SYMPOSIUM S
Integrative and Interdisciplinary Aspects of Intermetallics

November 29 - December 1, 2004

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* Invited paper
SESSION S1: Iron and Nickel Aluminides  
Chairs: I. Baker and Y. Mishima  
Monday Morning, November 25, 2004  
Room 208 (Hyatt)

8:30 AM S1.1  

This paper summarizes our recent research on solute effects in Ni-rich NiAl alloys. As Ni and Co solute atoms occupy the Al sublattice sites in NiAl, normal lattice contraction and solid solution hardening have been observed. On the other hand, as Fe, Mn and Cr solute atoms occupy the Al sublattice, experimental studies reveal unusual lattice dilation and resultant solid solution softening in NiAl alloys, all of which can not be explained by the current solid solution theories. To explain these results, the first-principles calculations reveal the development of a large electron spin polarization only when the solute atoms of Fe, Mn and Cr are surrounded by Ni atoms in NiAl lattice. This spin polarization results in a large magnetic moment (e.g., 2.5 μB per Fe atom) that dilates the lattice parameter and affects mechanical properties. The calculated magnetic moment is unambiguously supported by electron energy-loss spectroscopy (EELS) and the measurements of magnetic susceptibility as a function of temperature. Effect of magnetic interaction on alloy design of NiAl alloys will be discussed. Research sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-000OR22725 with UT-Battelle, LLC.

9:00 AM S1.2  
Microstructures and Mechanical Properties of NiAl-Mo Composites, Hongbin Bi1, 2 and Easo P. George1, 2; Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; 3Materials Science and Engineering, The University of Tennessee, Knoxville, Tennessee.

NiAl-Mo ternary eutectic alloys were directionally solidified in a high-temperature optical floating zone furnace to obtain well-aligned microstructures consisting of NiAl matrix and continuous Mo fibers having a square cross-section. With increasing growth rate, the spacing and the size of the Mo fibers decreased, with fiber small as 40μm in diameter and 60μm in length in the as-cast section obtained at a growth rate of 80 mm/h. Electron backscatter diffraction patterns identified the growth directions and Mo/NiAl interface boundaries to be parallel to <100> (110), respectively, in both Mo and NiAl. Tensile tests were used to investigate the temperature dependence of the strength and ductility of the composite. Its ductile-to-brittle transition temperature was found to be 675°C, and its yield strength about 1.5 times that of NiAl single crystal at 800°C. * Research sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract DE-AC05-000OR22725 with UT-Battelle, LLC.

9:15 AM S1.3  
Thermal Stress Induced Cracking of NiAl Thin Films Constrained by a Si Substrate, Gerhard Dehm1, Patrick Wellner2, Oliver Kraft2, Janis Andersson3, Helmut Clemens4 and Eduard Arzt4; 1Max Planck Institute for Metals Research, Stuttgart, Germany; 2Institut fuer Materialforschung II, Forschungszentrum Karlsruhe and Institut fuer Zuverlassigkeit von Bauteilen und Systemen, Universitaet Karlsruhe, Karlsruhe, Germany; 3Institute of Polymeric Mechanics, Riga, Latvia; 4Physical Metallurgy and Materials Testing, University of Leoben, Leoben, Austria.

It is well established that flow stresses of metallic thin films increase with decreasing thicknesses from values significantly exceeding those of the corresponding bulk material. However, it is unclear whether similar size effects are present in the craze behavior of brittle thin films constrained by a substrate. Whereas theoretical models are reported in literature, a conclusive experimental picture has not yet been obtained. In the present study we analysed the cracking behavior of intermetallic NiAl films on Si substrates as a function of the Al content (45.0 to 52.4 at-percent) and film thickness (400 to 3000nm). The films were thermally cycled to a maximum temperature of 1000K, resulting in a thermal strain of up to 0.9 percent. Ni-rich NiAl films were found to sustain higher tensile stresses than Al-rich films. Al-rich NiAl films failed by the formation of intergranular cracks, which extends from the Si substrate. The fracture toughness of the Al-rich films was determined from the fracture stress, substrate crack depth and film thickness. In contrast to the fracture toughness, the fracture stress was found to increase with decreasing film thickness indicating that the film thickness corresponds to a critical crack length.

9:30 AM S1.4  
First-principles Study of Structural and Defect Properties in FeCo Intermetallics, Maja Krcmar, Chong Long Fu and James R. Morris; Oak Ridge National Lab, Oak Ridge, Tennessee.

Using first-principles calculations and statistical mechanical modeling, we investigate magnetism-induced structural stability, defect structure, and order-disorder transition characteristics in FeCo alloys. We find that the ordered B2 structure is stabilized by the presence of magnetism. However, B2 FeCo is only weakly ordered: antistucture defects dominate point-defect structure on both sides of stoichiometry, and the presence of lattice vacancies is negligibly small. We then use density-functional theory to estimate the size of the antistucture defects and order-disorder transition temperature Tc as a function of alloy compositions near stoichiometry. We find that the stoichiometric FeCo has the highest Tc, and that Tc decreases parabolically with the increase in variation of off-stoichiometric composition from stoichiometry. The anti-phase boundary (APB) energies for <111> slip on the (110) and (112) planes are found to be low, despite the brittle nature of B2 FeCo. Due to the weak ordering, we also find that the structural stability of B2 FeCo is very sensitive to the reduction of dimensionality (3D to 2D), and the state of stress. Finally, we discuss conditions for stress-induced structural phase transformation in FeCo, suggesting the possibility of local structural changes under the influence of stress near/around the B2-FeCo phase transition.

9:45 AM S1.5  
Effect of Heat Treatments on Microstructures of Rapidly Solidified TiCo Ribbons, Kyosuke Yoshimi1, Akira Yamauchi2, Nakamura Ryusuke1, Sadahiro Tsurekawa2 and Shuji Handa3; 1Institute for Materials Research, Tohoku University, Sendai, Japan; 2Nanomechanics, Tohoku University, Sendai, Japan.

Effect of heat treatments (aging or annealing) on microstructures was investigated for rapidly solidified ribbons of near-stoichiometric TiCo. In as-cast ribbons, it was observed by TEM that a lamellar structure was developed and its crystal structure had been already B2-ordered, while a small amount of second phase was finely precipitated in grains and along grain boundaries. Some grains were dislocation-free but others contained a certain amount of curved or helical dislocations and loops having the Burgers vector parallel to <100>. There was no significant change in the microstructure of the ribbons aged at 573 K for 100 h. In the ribbons annealed at 573 K for 24 h, grain growth seemed to occur slightly. In addition, the dislocation density in the annealed ribbons was obviously higher than those in the as-cast and aged ribbons. It is considered that the increase of the dislocation density in the annealed ribbons would result from the condensation and/or absorption of supersaturated thermal vacancies. Therefore, the observation results indicate that a large amount of supersaturated thermal vacancies were retained in the TiCo ribbons by the rapid solidification. However, in DSC measurements, any peak did not appear for the TiCo ribbons, whereas relaxation peaks stemming from the condensation of supersaturated thermal vacancies have been often observed in the DSC curves of B2-type intermetallics such as FeAl and NiAl. Based on the obtained results, vacancy condensation behavior in B2-type TiCo is discussed.

10:15 AM S1.6  
Complex Intermetallic Compounds: Defects, Disorder, Details, W. Sprengel1, F. Bauer2, K. Sato3, X. Y. Zhang4 and Hans-Eckhard Schaefer5; 1Institute of Theoretical and Applied Physics, Stuttgart University, Stuttgart, Germany; 2Physical Metallurgy, Technical University Darmstadt, Darmstadt, Germany; 3National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan; 4Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao, China.

A short overview will be given on the thermodynamics of the formation of thermal defects in intermetallic aluminides. We focus on thermal vacancies [1] studied by the specific techniques of positron annihilation [2] and time-differential diffuse scattering [3] and discuss the results together with self-diffusion data [4]. We then demonstrate that these techniques can be employed for studying vacancies in compound semiconductors specifically [5]. Furthermore, structural order-disorder phase transitions can be investigated from an atomistic point of view [6] by making use of positron annihilation as shown in the exemplary case of Al-Ni Co quasicrystals. A few examples for the application of intermetallics will be given in the introductory part. [1] R. Wiesmann et al., PRL 75, 97 (1995); [2] X. Y. Zhang et al., Intermetallics 7, 277 (1999); [3] A. A. Rempel et al., PRL 89, 185501 (2002); [4] K. Sato et al., PRL 92, 127403 (2004).

10:45 AM S1.7  
Strengthening of Iron Aluminide Alloys for High-Temperature Applications, Andre Schneider, Martin Paul, Frank Steen and Gerhard Sauthoff; Max Planck-Institut for
An overview of materials developments of ferritic and Fe3Al-based iron aluminum alloys with strengthening precipitate phases is given in view of possible high-temperature applications. A development of high-temperature alloys for structural applications is to be focused on optimization of strength, creep and corrosion resistance at high temperatures and sufficient ductility at lower temperatures. This is discussed with respect to recent studies and ongoing work on Fe-Al-based alloys with strengthening precipitates, such as α-carbides Fe3Al2C in Fe3Al, MC carbides in ferritic steels, and metastable-phase Nial. The following alloy systems have been investigated: Fe-Al-C, Fe-Al-Ti, Fe-Al-Ta, Fe-Al-Ti-Nb, Fe-Ni-Al-Cr, and Fe-Al-M-C (M=Ti, V, Nb, Ta). The investigations have been focused on microstructure, constitution, mechanical properties, and high-temperature corrosion behavior of such Fe-Al-based alloys with Al contents ranging from 10 to 30 at. %, and problems and perspectives are outlined.

11:15 AM S1.8
Development of High Temperature Creep Resistance in Fe3Al Alloys, David G. Morris1, Maria A. Munoz-Morris1 and Carmen Bautin2. 1Physical Metallurgy, CENIM, CSIC, Madrid, Spain; 2Instituto de Ceramica y Vidrio, CSIC, Madrid, Spain.

