwinned metals and MNCs. Interface-driven mechanisms, such as confined layer slip, pseudomorphic phase transformation, and interface dislocation emission, are introduced. Calculations are applied to Cu/Nb, Cu/Ta, Zr/Nb, and Mg/Nb nanolaminates and compared to measured strengths over a wide range of layer thicknesses, plastic anisotropy, texture evolution, and/or transmission electron microscopy analyses. We discuss the origins of size effects in strength identified by the model and effective ways to tune the microstructure.

Two-dimensional layered materials such as graphene, MoS$_2$, and WSe$_2$ have attracted considerable interest in recent times as semiconductor after Si and becoming an important material platform in condensed matter physics and modern electronics and optoelectronics. The studies to date however generally rely on mechanically exfoliated flakes which always be limited to simple 2D materials, especially 2D lateral complicated structure can not be prepared through exfoliation strategy. Much like the traditional semiconductor technique, complicated structure such as controlling the space distribution of composition and electronic structure of two dimensional semiconductor material is essential to construct all modern electronic and optoelectronic devices, including transistors, p–n diodes, photovoltaic/photodetection devices, light-emitting diodes and laser diodes. And many physics phenomenon can only appear in more complicated structure. To fully explore the potential of this new class of materials, it is necessary to develop rational synthetic strategies of two dimensional lateral complicated struture,such as lateral heterostructure,superlattice, quantum well, etc. With a relatively small lattice mismatch (~4%) between MoS$_2$ and MoSe$_2$, or WSe$_2$ and WSe$_2$, it is possible to produce coherent MoS$_2$-MoSe$_2$ and WSe$_2$-WSe$_2$ heterostructures through a lateral epitaxial process (Fig.1a). Our studies indicate that simple sequential growth often fails to produce the desired heterostructures because the edge growth front can be easily passivated after termination of the first growth and exposure to ambient conditions. To retain a fresh, unpassivated edge growth front is important for successive lateral epitaxial growth. To this end, we have designed a thermal CVD process that allows in situ switching of the vapour-phase reactants to enable lateral epitaxial growth of single- or few-layer TMD lateral heterostructures. We used this technique to realize the growth of compositionally modulated MoS$_2$-MoSe$_2$ and WSe$_2$-WSe$_2$ lateral heterostructures. From the Fig.1 b, c, d, e we can see the formation of WSe$_2$-WSe$_2$ lateral heterostructures clearly. The WSe$_2$-WSe$_2$ lateral heterostructures with both p- and n-type characteristics can also allow us to construct many other functional devices, for example, a CMOS inverter. Fig. 1g is the optical image of the invert constructed using the WSe$_2$-WSe$_2$ lateral heterostructures and the curves of the output–input and the voltage gain. The voltage gain reaches as large as 24.

In a typical sequential-growth process for 2D lateral heterostructure, the excessive thermal degradation or uncontrolled nucleation during the temperature swing between sequential growth steps represents the key obstacle to reliable formation of monolayer
Technical Program

heterostructure or other lateral complicated structure. We designed a modified CVD system. We used a reverse flow from the substrate to the source during the temperature swing between successive growth steps. A forward flow from the chemical vapor source was only applied at the exact growth temperature. With such reverse flow, the existing monolayer materials will not expose to high temperature and chemical vapor source at the temperature increasing and decreasing steps to minimize thermal degradation and eliminate uncontrolled homogeneous nucleation. With a high degree of controllability in each step, the integrity and quality of monolayer heterostructures can be well preserved after multiple sequential growth steps. We used our approach initially for the general synthesis of a wide range of 2D crystal heterostructures. We also grew more complex compositionally modulated superlattices or multilayer heterostructures, the number of periods and repeated spacing can be readily varied during growth. HADDF-STEM analysis of the atomic structure of the lateral heterostructures and Multilayer heterostructures show the atomically sharp interface can be clearly observed.

14:25-14:50 Invited (1234551)

Direct Growth of Single Crystalline Graphene Film on Germanium Substrate
Zengfeng Di, Shanghai Institute of Microsystem and Information Technology, Chinese Academy of Sciences, China

Graphene has been predicted to play a role in post-silicon electronics due to the excellent electrical performance. Chemical vapor deposition (CVD) of graphene on transition metals has been considered as a major step towards commercial realization of graphene. However, fabrication based on transition metals involves an inevitable transfer step which can be as complicated as the deposition of graphene itself. By ambient-pressure CVD, we demonstrate large-scale and uniform deposition of high-quality graphene directly on a germanium (Ge) substrate. In addition, on both Ge (110) and off-cut Ge (100) substrate, the nucleated graphene domains are inclined to align along the identical direction, thus merging into the continuous graphene film without grain boundary, i.e., wafer-scale single crystalline graphene.