Most of the studies aimed at the development of creep-resisting Fe-Al intermetallics have been oriented at application temperatures of the order of 500-650°C, where these materials may compete with conventional stainless steels. The Fe-Al intermetallics are, however, particularly excellent in their oxidation and corrosion resistance at temperatures of order of 1000°C, where Chromium-Nickel steels are no longer able to withstand the aggressive environments. This presentation is part of a study aimed at the development of good creep resistance at such high temperatures. Studies of a variety of cast Fe3Al-based alloys will be reported, which are strengthened by solution or precipitate dispersoid-forming alloying additions. The alloys studied showed good strength from room temperature to about 500°C, but thereafter strength falls rapidly as thermally-activated deformation processes become operative. Solution additions are capable of producing good low temperature strength, but do not contribute significantly to creep strength at very high temperatures (above 700°C). Precipitation hardening has been examined in Nb-containing alloys, where Fe23AlLaves precipitates form at intermediate temperatures. These materials show good strength up to about 700°C, but at higher temperatures the fine precipitates coarsen excessively. Strengthening in this intermediate temperature range varies depending on whether the solute is precipitated prior to high temperature testing or concurrent with this. Studies with a variety of stable carbide and boride additions show relatively poor strengthening by the coarsely-distributed dispersions, but excellent thermal stability and good strength retention to very low strain rates. Alloying and structural parameters important for obtaining good high temperature creep strength in cast Fe3Al alloys will be discussed. Aspects such as segregation during solidification, as well as thermal stability of the structures obtained, contribute to controlling the strength of the materials as the mechanisms of creep deformation themselves.

11:30 AM S1.9
Pseudo-Elasticity of D03 Ordered Monocrystalline Fe3Al, Saurabh Kahan1, Hongbin Bei1, Donald W. Brown2 and Easo P. George1,2, 1Department of Materials Science and Engineering, University of Tennessee, Knoxville, Knoxville, Tennessee; 2Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; 3Los Alamos National Laboratory, Los Alamos, New Mexico.

Single crystals of Fe3Al were grown in an optical floating zone furnace and their pseudo-elastic behavior investigated by tension and compression experiments. The stress-strain behavior was studied as a function of orientation, strain rate and environment. The maximum recoverable strain was found to depend on strain rate. In situ electron backscatter diffraction as well as neutron diffraction were performed as a function of temperature and strain amplitude. The mechanism of deformation and recovery. * Research sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC, which manages and operates the use of the Los Alamos Neutron Science Center (LANSCE) at the Los Alamos National Laboratory. This facility is funded by the US Department of Energy under Contract W-7405-ENG-36.

11:45 AM S1.10
Effect of Alloying Elements (Ga, Ge, Si) on Pseudoelasticity in Fe3Al Single Crystals, Hirokuya Y. Yasuda1, Takashi Kase2, and Yukichi Umakoshi3, 1Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, Ibaraki, Japan; 2Materials Science and Engineering, Osaka University, Suita, Japan.

Pseudoelasticity in Fe3Al single crystals doped with a small amount of Ga, Si and Ge was investigated focusing on the antiphase boundary (APB) energy and the ordered domain structure. Single crystals of Fe-23at%Al and Fe-21at%Al-2at%X (X=Ga, Si, Ge) were grown by a floating zone method. In Fe-23at%Al single crystals, superplastic dislocations with Burgers vector (b) of ½<111> moved dragging APB during loading, while APB pulled back the dislocations during unloading. This resulted in giant pseudoelasticity regardless of martensitic transformation and the recoverable strain was about 5%. Ga addition was found to be effective in increasing the recovery strain compared with Fe-23at%Al. In contrast, both Si and Ge additions increased the amount of shape recovery. Stress at which the shape recovery started, was increased by Ga, Si and Ge additions. This means the APB energy increased by the additions, since the surface tension of APB pulling back the superpartials increases with increasing the energy. Ordered domains with displacement vector (R) of 1/3<112> in Fe-23at%Al and Fe-3Ga-Al-Ga alloys were observed to be small, less than 100nm. In contrast, Si and Ge additions increased the domain size to more than 500nm. The domain boundaries with ½<111>b played an important role in the individual motion of the superpartials with ½<112>b. Therefore, the fine domain structure increased the APB energy following the increase in the superpartial and kept the domain size small, resulting in the increase of recovery strain.
superkink motion and lead to a change in deformation mechanism for Ni3Al. To this end, the thermomechanical behavior of Ni3Al thin films was characterized using transmission electron microscopy. Based on stress-temperature data for the apparent onset of plasticity during repeated thermal cycling, the yield stress was seen to clearly decrease (from 1300 to 500 MPa) with increasing temperature (from 170 to 370°C). This hysteresis in yield stress was found by Kruml et al. for HCP-doped single crystal Ni3Al (300°C). The existence of such a peak in the current study suggests that a YSA could indeed exist in the thicker films (1 μm and higher). These apparently contradictory results, which nonetheless indicate the lack of a YSA for Ni3Al films 2 μm and thinner, will be discussed. Additionally, the effect of the thin film geometry on thermomechanical behavior will be presented and compared to that of other thin film systems.


Ni3Al with the L12 structure has been used as a main component of Ni-based superalloys because of an anomalous increase in yield stress at high temperatures. Control of duplex structure composed of ordered γ' and γ phases is important to improve mechanical properties of the superalloys. Since observation of small precipitates and the interaction between the precipitates and dislocations is very difficult in strongly deformed superalloys, transmission electron microscopy and magnetic technique was applied for it. γ phase shows ferromagnetism. Therefore, changes in size and shape of γ precipitates were observed in aged and cyclically deformed Ni3(Al,Ti) single crystals using magnetic technique and deformation mechanism of the single crystals was examined. Single crystals were grown from the arc melted Ni-18at%Al-4at%Ti ingots by a floating zone method and were homogenized at 1423K for 168h. After solution treatment at 1423K, γ phase was precipitated at 1735K. Specimens with a loading rate of 100 MPa/s for fatigue test were used and the plastic-strain-controlled fatigue test was performed at Δε = 0.2% at room temperature. Magnetization process of aged and fatigued specimens was measured by a vibrating sample magnetometer at temperatures between 77 and 273K. Spontaneous magnetization spontaneously increased with increasing volume fraction and size of γ precipitates during aging, while coercive force rapidly increased after a gradual increase. Change in the coercive force exhibits that γ precipitates change from spherical to plate-like shape. Spontaneous magnetization of specimens aged for 3h or 100h rapidly decreased with increasing number of cycles. The decrease in spontaneous magnetization is in size of γ precipitates due to cutting by dislocations during fatigue deformation because small γ precipitates with nano scale show superparamagnetism. Changes in size, shape and their distribution of γ precipitates in Ni3(Al,Ti) single crystals during fatigue will be quantitatively analyzed from magnetic properties and the deformation mechanism will be discussed.

2:30 PM S2.4 Shearing Mechanisms of the γ' Phase in Superalloys at Intermediate Temperatures. Michael John Mills1, Gopal B. Viswanathan1, Peter Saros1, Raymond Unocic1 and Deborah Whitis1; 1Materials Science and Engineering, The Ohio State University, Columbus, Ohio. 2Materials and Processes Engineering Department (MPEP), GE Aircraft Engines, Cincinnati, Ohio.

The deformation mechanisms in single phase Ni3Al and related L12 intermetallics have been extensively studied. One motivation for these studies has been the need to understand the microstructural changes that occur to the strengthening phase in the technologically important superalloys. In fact, the principal γ' shearing mechanism in superalloys at lower temperatures is γ' shearing that happens in single phase superalloys prior to the onset of plastic deformation. At intermediate temperatures (in the range from 873-1373K), it is now abundantly clear that the γ' shearing mechanisms in superalloys are quite different than those in single phase superalloys. Studies of substructures following creep of the superalloys Rene 88DT and M533 in the superplastic condition with bimodal γ' size distributions reveals at least two distinguish shearing processes. In coarser γ' microstructures, and higher stress levels, the γ' particles overcome shear bands and a nucleation of superplastic extrinsic stacking faults, with isolated Σ3<110> dislocations in the matrix. In finer microstructures and lower stresses, deformation twinning is the dominant mechanism. Our TEM observations support a twinning process similar to that proposed recently by Kolbe in which ½<112> Shockley partials cooperatively shear both matrix and γ' particles. Atomic rearrangements in the wake of these twinning partials enable the γ' particles to retain the L12 structure, and it is postulated that these atomic rearrangements correspond to the rate limiting, thermally activated process under these conditions. The implications of these results for modeling creep in superalloys will be discussed.

2:45 PM S2.5 Microstructures and Mechanical Properties in Ni, Si, Ni3Ta, Ti-Ni3Nb-Based Multi-Intermetallic Alloys. Takayuki Takaagi, Koji Ohira and Yasuyuki Kanou, Department of Metallurgy and Materials Science, Osaka Prefecture University, Sakai, Osaka, Japan.

The phase relation, microstructures, high-temperature deformation, and oxidation and corrosion behavior of intermetallic alloys based on Ni3Si-Ni3Ta, Ni3Ti, Ni3Nb pseudo-ternary alloy system were investigated. As the constituent intermetallic phases, L12(Ni3Si), DO22(Ni3Ti), DO3(Ni3Ta), and DO19(Ni3Nb) were identified and their phase fields were discussed based on the electrical and geometrical factors of constituent atoms. Among four intermetallic phases, five kinds of two-phase relations and two kinds of three-phase relations were found to exist. The prepared alloys exhibited widely different microstructures, depending on the number and kinds of the constituent intermetallic phases. Three-phase microstructures composed of L12(Ni3Si), DO22(Ni3Ti) and DO19(Ni3Nb) showed superior tensile strength and ductility in a wide range of temperatures. Also, the mechanical and chemical properties obtained in the present alloy system were compared with those reported in Ni3Al-Ni3Ta-Ni3Nb and Ni3Al-Ni3Ti-Ni3V-based multi-intermetallic alloys.

3:15 PM S2.6 Atomicistic Modeling of Advanced Intermetallic Alloys. Yuri Mishin, School of Computational Sciences, George Mason University, Fairfax, Virginia.

This talk gives an overview of the recent work on atomistic computer modeling of intermetallic compounds of the Ni-Al and Ti-Al systems. Atomic interactions in these systems are modeled by semi-empirical potentials that fit both experimental and first-principles data. The methodology includes a large variety of techniques ranging from harmonic lattice dynamics to molecular dynamics and Monte Carlo simulations. The properties studied include lattice characteristics (elastic constants, phonons, thermal expansion), point-defect properties, atomic diffusion, generalized stacking faults, dislocations, surfaces, grain boundaries, interphase boundaries, and phase diagram calculations. The talk emphasizes the recent progress in the understanding of diffusion mechanisms in NiAl and Ni3Al, calculation of stacking fault energies in NiAl, in relation to dislocation behavior, and calculation of gamma/gamma' interface boundaries in Ni-Al alloys. Preliminary simulation results for nucleation of gamma' particles in the gamma matrix will be discussed.


The microscopic mechanisms governing the deformation and fracture behavior and anomalous mechanical response of intermetallic alloys are still far from being well understood and remain a challenge for theoretical explanation. Progress in this complex area requires understanding such key phenomena as dislocation structure and mobility: while they have been characterized by mesoscopic length and energy scales, they are determined on the microscopic level by the electronic structure. To reach the goal of connecting the microscopic and mesoscopic scales in the analysis of dislocation structure and mobility, we employ a semi-empirical model of the Peierls-Nabarro behavior based on highly accurate first-principles calculations of the cleavage/shear energies and the modified semi-discrete 2D Peierls-Nabarro (PN) model with an ab-initio parameterization of the restoring forces. We present the results of fundamental comparative studies of the dislocation properties and the mechanical behavior for a new class of intermetallic alloys based on platinum group metals (PGM) which are being developed for ultra-high-temperature (UHT) applications, which include Ir3X, Rh3X (X = Ti, Zr, Hf, V, Nb, Ta) and Pt3X (X = Al, In, Ga), possess very high melting temperatures and superior environmental properties. The two-phase PGM-based superalloys are considered as a replacement for Ni-based superalloys for ultra-high-temperature further progress in the development of these alloys relies on understanding the fundamental factors that control their mechanical properties. Using our combined approach, we analyze the dislocation properties, structure and mobilities in single-phase intermetallics and, based on our analysis, provide an explanation of
The observed unusual features of the mechanical behavior of these alloys, and provide predictions of their high temperature yield stress behavior. We have investigated the connection of the mechanical behavior of PGM alloys with features of their electronic structure, and demonstrate that the peculiarities of their behavior are directly connected with structural stabilities, namely L12 → D03 for Ir(Re)1-x, X = O, Fe for Pt3X. Supported by the AFOSR (grant No. F49620-01-1-0106).

1:00 PM S2.8

A Bond-Order Potential Incorporating Analytic Screening Functions for the Molybdenum Silicides, Marc J. Cavallig1, Matous Mrovec2, Duc Nguyen-Manh3, David G. Pettifor4 and Vaclav Vitek2, 1Department of Materials Science and Engineering, University of Pennsylvania, Philadelphia, Pennsylvania; 2Fraunhofer IWM, Abingdon, United Kingdom; 3Department of Materials, University of Oxford, Oxford, United Kingdom.

The intermetallic compound MoSi2, which adopts the C114 crystal structure, and related alloys exhibit an excellent corrosion resistance at high temperatures but tend to be brittle at room and even relatively high temperatures. The limited ductility of MoSi2 in ambient conditions along with the anomalous temperature dependence of the CRSS of the (110)C114, (011)C100 and (010)C100 slip systems and deviation from Schmidt law behavior of the (013)C331 slip system can all be attributed to complex dislocation core structures. We have therefore developed a Bond-Order Potential (BOP) for MoSi2 for use in the atomistic simulation of dislocations and other extended defects. BOPs are a real-space, O(n), two-center orthogonal tight-binding formalism naturally able to describe systems which exhibit mixed metallic and covalent bonding, such as MoSi2. In this development novel analytic screening functions have been adopted to properly describe the environmental dependence of bond integrals. We have carefully assessed the transferability of other crystal structures and stoichiometries, notably C40, C49 and C54 MoSi2, A15 and D03 Mo5Si4 and D8s Mo8Si13 by comparing with ab-initio structural optimizations. Furthermore, we have studied the ideal tensile strength of C114 MoSi2 and compared the predictions with abinitio calculations. Finally, we report γ-surfaces for the (110), (011), (010) and (013) planes and use these to guide subsequent atomistic simulations of dislocation core structures. This research was supported by the U.S. Department of Energy, BES Grant no. DE-PG02-98ER45702 and the United Kingdom EPSRC.

Rodriguez

Role of Microstructure in Promoting Fracture and Fatigue Resistance in Mo-Si-B Alloys, Jamie Kruzic, Joachim Schneibel, David G. Pettifor, Peter Sarosi, Hanish L. Fraser and Michael J. Mills, Materials Science and Engineering, The Ohio State University, Columbus, Ohio.

Mo-Si-B “in-situ” metal matrix composites consist of Nb- and Nb5Si3 intermetallic phases in a body centered cubic Nb solid solution, and show promising potential for elevated temperature structural applications. Cr and Ti have been shown to increase the oxidation resistance at elevated temperatures compared to the binary Nb-Si system. In this study, the LENS<sup>TM</sup> (Laser Engineered Net Shaping) process is being implemented to construct the Nb-Ti-Cr-Si alloy system from elemental powder blends. Fast cooling rates associated with LENS<sup>TM</sup> processing yield a reduction in microstructural scale over conventional alloy processes such as directional solidification. Other advantages of LENS<sup>TM</sup> processing include the ability to produce near net shaped components with graded compositions as well as control of phase volume fractions resulting from the negative enthalpy of mixing associated with the silicide phases. Processing parameters can also be varied, resulting in distinct microstructural differences. Deposits were made with varying compositions of Nb, Ti, Cr and Si. The as-deposited as well as heat treated microstructures were examined using SEM and TEM techniques. The influence of composition and subsequent heat treatment on microstructure and mechanical properties such as microhardness and microtensile testing will also be discussed.

SESSION S3: Functional Intermetallics I

Chairs: T. J. Balk and G. Dulin
Tuesday Morning, November 30, 2004
Room 208 (Hynes)

8:30 AM S3.1

Microstructure and Mechanical Properties of Shape Memory Thin Films Formed by Sputtering, Akira Ishida, National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

Shape memory thin films formed by sputtering have been attracting great interest as powerful actuators in microelectromechanical systems (MEMS) such as microvalves, microfluid pumps, and micromotors. Their shape-memory properties were evaluated quantitatively with a small tensile tester and it was established that sputter-deposited thin films have mechanical properties superior to those of bulk alloys. The improvements in tensile strength, elongation and heat treatment result from the crystallization of amorphous films. While Ti-rich Ti-Ni bulk alloys show only blocky Ti<sub>3</sub>Ni<sub>2</sub> precipitates along the grain boundaries irrespective of heat-treatment, thin films show a peculiar structure evolution of (1)GP zones, (2)GP zones with Ti<sub>2</sub>Ni precipitates within the grains, (3)Ti<sub>2</sub>Ni<sub>1</sub> precipitates within the grains, and (4)Ti<sub>2</sub>Ni<sub>1</sub> precipitates along the boundaries with increasing heat-treatment time. These findings indicate that thin films might show an excellent combination of a large transformation strain and a small plastic strain. According to the high-resolution transmission electron microscopy observation of a deformed sample, the coherent interface of GP zones is effective to prevent dislocation movement, but allow twinning to pass through the GP zones. Another advantage is the fine structure was observed in ternary alloy films. Extremely small grain size in Ti-Ni-Zr.
Mechanical Properties of TiNiPt melt spun ribbons were investigated where the heat treatment temperature was systematically changed from 745K to 783K. A hot-forged bulk TiNiPt material with the similar chemical composition was also tested as a comparison. It was revealed by differential scanning calorimetry (DSC) that martensitic transformation temperature of the TiNiPt melt-spun ribbons previously measured as a function of the hot-forged bulk material, and that the martensitic transformation temperature decreases with increasing heat treatment temperature. It was also revealed by XRD theta-2theta analysis that the apparent phase at room temperature for TiNiPt melt-spun ribbons was Fe3B-type martensite for 125-nm-thick ribbons instead of B2 parent phase for the hot-forged bulk material. By mechanical tests at temperatures from room temperature to 373K, superelasticity around 5% was confirmed for the melt-spun ribbons when the test temperature is above the reverse martensitic transformation finish temperature. The stress for inducing martensitic transformation is discussed in terms of heat treatment condition. Microstructures of the TiNiPt melt-spun ribbons observed by transmission electron microscopy are also presented.

Laser Annealing of Shape Memory Alloy Thin Films: Microstructure Investigation. Xi Wang1, Joost J. Vlassak1, Fadila Khelfou2 and Yves Bellouard3; 1Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; 2Dept. of Physics, University of Antwerp, Antwerp, Belgium.

For a decade, shape memory alloys thin films have been considered for larger ones with a diameter of 300nm. From these observations the maximum strain is not localised at the matrix-precipitate interface but at a distance from it. The results are compared to density functional theory (DFT) calculations. Here, we present a microstructure investigation of Ti-rich Ni-Ti laser annealed thin films. Amorphous, 1.5 micron thick films were deposited on fused quartz substrates by means of UHV sputtering. Optical microscopy and X-ray diffraction were used to determine whether the films were crystallized and to establish the useful parameter space in terms of power density and scan speed. The microstructure of the crystallized regions of the films was characterized using transmission electron microscopy (TEM) in order to investigate the effects of power density and scan speed.

Factors to Control the Martensitic Transformation Temperature of TiNi Shape Memory Alloy Containing Ternary Elements. Hideki Hosoda1, Kenji Wakahama1, Shuichi Miyazaki2 and Kanzyu Insue2; 1Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Japan; 2Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki, Japan.

Ternary alloying elements are generally added to TiNi shape memory thin films such as a microgripper, a microconveyor, and a microrobot are demonstrated. 5%

Mechanical Properties of TiNi containing 10mol% melt-spun ribbons were investigated where the heat treatment temperature was systematically changed from 473K to 773K. A hot-forged bulk TiNiPt material with the similar chemical composition was also tested as a comparison. It was revealed by differential scanning calorimetry (DSC) that martensitic transformation temperature of the TiNiPt melt-spun ribbons was 69K higher than that of the hot-forged bulk material, and that the martensitic transformation temperature decreases with increasing heat treatment temperature. It was also revealed by XRD theta-2theta analysis that the apparent phase at room temperature for TiNiPt melt-spun ribbons was Fe3B-type martensite for 125-nm-thick ribbons instead of B2 parent phase for the hot-forged bulk material. By mechanical tests at temperatures from room temperature to 373K, superelasticity around 5% was confirmed for the melt-spun ribbons when the test temperature is above the reverse martensitic transformation finish temperature. The stress for inducing martensitic transformation is discussed in terms of heat treatment condition. Microstructures of the TiNiPt melt-spun ribbons observed by transmission electron microscopy are also presented.

Laser Annealing of Shape Memory Alloy Thin Films: Microstructure Investigation. Xi Wang1, Joost J. Vlassak1, Fadila Khelfou2 and Yves Bellouard3; 1Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; 2Dept. of Physics, University of Antwerp, Antwerp, Belgium.