14:50-15:10(1222644)

Numerical Study of Three-Body Diamond Abrasive Nanoindentation Single-Crystal Si by a Molecular Dynamics Simulation
Fa Zhang, Guizhou University, China

With the development of technology and industry, because of the high physical strength, wear resistance, high temperature resistance, corrosion resistance, oxidation resistance and other physical and mechanical properties of hard and brittle materials such as silicon germanium semiconductors, they can withstand the harsh working environment in which polymer materials and metal materials are difficult to handle. At the same time, exploring the indentation effect and accuracy of nanoindentation on materials has always been a key issue in material properties research. Most scholars use a two-body indentation model, but the actual operation tells us that the abrasive particles are rigid bodies of three-body motion (self-rotation of abrasive grains). We used molecular dynamics (MD) simulation to study the nanoindentation mechanism of three-body diamond abrasive grains rotating at different speeds on single crystal silicon materials. In-depth study of three-body diamond abrasive nanoindentation single crystal silicon process, indentation stress, dislocation, crack propagation, coordination number, defect atom, load, nanoindentation zone temperature and potential energy changes. The results show that the smaller the rotational speed of the three-body abrasive particles, the greater the stress in all directions, the more dislocations are easily observed in the workpiece, and the brittle-ductile transition is produced. In addition, the greater the rotational speed of the abrasive particles, the less the number of Si-II phase transitions in the workpiece, and the number of defective atoms inside the workpiece after the three-body abrasive nanoindentation is greater than the amount between the two. In addition, the faster the abrasive grain rotation speed is, the higher the temperature of the nanoindentation zone of the workpiece, the larger the potential energy is, the more obvious the atomic displacement movement in the workpiece is, and the more atomic motion inside the atom. The workpiece is biased in the direction of rotation. In addition, the faster the grain rotation speed of the three-body nanoindentation, the smaller the average load on the workpiece.

15:10-15:30(1308393)

Programmable and Multilevel Memory based on Graphdiyne-MoS2 Heterostructure
Jialing Wen, Zheng Zhang, Yue Zhang, University of Science and Technology Beijing, China

High-capacity storage is a pivotal component for integrated circuits as reducing the cost of constructing integrated circuits are more challenging and urgent nowadays with constrain of lithography technique and ever-growing amount of data. Two-dimensional materials with thickness, transparency, flexibility, startling physical and chemical properties are therefore being explored as multilevel memory. Multilevel memory is expected to enlarge capacity and realize low cost at the same time and the performance of two-dimensional memory is still under study. Graphdiyne (GDY), a new carbon allotrope with three hybridization
states and various chemical bonds, is able to provide carrier trapping sites in memory devices. Herein, a switchable multilevel bilayer memory device with GDY and MoS$_2$ is constructed. This hetero-bilayer memory with GDY enabling efficient and reversible charge trapping states for MoS$_2$ channel is based on field effect transistor and shows large memory window (100V), high degree of modulation (on/off ratio more than 10$^4$), and programmable multilevel states (9 levels or 5 levels under different procedures). Significantly, the memory device exhibits ultralong retention time at its off states at 10$^{12}$ A. Eventually, this GDY/MoS$_2$ memory transistor provides a novel application of GDY material and promotes process to deduce both power consumption and production cost of integrated circuits.

15:30-16:10  Tea Break

16:10-16:35 Invited (1269111)

Evaluating Microstructures and Interfaces by High Speed Nanoindentation Mapping

Eric Hintsala, Douglas Stauffer, America Youxing Chen, University of Minnesota University of North Carolina, America; Daniel Sorensen, Ohio State University, America; Joseph Stevick, Liquidmetal Technologies, America

Recent advancements in high speed nanoindentation mapping have many potential applications, but it can be argued that exploring mechanical properties distributions at materials interfaces is the most impactful. Not only are the strongest property variations typically observed at interfaces, but they are also crucial to performance of the bulk material or component. In some cases, they are introduced purposefully to enhance properties, but can also represent a common failure point. Owing to relatively easy sample preparation compared to the amount of testable area, these techniques can also be considered high throughput and can be adapted to exploring a variety of interfaces through control of the indent spacing, tip geometry and force/depth of the test. Furthermore, the technique can also be applied at elevated or cryogenic temperatures, and in controlled environments to limit problematic contamination or oxidation. Several examples will be presented and discussed that help illustrate the potential. One example is that of laser welding of bulk metallic glasses to titanium alloys for medical devices, an exotic materials joining problem where a variety of brittle intermetallics could form. Here, nanoindentation mapping was used as a critical input alongside structural characterization to optimize welding conditions by establishing statistical significance to the different processing conditions (primarily laser settings). Another example is that of evaluating potential nuclear materials at high temperature, such as additively manufactured and high entropy alloys with enhanced tolerance for absorbing radiation defects. Here, evaluating the stability of mechanical properties at elevated temperatures is possible, and, through use of correlated EBSD, these can be separated by the microstructurally constitutive phases. Looking towards the future, with the increasing ability and desire to link nanoscale properties to bulk performance, such techniques have great potential to deliver large datasets needed for evaluation and refinement of modelling efforts.

16:35-17:00 Invited(1232758)

Structural and Functional Properties of Ultrafine-Grained Si Produced by High-Pressure Torsion

Yoshifumi Ikoma, Zenji Horita, Kyushu University, Japan

The application of severe plastic deformation (SPD) to semiconductor materials is of great interest for exploring novel properties associated with the formations of nanograins and metastable phases. Among various SPD techniques, high-pressure torsion (HPT) is applicable to brittle materials such as intermetallics, ceramics and semiconductors. We have applied HPT to crystalline Si, and found the formation of nanograins including diamond-cubic Si-I and metastable phases such as body-centered-cubic Si-III and rhombohedral Si-XII as well as amorphous phase. The formation of metastable phases was enhanced by imposed shear strain. The electrical resistivity increased after compression without anvil rotation, while it decreased after HPT processing for 10 revolutions due to the formation of metastable phases, especially semimetallic Si-III. Photoelectron spectroscopy measurements revealed that the valence band (VB) spectra corresponded to a mixture of Si-I, Si-III and Si-XII, and the VB edge was located at ~0.3eV. After annealing at 200℃, the VB spectra changed to a mixture of Si-I and hexagonal-diamond Si-IV, and the VB edge shifted by ~0.4eV. It was found that the Si-III/XII phases in the HPT-processed samples have a smaller bandgap than that of Si-I. No appreciable photoluminescence (PL) was observed from the HPT-processed samples because of a high density of lattice defects. A broad PL peak in the visible light region appeared after annealing at 600℃. Since high-resolution transmission electron microscopy observations revealed that Si-III and Si-XII phases disappeared and Si-I nanograins remained after annealing, the observed PL was due to a quantum confinement effect. These results conclude that the HPT processing is promising for the development of various functional properties of Si.
17:00-17:20 (1233679)

The Effect of Gradient Order on Mechanical Behaviors of Gradient Nanotwinned Cu

Zhao Cheng, Institute of Metal Research, Chinese Academy of Sciences, China / University of Science and Technology of China, China; Lei Lu, Institute of Metal Research, Chinese Academy of Sciences, China

Gradient structures, such as gradient nanotwinned structures and gradient nano-grained structures, possessing unique strengthening and work hardening, have opened an avenue towards understanding the gradient structure-related mechanical behavior. Gradient nanostructured metals with a normal gradient order (hard surfaces and soft core) or the reverse gradient order (soft surfaces and hard core) were often believed to possess superior mechanical properties with respect to their homogeneous counterparts due to the extra incompatible plastic deformation along the gradient depth. However, quantitative understanding the effect of gradient order on the mechanical behavior of the gradient metals remains unclear.

In this study, we designed and fabricated two gradient nanotwinned Cu with reverse gradient orders but the same structural gradient, including GNT-I with a normal gradient order (hard surfaces and soft core) but GNT-II with the reverse one. Tension tests indicated that GNT-I exhibits a higher strength and a lower surface roughening during deformation than GNT-II. Interestingly, GNT-I has the faster elastic-plastic transition than GNT-II, which is demonstrated by much steeper tensile curves and work hardening-true strain curves at small strains.

The systematic study on lateral deformation of GNT tensile samples reveals that the normal gradient order offsets but the reverse order aggravates the surface effect that the plastic deformation starts at the surface and then extends into the sample core due to the less constraint of grains near surface. Such a stronger constraint is therefore generated in GNT-I and may postpone the yielding of the soft core and accelerate the progressive yielding from soft core to the hard surface, both of which lead to the faster elastic-plastic transition in GNT-I. The larger strain gradient detected in GNT-I is consistent with the mechanical properties that higher yield strength according to the classic theory of strain gradient plasticity. Besides, the finer grains and thinner twin lamellae at the hard surfaces are responsible for the lower surface roughening in the GNT-I during the tensile deformation. This study exploring deformation behaviors related to the gradient order in GNT metals will afford a guide for architecting gradient structures in metals for superior mechanical properties.
K. Nanocrystalline Materials, and Ultra-Fine Grained Materials: VI
Symposium Organizers:
Yue Zhang, University of Science and Technology Beijing, China; Zhiyong Tang, National Center for Nanoscience and Technology, China; Nobuhiro Tsuji, Kyoto University, Japan; Jae-il Jang, Hanyang University, Korea; Kenong Xia, University of Melbourne, Australia; Nathan Mara, University of Minnesota, USA

Thursday AM  Room: Room 309(3rd Floor)
August 22, 2019  Symposium: K

Chairs:

8:30-9:00 Keynote (1235345)
**Fabrication and Mechanical Behavior of Porous Cu via Chemical De-Alloying Method**
*Fei Chen, Hao Wang, Qiang Shen, Lianmeng Zhang, Wuhan University of Technology, China; Lavernia, University of California Irvine, America*

We report on a study of the synthesis and deformation of two different porosity structure: nano-sized and micron-sized porous Cu, processed via chemical de-alloying of Cu-Al intermetallic compounds and Cu-Fe solid solution in 20 wt% NaOH aqueous solution and 5 wt% H2SO4 respectively. We studied the relationship between phase composition/microstructure of the precursor alloys and structural properties (volume fraction of porosity, density, pore size distribution) of porous Cu. Moreover, the compressive strength of bulk nano/micron porous Cu were measured and the effect of volume fraction of porosity on mechanical properties was studied. The results show that the microstructure of both nanoporous Cu and micron porous Cu are characterized by bi-continuous interpenetrating ligament-pores. The ligament size of nanoporous Cu is 130±20nm -170±20nm, and its corresponding compressive strength increases with decreasing volume fraction of porosity; as porosity increased 56.372% to 73.972%, the compressive strength decreased from 17.1871MPa to 2.7170MPa. Moreover, the average pore size of micron porous Cu is in the range of 1.5~4.0 μm.