For a decade, shape memory alloys thin films have been considered for larger ones with a diameter of 300nm. From these observations the maximum strain is not localised at the matrix-precipitate interface but at a distance from it. The results are compared to density functional theory (DFT) calculations. Here, we present a microstructure investigation of Ti-rich Ni-Ti laser annealed thin films. Amorphous, 1.5 micron thick films were deposited on fused quartz substrates by means of UHV sputtering. Optical microscopy and X-ray diffraction were used to determine whether the films were crystallized and to establish the useful parameter space in terms of power density and scan speed. The microstructure of the crystallized regions of the films was characterized using transmission electron microscopy (TEM) in order to investigate the effects of power density and scan speed.

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phase transition was expanded and the thermal hysteresis between the high temperature phase and the low temperature phase was narrow, in comparison with that of non-treated. X-ray diffraction measurements of the compounds, a trial actuator consisting of a FePd wire and conventional inconel wire has been constructed. The actuator has properly functioned with heat cycles.

11:15 AM S3.9

The L1_0-ordered intermetallic phase FePd can be established in the vicinity of the equiatomic composition for temperatures below about 920K and is a member of a class of uniaxial ferromagnetic intermetallics that exhibit large magnetocrystalline anisotropy and includes also FePt, MnAl and CoPt. Permanent magnet alloys based on these anisotropic ferromagnetic intermetallics are currently of interest for novel and advanced applications in the information technology sector. The technologically relevant properties depend very strongly on the morphology and scale of the microstructure and the defect structures produced during solid-state processing. Here the equiatomic FePd alloys are used as model systems to investigate some aspects of the processing-structure-property relationships in this class of L1_0-ordered ferromagnets during the latter stages of order annealing after cold-deformation. Depending on the details of the processing parameters employed during the combations of cold-deformation and annealing treatments a number of morphologically different microstructures with relevant average scale (e.g. grain size) ranging from the nanometer to the conventional micron range can be established. Combinations of property measurements using a vibrating sample magnetometer and microstructural studies by scanning and transmission electron microscopy, including dynamic in-situ heating observations, have been performed on the transformation processed FePd with equixed morphology during annealing. Mechanisms for defect-interactions and defect genesis during the grain growth stage have been identified. Activation energies for the dominant elementary diffusional processes during grain coarsening have been determined. A Hall-Petch-type relationship has been established between the coercivity and the average grain size of the equixed L1_0-FePd intermetallics. Implications for bulk-processing of FePd-based and other L1_0-ordered intermetallics are discussed. Support from the National Science Foundation (DMR) is gratefully acknowledged.

11:30 AM S3.10
Comparison of Temperature Driven Ordering and its Influence on the Magnetic Behaviour in Bulk and Thin Film L1_0 Ordered FePd. Wolfgang Pfeiler1, Chassak Isro2, Wolfgang Puech1, Peter Franz Rogl2, William A. Soffa3, Rafał Kozubski4, Guy Schmerber5 and Veronique Pierron-Bohnes6; 1Institut für Physik, University of Vienna, Vienna, Austria; 2Physikalische Chemie, University of Vienna, Vienna, Austria; 3Materials Science and Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; 4M. Smoluchowski Institute of Physics, Jagiellonian University, Cracow, Poland; 5Institut de Physique de la Matière Condensée, ICPEMC-CNRS-ULP, Strasbourg, France.

L1_0-ordered intermetallics are promising high density magnetic recording materials. The magnetic anisotropy is due to the 90° tilt of the c-axis of the tetragonal structure as the easy axis of magnetization. We present preliminary results on FePd films (thickness 50 nm) sputtered on Si and epitaxially co-deposited on MgO substrates. The diffraction patterns from bulk material (thin foil thickness <10µm). Results from X-ray diffraction, electrical resistivity (in-plane) and magnetization (in-plane and out-of-plane) are presented. The cold-rolled thin foil is initially ordered and orders during a subsequent isochronal annealing procedure which is monitored by X-ray diffraction, electrical resistivity and magnetization behaviour. FePd film as-sputtered on Si substrate is fine grained (grain size <100nm) and the film order L1_0 increases upon annealing. During annealing the grain size increases (final grain size 30nm) and the film orders L1_0 with an increase in all variants of ordered domains. FePd film epitaxially grown on MgO(001) is already highly ordered in the as-prepared state and shows a nearly single-crystalline structure (c-axis perpendicular to sample surface). During isochronal annealing the degree of order further increases and above 650K starts to decrease again. The magnetization curves reflect the initially high magnetic anisotropy (easy axis c-axis perpendicular to film surface). Starting with annealing at 500K an ever increasing number of ordered domains re-orient their c-axis into the film plane. This surprising effect is in correspondence with recent Monte-Carlo simulations of ordering kinetics in L1_0-ordered nanolayers.

SESSION S4: Functional Intermetallics II
Chairs: E. P. George and Y. Ummkoshi
Tuesday Afternoon, November 30, 2004
Room 208 (Hynes)

1:30 PM S4.1
Sub-nano and Nano-Structures of Hydrides of LnAl and its related Intermetallics. Hsiao Akiba, Koushi Sakaki and Yumiko Nakamura; Energy Electronics Rl, AIST, Tsukuba, Ibaraki, Japan.

Sub-nano and nano-structures of intermetallics such as LnAl and its related alloys were studied by in-situ X-ray and neutron diffraction methods. From the profile shape analysis, changes in lattice strain and crystalline size during hydrogenation/dehydrogenation were estimated, while the crystal (sub-nano) structures were refined by the Rietveld method using diffraction patterns. We also measured defects induced during the propagation of a shock wave. Recent research has indicated that the strain-induced paramagnetic to ferromagnetism transition in FeAl arises mostly from the generation of APB tubes, where Fe atoms can have 23 like neighbors. The resulting saturation magnetization, $M_S$, is related to the degree of deformation. A quantitative model will be presented that describes the effects of both of these parameters in terms of the local environment theory applied to the tubes in APB tubes. The behavior of both ternary FeAl-based alloys and of the L1_0-structured intermetallic compound Fe$_2$AlMn will be explained by considering the site preferences of ternary atoms on the Fe and Al sublattices. TEM observations of APB tubes in highly-strained single crystals of Fe$_2$Al and Fe$_2$AlMn will be presented, and the annealing out of APB tubes and the associated activation energy will be discussed. The effects of plastic strain on the magnetic properties of some strongly-ordered L1_0 intermetallics are also outlined. Research sponsored by NSF grant DMR 9973977 and NIST grant 60NANB2D0120.

2:00 PM S4.2

Ni$_3$Al intermetallic compound has attractive high temperature properties, such as high strength and good oxidation/corrosion resistance, and is possible to be used for high temperature chemical processing and manufacture. Until now, the catalytic properties of Ni$_3$Al were rarely investigated since the leaching of aluminum from Ni$_3$Al is difficult compared with those of NiAl and Ni$_2$Al$_3$. In the present work, the surface and catalytic properties of Ni$_3$Al were characterized by hydrogen production reactions. X-ray diffraction and Raman scattering were the characteristic methods of Ni$_3$Al without allantoi leaching showed a limited catalytic activity for the methanol decomposition (CH$_3$OH → H$_2$+CO), and no activity for the steam reforming of methanol (CH$_3$OH +H$_2$O → H$_2$+CO$_2$)
in the temperature range of 513-635 K. The activity of Ni3Al for the methanol decomposition was improved significantly by alkali leaching. The alkali leached sample showed a better performance for the methanol decomposition than nickel catalyst above 573 K. Furthermore, Ni3Al catalyst suppressed the formation of methane, i.e. it showed higher selectivity for the methanol decomposition reaction than nickel catalyst. The results suggests that Ni3Al is a potential catalyst for hydrogen production reaction.

2:15 PM S4.3
Ultra-Strong and Ductile Hypereutectic Ti-based Bulk Alloys, Dimitri V. Louguine1, Larissa V. Louguina2, Hatemi Kato3 and Akihisa Inoue1,1 Institute for Materials Research, Tohoku University, Sendai, Japan; 2Research and Development Project, CREST, Japan Science and Technology Agency, Sendai, Japan.

Typical commercial Ti-based alloys have an ultimate tensile strength slightly exceeding 1 GPa which taking into account relatively low density of Ti (4.5 Mg/m³) implies their high strength/density ratio. Moreover, Ti-based alloys exhibit surprisingly high ductility. For example, mould casting technique allows to produce Ti-based bulk glassy alloys having a high strength of about 2 GPa which, however, suffer from the lack of ductility. In the present work we study a number of binary Ti-TM (TM other transition metals) and ternary Ti-Fe-TM as well as Ti-Fe-Re (RE-rear-earth) alloys and report on the formation of the high-strength and ductile hypereutectic Ti-Fe, Ti-Fe-Cu and Ti-Fe-Nd alloys. The alloys were produced in the form of the arc-melted ingots 20-30 mm diameter and 10-15 mm height. The structure of the high-strength and ductile hypereutectic alloys studied by X-ray diffractometry and scanning electron microscopy is found to consist of a primary cubic or a metastable bcc (TiFe-phase or a solid solution on its base) and a dispersed eutectic consisting of Pm3m intermetallic compound + BCC Im3m beta-Fe supersaturated solid solution phase. The mechanical properties of the samples could be improved by suitable heat treatments tested by an Instron-type machine. The Ti65Fe35 alloy having hypereutectic structure consisting of the primary TiFe phase and submicron-size eutectic (TiFe + beta-Fe) structure showed excellent mechanical properties: a Young’s modulus of 140 GPa, a high compressive strength of 2.2 GPa, a 0.2% yield strength of 1.8 GPa and 6.7% ductility. The addition of Cu improves ductility of the alloy, while the addition of Nd improves both strength and ductility. Ni and Mn additions cause embrittlement owing to the formation of the alternative intermetallic compounds. The mechanical properties of the arc-melted Ti70Fe15Cu15 and Ti62Fe33Nd5 alloys are: a Young’s modulus of 120 and 160 GPa, a high mechanical strength of 1.78 and 2.49 GPa, a 0.2% yield strength of 1.53 and 1.5 GPa as well as 8 and 10% ductility, respectively. The round-shaped Pm3m intermetallic TiFe (or TiFe(Cu)) phase, the supersaturated Im3m beta-Fe solid solution and the disperse eutectic structure obtained result in a high strength of the Ti-Fe, Ti-Fe-Cu and Ti-Fe-Nd alloys which in addition to that exhibit significantly better ductility compared to that of bulk glassy alloys because the ductile beta-Fe solid solution phase enables plastic deformation of the sample. The deformation behaviour and the factors that affects the Ti-based alloys are studied in details. The reasons for the high strength and good ductility of the hypereutectic alloys are discussed. The Ti-based alloys are characterized by low cost of the alloying elements and do not require an additional injection mould casting procedure.