9:00-9:30 Keynote (1232984)
**Dynamic Tensile Extrusion Behavior of OFHC Cu Having Ultrafine Grains and Fine Grains**
*Kyung-Tae Park, Hanbat National University, Korea; Keunho Lee, Keunho Lee, LeeJu Park, Seong Lee, Agency for Defense Development, Korea*

A series of dynamic tensile extrusion (DTE) tests was performed on ultrafine grained (UFG) OFHC Cu to examine the metal jet formability under dynamic loading conditions (i.e. deformation at extremely high strain rates).The UFG Cu bars were fabricated by equal channel angular pressing (ECAP) with the route Bc and then annealing was conducted at 150°C and 200°C for 1hr in order to control the grain size. The as-ECAPed grain size of ~0.9 micrometer increased to 2.8 micrometer and 4.7 micrometer after 150°C and 200°C annealing. The spherical balls of the diameter 7mm were carefully machined from the as-ECAPed bar and the subsequently annealed bar. The DTE tests were conducted by launching the balls to the conical DTE die at a speed of ~ 480m/s using a vacuumed gas gun system. The DTE fragments were softly recovered and their microstructure was characterized by using EBSD technique. The DTE ductility (the sum of elongation of each fragment) increased as increasing the grain size under the present grain size range. The computational analysis showed that the temperature of the specimen during DTE increased over ~ 600°C locally due to adiabatic heating, and the maximum strain rate reached 10^6/s. The EBSD analysis revealed that, regardless of the initial grain size, <001> + <111> dual fiber texture was strongly developed along the DTE direction. Besides, it was evident that dynamic recrystallization in the as-ECAPed condition was more frequent compared to the annealed condition. Based on the present extremely high strain rate DTE tests, the metal jet formability of OFHC having the grain size ranging from ultrafine size (~ 1 micrometer) to fine size (2~5 micrometer) is discussed.

9:30-9:55 Invited (1224773)
**Grain Refinement in a Metastable Beta Ti Alloy Deformed to Large Strains at High Strain Rates**
*Ahmad Zafari, The University of Melbourne, Australia*

It has long been known that severe plastic deformation (SPD) can effectively refine grains in metallic materials. However, the roles played by stress induced transformation, in particular martensitic transformation, as well as strain rate in the grain refinement have received little attention. Hence, we studied these two factors in a metastable β Ti-5Al-5V-5Mo-3Cr (Ti-5553) after conducting high pressure torsion (HPT) and shear punching (SP) at different strain rates of 0.02~200s⁻¹. It was revealed that the presence of the martensitic α"
in Ti-5553 resulted in grains of 30~50nm, much smaller than those of 100nm formed in more stable β Ti alloys, such as Ti-20Mo, in which no stress induced martensitic transformation (SIMT) occurred. The formation of thin α’ plates divided β grains into smaller domains and imbalanced dislocation generation and diffusion at grain boundaries. The rates of dislocation generation and absorption at the boundaries were further manipulated by varying strain rate during SP. SP to large plastic strains up to 40 was conducted at various speeds, creating severe plastic deformation at high shear rates up to 200s⁻¹. Grain refinement in the resulting shear bands (SBs), which experienced even faster deformation due to strain localisation, was studied using extensive TEM. Significant grain refinement occurred at < 40s⁻¹, producing β grains of ~5–10nm, considerably smaller than those obtained by HPT to a much higher strain of 240 at a slow shear rate of 0.5s⁻¹. The applied shear strain rate of 2s⁻¹ was found to be an optimum for attaining a pure nano-β grain structure thanks to maximum dislocation activity and complete α’ to β reverse transformation. However, some stress induced α’ remained as strain rate deviated from this value and much coarser β grains formed at the strain rates of > 40s⁻¹. The effects of strain rates on grain refinement and the minimum achievable grain sizes at high strain rates are explained based on classical dislocation dynamics.

9:55-10:15 (1222554)

Microstructures and Tensile Properties of Ultrafine Grained Al-Mg Alloys Fabricated by High Pressure Torsion and Subsequent Annealing

Xiaodong Lan, Si Gao, Myeong-heom Park, Akinobu Shibata, Nobuhiro Tsuji, Kyoto University, Japan/Element Strategy Initiative for Structural Materials, Japan

Al-Mg alloys have been widely used because of their good combination of low density, high strength and excellent formability. Previous studies have shown that increasing Mg content leads to simultaneous improvement of strength and ductility of Al-Mg alloys. On the other hand, it is well-known that grain-size refinement can effectively improve strength of metallic materials according to Hall-Petch effect, but ductility is deteriorated when the grain size is decreased down to sub-micrometer sizes (ultrafine range), which is known as a trade-off relation between strength and ductility. Although intensive studies have been carried out on Al-Mg alloys, the combined effect of Mg contents and grain size on mechanical properties of Al-Mg alloys remains unclear, especially when the grain size is refined down to the ultrafine grain size range. The present study aims to investigate microstructure and mechanical properties of UFG Al-Mg alloys having different Mg contents.

Al-xMg (x=2.5, 5 and 7.5wt.%) specimens having different grain sizes were fabricated by high pressure torsion (HPT) and subsequent annealing process. Microstructural characterizations were conducted on transverse sections of the HPT disks by using SEM-BSE and EBSD. Tensile test was conducted at a strain rate of 8.3×10⁻⁴s⁻¹ at ambient temperature, using miniaturized tensile test specimens cut from the HPT discs. Digital image correlation (DIC) method was used to characterize local deformation behavior as well as to precisely measure tensile elongation in the tensile tests. It was found that yield strength and ultimate tensile strength significantly increased by refining the grain size down to sub-micrometer but tensile elongation substantially decreased by the grain refinement in all three kinds of Al-Mg alloys. However, at all the grain sizes, specimens with higher Mg content maintained larger tensile ductility than those with lower Mg content. It was found that strain hardening rate during the tensile test significantly increased with increasing Mg content at similar grain sizes, which should account for the larger tensile ductility in the specimens with higher Mg content. On the other hand, it was noteworthy that yielding behavior changed from continuous yielding to discontinuous yielding with decreasing the grain size in all alloys. The effects of grain size and Mg content on the yielding behavior, yield strength and strain hardening behavior of the specimens are discussed in details by comparing Al-xMg alloys with pure Al in the presentation.

10:15-10:35 (1222138)

Influence of Cryomilling and High Pressure on the Consolidation of Aluminum by Spark Plasma Sintering

Dongming Liu, Zhongyu Wang, Jiale Zheng, Shandong University, China

There has been a significant interest in the development of bulk nano-structured or ultra-fine grained materials because the grain refinement may lead to substantial changes in both mechanical and physical properties. We report on cryo-milling as well as spark plasma sintering (SPS) are employed to consolidate nano-structured pure aluminum. Cryomilling is a special kind of a mechanical attrition technique, in which the powder and the milling balls are immersed in a cryogenic medium (e.g., liquid nitrogen or liquid argon) during the milling process. In the case that liquid nitrogen is used as the cryogenic medium, the extremely low temperature can suppress the recovery and recrystallization and lead to finer grain structures and more rapid grain refinement. Meanwhile, the incorporation of the nitrogen into the powder particles improves the mechanical properties and the thermal stability of the microstructure.
Spark plasma sintering (SPS) is a recently developed powder consolidation technology, in which a pulsed high-amperage, direct current (DC) is used concurrently with a superimposed uniaxial pressure to consolidate powders. In comparison with well-established consolidation methods such as hot pressing, where the samples are heated externally, the thermal energy during SPS is generated in-situ, i.e., by the powder being sintered in case of conductive powder as well as by the mold elements. This technique possesses the advantage of sintering powders under conditions of high heating rate (up to 1000°C/min). Consequently, materials can be consolidated at relatively low temperatures and in a shorter time interval, and thereby minimize changes to the starting microstructures, relative to conditions generally present during conventional consolidation methods.

In this paper, nearly full dense bulk nanostructured aluminum is fabricated by cryomilling and high-pressure spark plasma sintering. The consolidated material demonstrates a compressive yield stress as high as 380MPa, which is significant higher than commercial strain hardened aluminum. Microstructural observation shows that it has a bimodal structure, i.e., ultra-fine grains are embedded a nano-sized grain of aluminum. In addition, the effect of cryomilling and high pressure (up to 500MPa) on the consolidation is thoroughly analyzed. It is also shown that the change in the carbon and oxygen level is insignificant during spark plasma sintering. There is a slight decrease in the nitrogen content. And the hydrogen level is significantly reduced. Quantitative calculation was performed to analyze the contributors to the high strength. It is shown that the strengthening mechanisms include grain boundary strengthening, second phase strengthening and dislocation strengthening.

10:30-10:45  Tea Break

11:10-11:35 Invited (1224111)

The Effect of Solutes and Second-Phase Particles on Grain Refinement during ECAP in Al Alloys

Yan Huang, Brunel University London, England

The exploitation of the optimized effect of solute additions and second-phase particles has potential to develop commercially viable routes for producing true bulk nanograindium alloys. In the present work, the effect of solute elements and second-phase particles in aluminium on inhibiting dynamic recovery and pinning boundary migration during SPD by equal channel angular pressing (ECAP) was investigated. ECAP was carried out, at room temperature and cryogenic temperatures down to 77K, using either a 90° or 120° die and following route A, to various true strains of up to 15. The deformed microstructures were characterized by FEGSEM, TEM and EBSD. Experimental results showed that increased Mg addition led to more instability in the early stages of deformation and consequently improved grain refinement as medium to high angle boundaries were generated by shear banding. At room temperature, the average grain width after a strain of 10 was stabilized at ~550nm in an Al-0.1Mg alloy and ~120nm in an Al-3Mg alloy. However, shear banding in the supersaturated Al-4Cu was found even more severe and the shear bands carried most of the strains in the first two ECAE passes. The grain width obtained after the same strain of 10 was down to ~65nm, which is a true nanoscale. The combined effect of Al,Sc particles and Mg solute in Al-0.25c-0.1Mg and Al-0.2Sc-1Mg alloys resulted in finer structures than in the Al-0.1Mg and Al-1Mg solid solutions. In the Al-4Cu alloy, the nanoscale equilibrium q-phase (Al2Cu) particles showed limited impact on the microstructure upon further deformation. The minimum grain width...
K-1: Engineering on Ni-Co-S Bifunctional Electrocatalyst for Water-Splitting (1283356)

Jing Wu, Huijing Guo, Zhuo Kang, Yue Zhang, University of Science and Technology Beijing, China; Yue Zhang, University of Science and Technology Beijing, China

Efficient evolution of hydrogen and oxygen simultaneously with earth-abundant, highly active, and robust bifunctional electrocatalysts has been a significant concern for water splitting. Herein, non-noble metal based Ni-Co-S bifunctional catalysts with tunable stoichiometry and morphology were realized. The electronic structure engineering and subsequent morphological design synergistically contributed to significantly elevated electrocatalytic performance. Overpotentials $\eta$ of 243mV (vs.RHE) for OER and 80mV for HER in 1M KOH solution, as well as Tafel slopes of 54.9mV/dec for OER and 58.5mV/dec for HER were demonstrated together with prominent stability. In addition, DFT calculation was conducted to indicate the optimal electronic structure via visually mapped electron density difference of catalysts, and representatively verify that the enhanced OER activity of NiCo$_2$S$_4$ nanosheets is concerned with the Co top site on (110) surface. Moreover, the tandem bifunctional NiCo$_2$S$_4$ anode and cathode exhibited a required voltage down to 1.58V ($J=10\text{mA/cm}^2$) for simultaneous OER and HER, and no obvious performance decayed after 72h. When integrated with a GaAs solar cell, such photo-assisted water splitting electrolyzer acquired a certificated solar-to-hydrogen efficiency up to 18.01%, further demonstrating the feasible engineering protocols as well as the promising potential of bifunctional NiCo2S4 for large-scale overall water splitting.