2:30 PM S4.4
In-situ Observation of Surface Relief Formation and Dissipation during Order-Disorder Transition of Equi-atomic CuAu alloy using Laser Scanning Confocal Spectroscopy, Seiji Miura, Hisayuki Okuno, Kenji Ohkubo and Tetsuo Mohri; Mat. Sci. and Eng., Hokkaido Univ., Sapporo, Japan.

In-situ observation of the surface relief formation and dissipation associated with the twinning during the order-disorder transitions among CuAu-I (L10), CuAu-II (PAP) and disordered fcc phases was conducted using Laser Scanning Confocal Spectroscopy equipped with a image furnace. Retro effect was confirmed in poly-crystalline samples, however no evidence was found in single crystal samples. Also it is observed in poly-crystalline samples that the disordering temperature is affected by the change of relieves is different from grain to grain, which is relevant to grain boundary crackings during transitions. The crystallographic orientation relationship among grains investigated by FESEM/EBSD was taken into account to explain the phenomena.

3:15 PM S4.5

A brief overview of the current state of thermoelectric materials research will be presented. Thermoelectric devices can be used for the conversion of heat into electricity or for solid-state refrigeration where the only moving parts are electrons and holes. The poor efficiency of the thermoelectric devices limits their use to niche applications such as the spot cooling of electronics or the generation of electrical power for NASA’s deep space probes. To improve the efficiency of thermoelectric devices requires developing materials with larger values of the Seebeck coefficient and the figure of merit Z = S² * |kappa| / T, where S is the Seebeck coefficient, kappa is the thermal conductivity and T is the absolute temperature. A variety of new approaches and materials have been proposed over the past 15 years and many of these ideas will be discussed. Classes of materials that will be covered include: mixed-valence intermetallic compounds, Kondo insulators, skutterudites, clathrates, half-heusler alloys, thin film superlattices structures and with and without quantum confinement, nanowires, nanotubes, and bulk materials with nanocrystal inclusions.

3:45 PM S4.6
Thermoelectric Performance of Ru2Si3 and Re-doped Ru1.95-Re0.05Si3 Alloys, Benjamin Andrew Simkin and Haruyuki Inui; Materials Science and Engineering, Kyoto University, Kyoto, Kyoto, Japan.

Ruthenium sesquisilicide (Ru2Si3) has received extensive attention in recent years as a high-temperature thermoelectric compound due to an attractive combination of large Seebeck coefficients and high thermal conductivities at elevated temperatures. The principal limitation to Ru2Si3 as a thermoelectric has been the poor electrical properties shown by this compound. The poor observed electrical resistivities have been attributed to a number of factors, including the presence of grain boundary contributions to the polycrystalline and single crystal samples. Attempts to improve conductivity through the growth of single crystals has yielded mixed results. A recent study has shown that the high-temperature tetragonal phase TiFe phase has resulted in a significant increase in the electrical resistivity change in Ru2Si3 induced a [100]-[001] 90 degree rotational domain structure in Ru2Si3 that is inherent to the compound, and tends to limit the attainable crystal perfection. However, the [010] direction common to both domain orientations has also been shown to posses superior thermoelectric properties to the [100]/[001] orientation, principally due to the higher thermopower along this orientation. Because this phase change appears to be inherent to the compound and not diffusionally driven it is expected that further enhancements to conductivity will be difficult without alteration of the composition. Initial studies of the compound Re4Si5 have suggested an extensive solubility range for Re in the Ru2Si3 structure, opening the possibility for significant conductivity improvements to Ru2Si3 through substitutional doping of Re for Ru. Arc-melted samples of Ru1.8-Re0.2Si3 show a tendency toward increasing n-type conductivity, with simultaneous improvements in conductivity. Additionally, Re additions appear to cause ordering of the low temperature orthorhombic Ru2Si3 structure into a tetragonal (Ru-Re)Si2 structure with a=22.75Å and c=8.89Å.

4:00 PM S4.7
Mechanical Aspects of Structural Optimization in a Bi-Te Thermoelectric Module for Power Generation, Yujio Nakatani, Reiki Takaku, Takehisa Hino, Takahiko Shinou and Yoshinori Itoh; Power and Industrial Systems R&D Center, Toshiba Corporation, Yokohama, Japan.

We are currently developing a low-cost and high-efficiency Bi-Te thermoelectric module for power generation. This module aims at heat recovery at temperatures of 150 degrees C or less. Though the total amount of waste heat energy below 150 degrees C is considerably large, it has not been utilized because the density of energy is low. In addition, the utilization of such low-density energy has been considered economically uncompetitive. Therefore, our objective is to enhance the thermoelectric efficiency and to improve the cost performance of the module. One of the methods to improve the cost performance is to achieve a higher range, without maintenance, based on the enhanced reliability of the module. Our module consists of the Bi-Te thermoelements and electrodes of Mo and Al formed by atmospheric plasma spraying. In such multilayer coating structures, the thermal stress and/or strain is induced by the differences between the thermal expansion coefficients of each material, and this can decrease the long-term reliability. This paper describes the analytical studies on evaluation and improvement of the interface integrity of the Bi-Te thermoelements and the coatings caused by the thermal strain. To estimate the strain state on the interface, the three dimensional elastic-plastic finite element method (FEM) was performed. The analysis revealed that compressive plastic strain occurred at the coating layer near the interface and this caused tensile residual strain on the interface when cyclic heat was loaded. Then, a statistical optimization analysis based on the FEM and design of experiment (DoE) method was conducted, and the optimum shape and dimensions of the Bi-Te thermoelement and the coating...
thickness required for decreasing the thermal strain were obtained. We have participated in “The Japanese National Project on Development of Advanced Thermoelectric Conversion Systems” supported by the New Energy and Industrial Technology Development Organization (NEDO).

4:15 PM S4.8
Effects of Ga- and In-Doping on the Thermoelectric Properties of Ba-Ge Clathrate Compounds. Norihito L. Okamoto and Haruyuki Inui; Materials Science and Engineering, Kyoto University, Kyoto, Japan.

There has been great interest in inorganic clathrate compounds in hopes of producing more efficient thermoelectric devices because they have very low thermal conductivity and relatively high electronic conductivity. Clathrate compounds possess polyedral cages encapsulating guest atoms. Although the low thermal conductivity in clathrate compounds is believed to result from the rattling motion of the guest atoms within the cages, the relationships between crystal structures and thermoelectric properties have not well known yet. In our research of the thermoelectric properties for the ternary type I clathrate compounds in the Ba-Ge-Ga system, we have found that Ba$_4$Ga$_2$Ge$_6$ exhibits a value of Seebeck coefficient as high as -280 $\mu$V/K and the largest thermoelectric figure of merit in the ternary system. In Ba$_4$Ge$_2$Ga$_6$, Ge and Ga atoms constitute the cage structure and they are randomly distributed in the cage structure. Upon substituting Ga atoms in Ba$_4$Ga$_2$Ge$_6$ by In atoms, the thermoelectric power factor increases while the lattice thermal conductivity decreases, leading to a much higher thermoelectric figure of merit (ZT=1).

4:30 PM S4.9
Magnetic Properties of E$_2$-Base Co$_3$AlC and the Correlation with the Ordering of Carbon Atoms and Vacancies. Yoshinori Kimura, Fu-Gao Wei, Hideyuki Ohtsuka and Yoshinoshi Mishima; 1Materials Science and Engineering, Tokyo Institute of Technology, Yokohama, Japan; 2National Research Institute for Materials Science, Tsukuba, Japan.

The E$_2$ type intermetallic compound Co$_3$AlC is a quite hopeful strengthening for a new class of Co-base heat resistant alloys since its ordered crystal structure is almost the same as that of L1$_2$ Ni$_3$Al. The difference between L1$_2$ and E$_2$ is distinguished by a carbon atom occupying the octahedral interstice at the body center. It is believed that Co$_3$AlC and Ni$_3$AlC form continuous solid solution in Co-Ni-Al-C quaternary system. Our group have reported that the extra ordering of carbon atoms and vacancies is observed in Co$_3$AlC and (Co,Ni)$_3$AlC most likely due to minimizing the elastic strain energy. Half of carbon atoms are replaced by vacancies at every other body center site as if E$_2$ and L1$_2$ were arranged alternatively in every other unit cell. Thus it should be denoted as E$_2$Co$_{0.5}$C$_{0.5}$ although it remains controversial that whether the ordering is long range or short range. We also have reported that Co$_3$AlC phase exhibits weak ferromagnetism while Co$_3$AlC$_{0.1}$ at room temperature of 100 K is unusually high compared with the same small magnetization. In the present work, the relationship between the anti-phase boundary (APB) and the ferromagnetism is evaluated using TEM and VSM for considering the phase stability of Co$_3$AlC with its ordered crystal structure. We believe that the unusual ferromagnetism of E$_2$-base Co$_3$AlC must be correlated with its ordered crystal structure and should be strongly affected by the extra ordering of carbon atoms and vacancies. The anti-phase domain (APD) size, i.e., the total amount area of APB, was controlled by heat treatment conditions. Magnetization measurements at room temperature 4.5 K have revealed that the magnetization behavior is clearly affected by APD size. Moreover, effects of the Ni addition were investigated on the magnetic properties and the phase stability of E$_2$Co$_3$AlC against E$_2$Ni$_3$AlC and/or L1$_2$ Ni$_3$Al. It is quite beneficial to investigate the phase stability of (Co,Ni)$_3$AlC in Co-Ni-Al-C quaternary system for designing attractive heat resistant alloys based on (Co,Ni)$_3$AlC.

4:45 PM S4.10

Sigma-phases are complex tetragonal structures that form in transition metal alloys having the average number of s+d electrons in the interval 0.5–7.4. These phases, which may form during solidification or heat treatment, usually cause embrittlement of the alloys. A well known example is the FeCr sigma-phase, which formation has a deleterious effect on the mechanical properties of stainless steels. Like other sigma-phases, the FeCr and other iron-based sigma-phases possess high degrees of chemical disorder among the five crystallographically inequivalent sublattices. We perform a theoretical study of the thermodynamic properties of several binary sigma-phase alloys (Fe-V, Fe-Cr, and Fe-Mo) using ab initio total energy calculations combined with the compound energy formalism developed in CALPHAD. We show that a proper thermodynamic description of these systems can only be achieved on the basis of spin-polarized calculations (the magnetism of these systems can be described by the distorted Jahn-Teller model). The local spin moments on the iron atoms occupying the high-coordination sites in the sigma-phase structure are found to reach high values of about 2 Bohr magnetons. Using the calculated total energies we were able to reproduce the experimental site occupancies in the iron-based sigma-phases. The calculated electronic structure and thermodynamic properties are discussed and compared with results of previous studies.