K-2: Magnetic Metal Cobalt/Reduced Graphene Oxide (M-Co/RGO) Nanocomposites with Tunable and High Performance Electromagnetic Wave Absorption Capabilities(1309550)

Qi Li, Qingliang Liao, Yue Zhang, University of Science and Technology Beijing, China

From the half-century, with the broad application of electronic devices and wireless communication equipment in civil and military fields. Electromagnetic interference (EMI) or electromagnetic pollution have been paid more and more widespread attention all over the world. To address the problem effectively are not only beneficial for national defense security, but also for the improvement of the harmonious human living environment. The magnetic metal nanoparticles have attracted considerable attention due to the excellent EM wave absorption properties as well as the controllable synthesis of dimensions and shapes. Consequently, RGO has been functionalized by various magnetic metal nanocrystals. Herein, the prepared M-Co nanoparticles have relative high saturation magnetization (Ms) and hysteresis, which have potentials to be employed as excellent magnetic loss EM wave absorption materials. Magnetic metal cobalt (M-Co) nanoparticles are evenly dispersed on reduced graphene oxide (RGO) without pronounced aggregation. The M-Co/RGO nanocomposites exhibit tunable and high-performance electromagnetic (EM) wave absorbing properties. The excellent EM wave absorbing properties are due to the dielectric-magnetic loss synergistic effect as well as the well-matched impedance matching. When the mass ratio of M-Co and RGO is 5:1 and the filler loading of the nanocomposite is 20%, the maximum RL reach as strong as $-32.8 \text{dB}$ at 11.7GHz with a thickness of 2.2mm, and the effective absorption bandwidth up to 5.7GHz at a thickness of 1.8mm. The highly excellent EM wave absorbing performance mainly originates from synergistic effect of dielectric loss and magnetic loss as well as well-matched impedance matching. The EM wave absorbing mechanisms of M-Co/RGO nanocomposites including dipole polarization relaxation, interfacial polarization relaxation, eddy current loss, natural resonance and conductive loss. The novel M-Co/RGO nanocomposites have superior potentials to be designed as tunable and high-performance absorbents.

K-3: Crystal Orientation Engineering of NiOx Hole Transport Layer in Perovskite Solar Cells(1322735)

Mingyue Shi, Haonan Si, Zhuo Kang, Yue Zhang, University of Science and Technology Beijing, China

Hybrid organic-inorganic perovskite solar cells (PSCs) have attracted great attention over the past years due to their high solar to electric power conversion efficiency
Graphene quantum dots (GQDs) have attractive properties and potential applications. However, their various applications are limited by a current synthetic method which requires long processing time. Here, we report a facile and remarkably rapid method for production of GQDs exhibiting excellent optoelectronic properties. We employed the pulsed laser ablation (PLA) technique to exfoliate GQDs from multi-wall carbon nanotube (MWCNTs), which can be referred to as a pulsed laser exfoliation (PLE) process. Strikingly, it takes only 6min to transform all MWCNTs precursors to GQDs by using PLA process. Furthermore, we could selectively produce either GQDs or graphene oxide quantum dots (GOQDs) by simply changing the organic solvents utilized in the PLA processing. The synthesized GQDs show distinct blue photoluminescence (PL) with excellent quantum yield (QY) up to 12% as well as sufficient brightness and resolution to be suitable for optoelectronic applications. We believe that the PLE process proposed in this work will further open up new routes for the preparation of different optoelectronic nanomaterials.

Moreover, we present the GQDs and GOQDs can be selectively prepared by easy and simple pulsed laser ablation in liquid (PLAL) method by controlling the laser wavelength. The obtained GQDs and GOQDs showed a significantly different optoelectronic nature mainly due to the existence of surface oxygen-rich functional groups (e.g. carboxyl or hydroxy groups). Also, we described a possible mechanism for the formation of oxygen functional groups during the PLAL process based on the Coulomb explosion model, which can give further insight for designing functional carbon materials.

K-5: Temperature Dependence of Harmonic Structure Designed SUS316L Austenitic Stainless Steel Deformation(1235075)

KOKI YAGI, Morihiro Hariki, Masashi Nakatani, Bhupendra Sharma, Mie Kawabata, Kei Ameyama, Ritsumeikan University, Japan; Cinzia Manapace, University of Trento, Japan

SUS316L austenitic stainless steel has variety of industrial applications because of its favorable ductility and corrosion resistance properties. However, relatively low yield strength may make it unsuitable for many structural applications. In stainless steel, the high strength can easily be achieved by many grain refinement methods, but usually with a pronounced sacrifice of ductility. In recent years, to solve this strength-ductility trade-off problem, a new concept of heterogeneous microstructural design called "Harmonic Structure" has been proposed. Harmonic Structure (HS) consists of coarse-grains areas like island surrounded by a continuous fine-grains counterpart three-dimensional network structure. At room temperature, the Harmonic structured SUS316L specimen presented a good combination of high strength and improved uniform elongation. However, under different temperature conditions, the mechanical properties of Harmonic structured SUS316L is not reported yet. Therefore, the present study focuses on the analysis of mechanical properties of Harmonic structured SUS316L at various temperatures.