S5.1
Effect of Heat Treatment Conditions on Multistage Martensitic Transformation in Aged Ni-rich Ti-Ni Alloys. Minoru Nishida, Toru Hara, Yasuhiro Morizono; Materials Science and Engineering, Kumamoto University, Kumamoto, Japan; 2National Institute of Materials Science, Tsukuba, Japan.

It has been demonstrated with systematic experiments that the appearance and disappearance of multistage martensitic transformation (MMT) in aged Ni-rich Ti-Ni alloys depend on the heat treatment conditions. No multistage transformation occurs when the total oxidation of Ti and Ni and/or the preferential oxidation of Ti is prevented and the purification of heat treatment atmosphere in an evacuated quartz tube is achieved. The heterogeneity in precipitation morphology of Ti3Ni4 phase which is responsible for the multistage transformation can be suppressed with the regulation of heat treatment atmosphere as mentioned above. We have also concluded that the multistage martensitic transformation in aged Ni-rich Ti-Ni alloys is an extrinsic nature, i.e., a kind of artifact during the heat treatment. Effect of alloy composition and specimen size on MMT is also discussed.

S5.2
Application of CSL Model to Deformation Twin Boundary in B2 Type TiNi Compound. Minoru Nishida, Mitsuhiro Matsuda, Yasuhiro Morizono, Towako Fujimoto and Hideharu Nakashima; 1Materials Science and Engineering, Kumamoto University, Kumamoto, Japan; 2Department of Material Sciences, Interdisciplinary Graduate School of Engineering Science, Kyushu University, Fukuoka, Japan.

Near-equiatomic TiNi compound exhibits superior shape memory and superelastic properties. The other peculiar property is anomalous ductility at wide temperature range in comparison to another Ti-rich memory alloy such as NbTi. In this study we have focused on the deformation structure of B2 parent phase around 573 K, since interesting interactions of planer defects have been found. The specimen used is Ti50Ni50Fe2 (Ms = 235 K) in which there is no decomposition of secondary phase during tensile test at elevated temperature and no martensitic transformation during cooling after the test. Serrations are seen in stress-strain curve obtained around 573 K, which corresponds to the formation of various planer defects with twin relation. The dominant interaction defect found in the specimens showing serration is {114} compound twin. The other defects are in mirror symmetry with respect to {113}, {115}, {447} planes and so on, which have been confirmed by electron diffraction experiments. These defects are considered to be $<110>$ symmetric tilt boundaries in bcc structure by ignoring the atomic arrangement of B2 structure and are characterized with $\Sigma$ value based on coincide site lattice (CSL) model. For instance, the $\Sigma$ value of {114}, {113}, {115} and {447} boundaries are $\Sigma$ 9, $\Sigma$ 21, $\Sigma$ 27 and $\Sigma$ 81, respectively. Lots of (1-14) defect initially form at grain boundary and grow into grain interior. Some of those defect to (114) defect. In such case (4-47) or (4-47) $\Sigma$ 81 defect is always observed at the interfaces of (1-14), $\Sigma$ 9 and $\Sigma$ 21, 27 and 81, respectively. The (1-14) defect initially form at grain boundary and grow into grain interior. Some of those defect to (114) defect. In such case (4-47) or (4-47) $\Sigma$ 81 defect is always observed at the interfaces of (1-14), $\Sigma$ 9 and $\Sigma$ 21, 27 and 81, respectively.
discussed on the basis of CTEM and HREM observations.

**S5.3** Structure and Physical Properties of Cu$_2$MnAl Alloy Films with Significantly Different Atomic Orders. BumJoong Kim$^1$, Nina Lee$^1$, Meidong Huang$^2$, Kiwon Kim$^3$, Yury V. Kudryavtsev$^2$ and YoungPak Lee$^2$. $^1$1-Ps & Dept. of Physics, Huayang University, Seoul, South Korea; $^2$Dept. of Physics, Seoul University, Asan, South Korea; $^3$Institute of Metal Physics, Kiev, Ukraine.

Rapidly evolving field of spintronics stimulates a deep interest in ferromagnetic oxides with full spin polarization at the Fermi level $E_F$. These so-called half-metallic ferromagnets are favorable candidates for application as electrodes for spin-polarized current injection into semiconductors. A significant number of intermetallic semi-Heusler and true Heusler alloy films have been predicted to be theoretically half-metals and their films might be used as spin-injectors in such devices. True HA have a formula of X$_2$YZ, where X and Y are transition metals and Z is a s-p element, and a crystalline structure of L2$_1$. However, recent experiments indicate a limited success in the carrier injection with a high spin polarization from ferromagnet into semiconductor. It is thought to be related to poor interfacial properties (both electrical and magnetic) due to the insufficient spin polarization at $E_F$, caused by site disorder. Hence, it is important to understand the effect of structural disorder on the physical properties of these alloy films. In this study, the structure and the physical properties of Cu$_2$MnAl Heusler alloy films with significantly different atomic orders were investigated. The bulk alloy was prepared by melting Cu, Mn and Al pieces together in an arc furnace with a water-cooled Cu hearth. The x-ray fluorescence from ferromagnet into semiconductor. The transport properties of thin structural properties of films were analyzed by x-ray diffraction and specimens annealed in a furnace for various times and temperatures.

**S5.4** Laser Annealing of Shape Memory Alloys Thin Films: Oxide Growth, Stress and Microtwins. Fadila Khelfaoui$^2$, Yves Belaudouine$^2$, Xi Wang$^3$, Joost Vlassak$^2$ and Thomas Gessmann$^4$. lCAT, Rensselaer Polytechnic Institute, Troy, New York.

Laser annealing of shape memory alloys (SMA) has recently emerged as a powerful tool to selectively introduce shape memory properties in amorphous thin films. In particular, the combination of annealed and non-annealed zones in a same substrate can be used to create an intrinsic Two-Way Shape Memory Effect (TWSME), which is predicted from the relation between the crystalline and amorphous phases. A significant number of intermetallic semi-Heusler and true Heusler alloy films have been predicted to be theoretically half-metals and their films might be used as spin-injectors in such devices. True HA have a formula of X$_2$YZ, where X and Y are transition metals and Z is a s-p element, and a crystalline structure of L2$_1$. However, recent experiments indicate a limited success in the carrier injection with a high spin polarization from ferromagnet into semiconductor. It is thought to be related to poor interfacial properties (both electrical and magnetic) due to the insufficient spin polarization at $E_F$, caused by site disorder. Hence, it is important to understand the effect of structural disorder on the physical properties of these alloy films. In this study, the structure and the physical properties of Cu$_2$MnAl Heusler alloy films with significantly different atomic orders were investigated. The bulk alloy was prepared by melting Cu, Mn and Al pieces together in an arc furnace with a water-cooled Cu hearth. The x-ray fluorescence from ferromagnet into semiconductor. The transport properties of thin structural properties of films were analyzed by x-ray diffraction and specimens annealed in a furnace for various times and temperatures.

**S5.6** Formation of Defect Structures During Annealing of Cold-deformed L10 Ordered Equiatomic FePd Intermetallics. Anirudha Rajendra Deshpande and Jorg M.K. Wierzek. MSE, University of Pittsburgh, Pittsburgh, Pennsylvania.

Equiatomic L10 ordered FePd intermetallics form a good model system for investigation of effects of microstructure on hard magnetic properties in the class of ferromagnetic L10 intermetallics, such as FeCo$_2$Ge$_2$, Co$_2$MnGa, etc. The L10 ordered intermetallics are currently of interest for applications in information technology. The FCC to L10 order process in FePd intermetallics leads to a polytwinned microstructure. The magnetic behavior is discussed through a correlation with the LRO. Alternative thermomechanical processing routes have been employed to avoid the polytwinned structure. These thermomechanical strategies employ a combined reaction of annealing of cold deformed metastable FCC phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains. Various planar defect structures, such as grain boundaries, stacking faults, microtwins, anti-phase structural domain boundaries, evolve in the recrystallized L10 phase concomitant with the ordering process and produce microstructures consisting of recrystallized, L10 ordered grains.

**S5.7** Crystal Structure and Thermoelectric Properties of Al-containing Re Silicides. Eiji Terada$^1$, Min-Wook Oh$^3$, Myung-Hoon Oh$^3$, Dang-Moon Wee$^2$ and Haruyuki Inui$^3$. $^1$Materials Science and Engineering, Kyotou University, Kyoto, Japan; $^2$Materials Science and Engineering, KAIST, Daejon, South Korea; $^3$Materials Science and Engineering, KNUT, Gumi, South Korea.

Binary rhenium silicide is interesting owing to potentials as a promising candidate material for thermoelectric power devices. To compare oxide growth behaviors, similar experiments were done on specimens annealed in a furnace for various times and temperatures. For laser annealed thin films, unlike furnace-annealed ones, it is found that the oxide thickness can be reduced by the use of a CO$_2$ laser. The stoichiometry of the silicide is determined to be ReSi$_{0.75}$ instead of ReSi$_2$. The crystal structure belongs to the monoclinic system with an ordered arrangement of Si vacancies in the parent C11b lattice. In the microstructure, twins exists with their boundaries strictly parallel to [001]$_{C11b}$. Binary ReSi$_{0.75}$ exhibits significantly anisotropic thermoelectric properties such that the value of Seebeck coefficient along [100]$_{C11b}$ is positive (280 uV/K at 330 K) while it is negative (-300 uV/K at 600 K) along [001]$_{C11b}$. This result may reveal the high...
anisotropic electronic structure of ReSi$_{1.75}$. The dimensionless figure of merit (ZT) for binary ReSi$_{1.75}$ is as high as 0.7 at 1073 K when measured along the [001] direction. The ZT values for ternary ReSi$_{1.75}$ were as high as 0.5 at 1050 K. We have tried to improve the thermoelectric properties of ReSi$_{1.75}$ by adding Al. The substitution for Si in Re silicides improves the ZT value along [001]. The temperature dependence of electrical resistivity was investigated [100]. It indicates that the conduction mechanism changes from that of semiconductor to that of metal upon alloying with Al and the value of electrical resistivity at room temperature for Al-added alloys is significantly reduced by one order of magnitude compared to pure ReSi$_{1.75}$. The anisotropy is approximately 2:1 with electrical resistivity locally between twins. In these layers the arrangement of Si vacancies is distinct from that in the binary counterpart. The effects of this microstructural change caused by alloying with Al on the thermoelectric properties will also be presented.

S5.8
Identification of the Chirality of Intermetallic Compounds by Electron Diffraction

S5.11
Abstract Withdrawn

S5.12
Ab Initio Calculations of Phase Stability, Defect Energies, and Theoretical Strength in Ductile RM B2 Compounds

S5.13
B2 Phases and their Defect Structures: Part II. Ab initio Vibrational and Electronic Free Energy in the Al-Ni-Pt-Ru System

Over the past decades, CALPHAD modeling has been increasingly used to describe the thermodynamic properties of a great number of material systems. Thans to these models, the time required for the development of a given material can be significantly reduced, as the knowledge of the thermodynamic stability of the different phases in an assigned system allows the focusing of the experimental work only on the relevant compositional and thermal regions of the phase diagram. Unfortunately, in many cases, especially in high temperature materials, experimental information needed as input in CALPHAD models is lacking. Fortunately, thanks to recent progress in computational materials science, the gap in experimental knowledge...
can be filled with self-consistent ab-initio studies. Considerable progress has been made regarding total energy calculations at 0 K of concentration, order, an accurate description of electronic and vibrational properties of crystalline materials is needed if one is to develop a complete thermodynamic description of any given phase, as they allow the description of the thermodynamics of crystals at finite temperature. Different types of extrinsic and intrinsic defects to create a model of the B2 phases with large amounts of constitutional defects. Free energies which were calculated using the density functional theory system is presented. This complements the ongoing efforts in this group to calculate the 0K energetics of this quaternary system. Density Functional Theory within the GGA (and LDA) approximation is used to calculate the vibrational entropy of the B2 phases via the standard method. The electronic free energy is calculated by integrating the electronic DOS at different volumes. By combining the 0K energetics with the contributions to the free energy of vibrational and electronic degrees of freedom, thermal properties such as enthalpy, specific heats, entropies and so forth are calculated and compared to the available experimental results. Additional results using the much simpler Mean Field Potential theory are also presented for comparison. The results from this work will be employed as input data in the CALPHAD thermodynamic optimization of the modified A2/B2 sublattice model, which allows the description of both the disordered and ordered bcc structures with a single Gibbs energy function.

**B2 Phases and their Defect Structures: Part I. Ab initio Enthalpies of Formation and Entropy of Mixing in the Al-Ni-Pt-Ru System**

Sara Prina, Ramseyo Arroyave, Chao Jiang and Zhi-Kui Liu; Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

Ordered B2 phases are often used in high-temperature structural applications due to their high melting temperatures, good oxidation resistance in harsh environments, low mass density and superior mechanical properties. B2 phases often exist over a composition range as the result of defects in the crystal structure. Defects are either anti-site atoms or vacancies, or in cases where the deviation from stoichiometry extends to both composition sides, a combination of defects. A thorough knowledge of the fundamental physical and chemical properties of the different defect structures is essential in the development of B2-based alloys. In a systematic first principle study of the Al-Ni-Pt-Ru system, the enthalpies of mixing for the disordered A2 phases have been calculated with Special Quasirandom Structures (SQS) by mimicking the random bcc alloy with the local pair and multisite correlation functions. The enthalpies of formation for stoichiometric B2 phases (NiAl, PtAl, RuAl) have been calculated using both the GGA and LDA approximations, while the enthalpies of formation for B2 phases with large amounts of constitutional defects (both vacancies and anti-site atoms) were also calculated using the SQS approach. The calculated B2 lattice parameters for the different defect structures were compared with experimental results. It was also shown how these values, together with vibrational and electronic free energies which were calculated using the density functional theory within the GGA and LDA approximations, can be used as input values for the modeling of sublattices using the modified sublattice model. The modified sublattice model describes the bcc phases with one Gibbs energy function and simplifies the CALPHAD model significantly.

**Thermal Analysis of Relaxation Processes of Supersaturated Vacancies in B2-Type Aluminides**

Kyotaro Nakamura, Chao Jiang and Zhi-Kui Liu; Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

Relaxation behavior of supersaturated vacancies in B2-type aluminides such as NiAl and CoAl was studied by the differential scanning calorimetry (DSC) method. Typical DSC peaks were observed only at first heating runs, depending on alloy composition and quenching temperature. In stoichiometric NiAl and CoAl water-quenched from 1773 K, a single exothermic peak appeared near 950 K. On the other hand, two exothermic peaks appeared in Al-poor Ni-68at%Al and Co-42at%Al alloys quenched from 1773 K. One of the two exothermic peaks in Co-42at%Co disappeared as quenching temperature decreased from 1773 to 1573 K. The exothermic peak was not observed for the samples furnace-cooled and quenched below 1473 K. In the Al-rich side, no peak was also observed for NiAl and CoAl. Furthermore, the DSC measurements were extended to the rapidly solidified B2-type aluminide ribbons where the vacancies with higher concentrations in the solid state could be quenched and reduced. Relaxation processes of supersaturated vacancies are kinetically analyzed, and then systematically discussed comparing with properties on diffusion and point defects for the B2 aluminides.

**Microstructure and Mechanical Properties of Fe-Ni-Mn-Al Alloys**

Markus W. Wittmann, Ian Baker, James Hanns and Paul Munroe; 1Dartmouth College, Hanover, New Hampshire, 2University of New South Wales, Sydney, New South Wales, Australia.

A series of Fe-Ni-Mn-Al alloys with elemental concentrations in the range 15 - 35 at. % were cast and found to have a microstructure consisting of alternating, coherent 50nm wide B2 and b.c.c. plates aligned along <100>. Based on the regular interval and connectivity of the two phases, formation of the microstructure is attributed to spinodal decomposition. Mechanical tests on one of these alloys, Fe-20Ni-25Mn-25Al, revealed yield strengths of 1.3 - 1.5 MPa between 300 and 600K and a strength of about 200 MPa at temperatures up to 1073K. Heat treatments of one hour at 1073K resulted in coarsening of the microstructure and formation of a b.c.c. phase within the b.c.c. matrix. The results of an investigation into the effects of both heat treatments and compositional variations on the microstructure and mechanical properties of the multi-phased alloys will be presented.

**Thermomechanical Treatment of a Fe-Al alloy**

Joachim Konrad, Andre Schneider, Stefan Zaefferer, Georg Frommeyer and Dierk Rasche; 1Materials Technology, Max-Planck-Institut für Eisenforschung GmbH, Düsseldorf, Germany; 2Material Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

Di-Al-based alloys are regarded as promising for high temperature applications in corrosive atmospheres. Generally Fe-Al shows highest strength compared to disordered FeAl alloys and good resistance in oxidizing and sulphidising atmospheres. The lack of ductility can be overcome by applying a thermomechanical treatment, such as rolling at high temperatures in the A2 (disordered bcc structure) and in the B2 (ordered structure) regime. Subsequent recrystallization decreases ductility again. A thermomechanical treatment including stepwise hot (T > 900kdeg°C) and warm (T > 400kdeg°C) rolling has been established. Aims of the rolling step are the refinement and homogenization of the microstructure and the improvement of formability by strain induced recrystallization. The former is achieved by hot rolling conditions leading to a combination of dynamic and post dynamic recrystallization resulting in a characteristic corrugated texture and a controlled grain size. The latter aim is achieved by the movement of single fold dislocations initiating disorder by generating anorthic phase boundary movement subsequent warm rolling this is utilized to improve the ductility. A controlled final heat treatment is applied to produce a fully recovered microstructure and to stabilize the state of improved ductility by reducing driving forces for recrystallization. The influence of hot and warm rolling on the texture evolution and mechanical properties have been investigated. Significant differences in the microtexture according to in-grain orientation gradients and stored energy in correlation with hot rolling temperature have been observed. Warm rolling has been performed in the temperature regions of B2 and D06, regimes. The influence of rolling temperatures on ductility and recrystallization kinetics as well as texture and microtexture of the as-rolled state have been examined. On the basis of microtextures investigations by means of EBSD and Taylor type
texture simulation, a mechanism based on the activation of different slip systems is proposed to explain the observed behavior.

S5.19 Optimisation of Precipitation for the Development of Improved Wrought FeAl Based Alloys. Sotero Kobayashi, 1 Stefan Zaefferer, 1 Andre Schneider, 1 Dirk Rasche, 2 and George Frommeyer 2
1 Materials Technology, Max-Planck-Institute for Iron Research, Dusseldorf, Germany; 2Max-Planck-Institute for Iron Research, Dusseldorf, Germany.

FeAl based alloys with bcc structures (α : disordered A2 and ordered B2 or D03) have been considered as a structural material for high-temperature application against high-temperature oxidation and sulphidation. Serious problems for the application are the poor high-temperature strength, creep resistance and the limited ductility at low temperatures. Aiming at this work is to optimise the fabrication processes to satisfy both, strengthening by precipitates for an improved high-temperature strength and inhibition of recrystallisation that deteriorates room-temperature ductility as well as strength. A Fe26Al10Cr single-phase (α) alloy and two different two-phase (α+TiC) alloys with different volume fractions of TiC have been prepared, homogenised in the α single-phase region and rolled at 800°C in the two-phase region of α+TiC to a reduction of 65%, and the kinetics of static recrystallisation has been examined. Based on the results, an optimised thermo-mechanical process has been proposed for the development of a novel wrought FeAl-based alloys with strengthening MC carbides. In Fe26Al10Cr-3Ti-0.2C alloy with a higher amount of TiC the transus temperature between α and α+TiC at 1400°C and the initial rolling deformation of 80% and recrystallisation is very quick. Needle-like TiC particles with more than 1 μm in length formed during air cooling after homogenisation and coarsened during the rolling process. The hot deformation with such large TiC particles produces particle stimulated nucleation (PSN) and hence accelerates recrystallisation. While in Fe26Al10Cr-3Ti-0.2C alloy with a lower amount of TiC, particles with less than 1 μm formed during cooling after homogenisation and exhibited no acceleration of recrystallisation. Based on the results, the following process is proposed in order to accomplish both, strengthening by particles and inhibition of recrystallisation: hot deformation is performed in a single phase or two phase conditions with a small amount of TiC to obtain a high-temperature strength and inhibition of recrystallisation.