The SUS316L steel powder, prepared by plasma rotating electrode process (PREP), was used as a starting material. Average particle size in the initial powder was approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out for 180ks, 360ks, and 540ks at a constant milling speed of 150 rpm. Subsequently, the milled powders were sintered by Spark Plasma Sintering (SPS) at 1223K temperature for 5.4ks, 360ks, and 540ks under argon gas atmosphere, at room temperature. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out for 180ks, 360ks, and 540ks at a constant milling speed of 150 rpm. Subsequently, the milled powders were sintered by Spark Plasma Sintering (SPS) at 1223K temperature for 5.4ks, 360ks, and 540ks under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter). The milling was carried out under argon gas atmosphere, at room temperature, wherein the ball-to-powder weight ratio was maintained at 2:1. Mechanical milling was carried out approximately 140μm. Mechanical milling was carried out in a planetary ball mill using SUS304 steel pots and balls (5mm diameter).
Harmonic Structure (HS) is one of heterogeneous microstructures, and it is characterized as 3D ultra-fine grains (UFGs) network structure, named "Shell", with uniformly dispersed coarse grains, "Core". The HS designed materials are able to achieve high strength and high ductility at the same time, compared with the homogeneous coarse grains structure materials. Distribution of strain at the Shell grains during plastic deformation exhibits such superior mechanical properties. In this study, thermo-mechanical processing (TMP), i.e. cold rolling and annealing, was applied to the HS designed pure Titanium, and its microstructure evolution and the mechanical properties are discussed. Fabrication of the HS is based on powder metallurgy route. That is, pure titanium powder was processed by Mechanical Milling (MM) for 360ks at room temperature under Argon gas atmosphere. During MM, nano grains formed in the surface layer of the powder by severe plastic deformation, whereas coarse grains were remained in the middle of powder particles. The Milled powder was subsequently consolidated at 873K for 3.6ks under vacuum by a Spark Plasma Sintering process. The sintered compacts have a topological three-dimensional connected UFG network structure, which is so called "Harmonic Structure". Average grain size and volume fraction of the shell were 19.7nm and 15.7%, respectively. Those compacts were provided to the TMP, that is, 10% cold rolling at room temperature followed by annealing at 873K for 1.8ks. There was few change in the Shell grain size after the TMP, while volume fraction of the shell increased from 15.7% to 23.1%. It is noteworthy that TEM observation revealed existence of dislocation free grains in the Shell region, though a typical deformed structure was observed in

K-7: Selective Recrystallization in Harmonic Structure Designed Pure Titanium via Thermo-Mechanical Processing(1222587)
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Harmonic Structure (HS) material consists of heterogeneous bimodal microstructure with coarse grained area "Core" surrounded by fine grained area "Shell" exhibited the excellent combination of mechanical properties. However, the Harmonic Structured materials have a limited improvement in mechanical properties as compared to its conventional homogeneous counterparts. Therefore, the present study focuses on the further improvement of mechanical properties of Harmonic Structured titanium (HS-Ti) by multi thermomechanical (Multi-TMP) process. In the present study, the Harmonic Structured pure-Ti was prepared by mechanical milling (540ks) followed by spark plasma sintering (1073K, 1.8ks) of PREP Ti powder. Then Multi-TMP process was carried out on as-fabricated HS-Ti specimens. The TMP conditions were as follows: accumulative cold rolling (CR) for 3 passes of 10%, 19%, and 27%, and subsequent annealing at 873K for 1.8ks after each pass. The microstructural analysis was performed by SEM and EBSD techniques. Preliminary microstructure analysis revealed that the microstructure of Multi-TMP processed specimens (CR + annealed) was apparently similar to the as-fabricated HS-Ti specimen. Moreover, the Shell and Core grain size including with overall grain size was approximately similar in all the cases. However, interestingly, the mechanical properties were significantly different. In particular, with increasing CR passes, the strength remarkably increases without significant reduction in ductility. The Kernel Average Misorientation (KAM) image presented that the misorientation increases, at Shell areas, with increasing cold rolling passes. In many HS materials, stress concentration has been reported to take place especially into the Shell region in the early stage of deformation and, as a result, dislocation emission and accumulation occurs in the vicinity of the Shell. Therefore, it was expected that due to those dislocations remained in the Shell region even after "CR + annealing" step, the dislocation density gradient successively generated by each Multi-TMP process. That is, it can be expected that the increasing strength after each CR pass is due to the dislocation strengthening, which play an important role as same as "ultra-fine grain Shell strengthening" in HS materials. In other words, as same as "grain size gradient", "dislocation density gradient" is quite effective mechanism for improving the mechanical properties in HS materials.
the Core region. These results imply that a selective recrystallization took place in the Shell region, but not in the Core grains. Mechanical properties were evaluated by tensile tests. Outstanding mechanical properties such as high proof strength, high UTS, a large elongation and a large tensile toughness were obtained in the TMP-HS Titanium. Increase of UFGs by the selective recrystallization in the Shell resulted not only in higher proof strength, but also the higher UTS with superior ductility. In other words, the remarkable improvement of mechanical properties was attributed to the development of the Shell structure by a selective recrystallization in the Shell. This work was supported by JSPS KAKENHI Grant Number JP18H05256. These supports are gratefully acknowledged.

K-8: Preparation and Application of High-Performance Graphene-Reinforced Copper Tungsten Alloys(1378760)

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In this work, the failed samples were analyzed. The method of damage for CuW80 products used for ultra-high voltage SF6 circuit breaker are mainly cracking, ablation and exfoliation. Therefore, the graphene was added into the electrical contact material of CuW, and the threshold of graphene oxide content was achieved through the calculation based on the percolation model. Furthermore, the process of surface modification and dispersibility improvement of graphene oxides was systematically studied. The optimal process parameters were gotten by a series of work on the fabrication and characterization of RGO/Cu composites. The relationship between density of skeletons and compacting pressure was established and green compacts of skeleton with different components for CuW electrical contact materials were prepared based on the relation curve. Then, CuW electrical contact materials with target composition were obtained by sintering at 930°C for 2h and infiltrating at 1050°C. Simultaneously, two processes of preparation were explored, including infiltration with pure tungsten and activated sintering infiltration.

CuW80 electrical contact materials modified with graphene (0.002~0.2wt.%) were fabricated by activated sintering infiltration method. The properties of the as-prepared materials, including density, hardness, electrical conductivity and bending strength, could reach the national standard of GB/T8320-2003, which completely meet the technical requirement of the research contents. The results revealed that the hardness, electrical conductivity and bending strength of the materials firstly increased and then decreased with an increase of the graphene content. The CuW80 possessed the best performance at the content of 0.002wt.% graphene in the matrix, specially, the wear extent, wear rate and the friction coefficient were 53%, 55% and 76% of the CuW80 products in use, respectively. The performance could satisfy the technical requirement of the project, where the wear performance should improve 20%. In addition, the arc erosion resistance of the as-prepared electrical contact materials under the condition of AC4 (AC, 380V and 120A) was evaluated through mass loss after experienced over 20 and 35 operations. It demonstrated that the mass loss was 44% and 63% of the ultra-high voltage electrical contact materials. The arc erosion resistance also reached the technical requirement of the project that a decrease of 20% in mass loss during arc erosion should achieved.
decreased with decreasing processing temperature, although the effect of temperature was not as strong as expected based on thermal activation analysis. Shear banding was found to be most effective in the grain refinement during early stages of deformation. Second-phase particles played an important role in stabilizing the obtained fine grain structure. The mechanisms of grain refinement during ECAP are discussed in the paper in association with the contribution of solute atoms and second-phase particles.

11:35-12:00 Invited (1235017)
Effect of Severe Plastic Deformation and Subsequent Aging on Strength-Ductility Balance of Ultra-Fine Grained Al-Si-Mg Alloy
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Ultra-fine grained metallic materials fabricated by severe plastic deformation processes exhibit quite high strength, but these ductility is often small. Several researchers report combination of ultra-fine grained microstructure with precipitates can improve the ductility. However, the relationships between mechanical properties and process conditions, such as deformation strain and heat treatment temperature have not been clarify yet. Thus, in this study, age hardenable aluminum alloys was deformed with various strain and aged under various conditions, and the mechanical properties were investigated.

Al-Mg-Si alloy sheets were solution treated and then deformed by cold-rolling with various reductions or severely deformed by accumulative roll-bonding (ARB) process. The deformed sheets were subsequently aged at 100°C or 170°C with various aging time. Hardness test and tensile tests for these specimens were carried out. In the specimens deformed by cold-rolling with reduction below 50%, the yield stress and tensile strength were increased and total elongation was decreased by aging at 170°C. The result shows trade-off relationship between the strength and ductility. On the other hand, both strength and ductility increased during aging in the specimens deformed with reduction above 50% or by ARB process. The result indicates that the strength and ductility were related to positive correlation when the specimens were pre-deformed severely. When the deformed specimens aged at 100°C, trade-off relationship was observed in the specimen deformed below 10% reduction. In the specimen deformed with reduction above 20%, positive relationship of the strength and ductility appeared. The results indicate that the mechanical properties depend on strain of deformation and aging temperature, and the relationships between strength and ductility can be controlled.

12:00:12:20(1312245)
Effect of Grain Size on Mechanical Properties of Mg-0.3at.%Y Dilute Alloy
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In this study, a Mg-0.3at.%Y alloy was provided for a severe plastic deformation by high pressure torsion (HPT) and subsequent annealing. After the HPT by 5 rotations, nanocrystalline structures with a mean grain size of about 240nm having deformed characteristics were obtained. Fully recrystallized microstructures with mean grain sizes ranging from sub-micron to several tens of micrometers were obtained by subsequent annealing at various temperatures for different holding times. Room temperature tensile tests revealed that ultrafine grained (UFG; grain sizes smaller than 1μm) specimens exhibited very high yield strength over 250MPa but quite limited tensile ductility. In contrast, good balance of strength and ductility was realized in fine grained specimens with grain sizes around 2 to 5μm. Particularly, the yield strength and total tensile elongation of a specimen with a mean grain size of 2.13μm were 180MPa and 37%, respectively, which were much higher than those of pure Mg having a similar grain size. The deformation microstructures of typical fine grained and coarse grained Mg-Y specimens were systematically observed by electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). It was found that the dominant deformation mode has been changed in the fine grained specimen. The significant effects of grain size and Y addition on the change of deformation mechanisms were discussed.