S5.20 Effect of Excess Vacancy on Antiphase Domain Growth in Fe3Al. Yukihiro Kamikawa, Takashi Higashimura, Yoritoshi Minamino and Nobuhiro Tsuchi; Department of Adaptive Machine Systems, Graduate School of Engineering, Osaka University, Osaka, Japan.

Growth of D02-type antiphase domain (APD) in Fe3Al was investigated by TEM observation and resistivity measurement focusing on the effect of excess vacancies introduced in the quenching process from disordered state. The variation of APD size exhibited considerable deviation from the conventional "parabolic-growth-law" in the early stage. The variation of the APD size was numerically calculated using diffusion data on the assumption that the migration of the APD boundaries was enhanced by non-equilibrium excess vacancies and the vacancy concentration decreased during the isothermal annealing for APD growth. The calculated variations of APD size could be successfully fitted to the experimental results in the cases that the quenching temperature (Tq) was in the B2 ordered phase region. Above Tq the annihilation of APD grows in the disordered bcc phase region. The APD growth in the latter case was much slower than expected from the calculation. This discrepancy was attributed to the rapid decrease in the vacancy concentration by vacancy clustering followed by heat treatment for further precipitation of very fine particles. The fine particles would also act to pin the boundaries of growing grains, thus leading to extended recovery rather than recrystallisation. In the Fe26Al10Cr-2Ti-SiC system this process is difficult to be realised due to the high precipitation temperature and quick precipitation kinetics of TiC. Phase diagram determinations by Thermo-calc and experiments revealed that the addition of (V, C) or (Mo, C) instead of (Ti, C) is effective to lower the precipitation temperature. A Fe26Al10Cr single-phase alloy had exhibited to inhibit recrystallisation during air cooling after homogenisation and coarsened during single phase or two phase conditions with a small amount of TiC. Determinations by Thermo-calc and experiments revealed that the considerable deviation from the conventional "parabolic-growth-law" in the APD boundaries was enhanced by non-equilibrium excess vacancies.

S5.21 Microstructure, Mechanical Properties and Wear resistance of Fe-Al based alloys with various alloying elements. Han-Sol Kim, Woon-Yong Kim and Tae-Youl Ra; Advanced Materials R&D Center, Korea Institute of Science and Technology, Incheon, South Korea.

We report on microstructure, mechanical properties and wear result of Fe-Al binary alloys with various alloying elements. The microstructures were examined by electron scanning (SEM) and X-ray diffractometry (XRD). XRD analysis showed that the stacking fault probability is proportional to a function of pressure. Line shifts and line profiles as a function of pressure were used to analyze the microstructural evolution of elastic and plastic strains. Plastic deformation is due to the onset of non-hydrostatic stresses and the introduction of stacking faults (FS). A volume incompressibility in the equation of state due to plastic deformation and the saturation of the stacking fault probability is followed by an energy-compensation of a full slip system. The compression of this state is isotropic and independent of the presence and type of the pressurizing medium. From the systematic shift of different Bragg peaks the stacking fault probability as a function of pressure is derived. The onset of incompressibility of the microstructural state corresponds to the onset of increase of the stacking fault probability. Using finite elasticity, it is shown that the stacking fault probability is proportional to an average of the deviatoric strains.

S5.22 Microstructures in Cold-Rolled Ni3Al Single Crystals. Kyosuke Kishida, Masahiko Demura and Toshiyuki Hirano; Materials Engineering Laboratory, National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

Considerable amount of studies on the deformation structures in Ni3Al base intermetallic alloys have been carried out so far, however, main interests were the dislocation structures near the yield point mainly because the anomalous yield behavior is one of the most attractive properties of Ni3Al. In contrast, the deformation structures in the Ni3Al-base alloys after large plastic deformation such as cold rolling have rarely been studied by transmission electron microscopy (TEM). Recently we have carried out systematic studies on cold rolling of binary stoichiometric Ni3Al single crystals with various initial crystal orientations and revealed that the cold rolling behaviors are strongly dependent on the initial crystal orientations, especially on the initial rolling direction (RD). Very thin foils with good quality in shape are available only when the initial RD is close to <001>, whereas macroscopic curling and cracking hinders high cold reduction for samples with other initial RD. Such an anisotropic rolling behavior must be closely related to the internal deformation microstructure developed during cold rolling. It is therefore necessary to investigate the characteristics of the microstructures in the cold rolled thin foils for understanding the details of the texture formation mechanism. In the case of Ni3Al base, the cold rolled structure is formed. Inside each band, a huge amount of extended intrinsic stacking faults on {111} planes is developed from relatively early stage of cold rolling. However, no deformation twins are found at any reduction. The presence of such extensive stacking faults is likely to impede uniform slip activation on the other slip planes, and eventually cause heterogeneous and concentrated shear along the other {111} planes. For further cold rolling, the stacking faults and/or the concentrated shear continues to develop on two {111} planes resulting in the formation of the microbands-like structure, which commonly observed in some rolled fcc metals. Band shear that composed of nano-sized grains develop only at reductions above 90% in the samples with the banded structure. Based on the results of the TEM observations, we will discuss the reason for the anisotropy in the cold rolling of the Ni3Al single crystals.
Nasimul Alam Syed, Shyamal Kumar Pabi and B.S. Murty;

The present study deals with the synthesis of nanocomposites of W-10, 20, 30% NiAl and W-10, 20, 30% Ni3Al by high energy ball milling of elemental blends of W, Cu, Ni and Al and looking at the possibility of formation of intermetallics in these systems by high energy ball milling. Mechanical alloying has been carried out in a Fritsch Pulversette P-5, using WC milling media. The ball milled powders have been characterized using XRD, TEM and SEM. Formation of intermetallics in these systems have been studied by measuring the lattice parameters from the corresponding x-ray diffraction patterns. All the systems investigated in the nanocomposites have been calculated from the x-ray peak broadening using Voigt peak profile analysis. The results indicate that nanocomposites with excellent homogeneity can be synthesized by mechanical alloying. The grain coarsening tendency of the nanocomposites after annealing for 2 hours at 400, 600 and 800°C has also been studied. Lattice parameter calculations in all the systems have been carried out both after milling and heat treatments. Interestingly, W shows an increase in the lattice parameter with decrease in the crystallite size suggesting lattice expansion in the nanocrystalline state. The decrease in the crystallite size of W with milling time is more rapid in case of elemental W in comparison to W-Cu nanocomposites, which can be attributed to the work hardening effect caused by ductile Cu. W-NiAl and W-Ni3Al systems show lower crystallite size of W due to the brittle nature of NiAl and Ni3Al. The grain coarsening rate, on heat treatment, of W is the lowest for elemental W in comparison to W-Cu nanocomposites. The sintering behavior of the nanocomposites will also be discussed.

**S5.26**

**Parameters of Dislocation Structure and Work Hardening of Ni3Ge, Nina A. Koneva**, Yulia V. Soloveva, Vladimir A. Starenchenko and Eduard V. Koslov;

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Ni3Ge intermetallic possesses L12 structure and remains fully ordered up to the melting temperature. Among other intermetallics with the L12 structure, Ni3Ge is one of the nanocrystalline materials fabricated by mechanical alloying. Dislocations in this intermetallic are Marsenkovski dislocations. In the present work, the following mechanical characteristics were studied in tension: yield stress τy, work hardening coefficient δσ = dσ / dε for five orientations of single crystals [001], [1 0 3], [4 0 9 17], [2 3 4] and [1 1 1]. Measurements were carried out in the 4.2 K - 1000 K temperature interval. The peak temperature τy(T) and the amplitude of stress at this temperature decrease strongly upon shifting the deformation axis from [001] to [1 1 1]. The temperature dependences of τy(T) and δσ(T) were compared. In addition, the dislocation structure was studied using TEM. The scalar dislocation density ρ was measured by the deformation profile from the yield stress up to failure. The temperature intervals of octahedral and cubic glide are distinguished. The type of active glide systems were identified using slip lines and data about the shape change of the crystal dislocation length. The main mechanism of dislocation movements is Kear-Wilsdorf locks. It was established, that the following well-known relationships is valid at temperatures above 177K: τ = τy + α Gbσgtpρ, where τy is the stress of self-locking (solid solution hardening or crystal lattice resistance) of dislocations, α is a parameter of dislocation interaction. Parameter α decreases linearly with temperature for all orientations of single crystals. At the same time, the dependence τy(T) has a shape of a curve with a maximum. It was established, that τy(T) and τy(T) dependences in the same manner with changes of the orientation of the deformation axis. Comparison of τy(T), τy(T) and τy(T) dependences was done on the basis of the modern concepts of work hardening. Using the following relationship: δσ = δσ / dε, the rate of dislocation accumulation, dp / dp, at different temperatures and different orientations of single crystals of the studied alloy was taken into account. The experiments showed that the maximum in dislocation density is reached at the same temperature as the maximum of τy and τy. It was established that the mechanisms of dislocation self-locking play an important role in the temperature dependence of work hardening of the Ni3Ge intermetallic.

**S5.27**

**Crystal Structure, Phase Stability and Plastic Deformation Behavior of Ti-rich Ni$_3$(Ti,Nb) Single Crystals with Various Intermetallic Phases in the System Ni$_3$Al - Ti - Nb**

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The Ni$_3$X-type intermetallic compounds (X=Al, Ti, Sn, Nb, V, etc.) are known to crystallize in various geometrically closely-packed (GCP) crystal structures: Li$_2$ for Ni$_3$Al, D$_{024}$ for Ni$_3$Ti, D$_{024}$ for Ni$_3$Sn, D$_{024}$ for Ni$_3$Nb, and D$_{024}$ for Ni$_3$V, respectively. The structures of those GCP Ni$_3$X-type phases are characterized by their two features: the stacking sequence of close-packed planes and the ordered arrangements of X atoms on the close-packed plane. The selection of crystal structure is known to be controlled mainly depending on the valence electron density ratio (e/a) and the atomic radius ratio (R$_{Ni}$/R$_X$). The compounds described above have relatively simple crystal structures based on 2-fold hcp, 3-fold fcc or 4-fold dipyramid structure, while the existence of GCP phases with long-period stacking sequences has also been reported in some binary and ternary systems. We investigated the phase stability of Ni$_3$(Ti$_{1-x}$Nb$_x$) single crystals with different Nb content (x=0, 0.01, 0.03, 0.05 and 0.1), and found the formation of some metastable GCP structures with long-period stacking sequences such as 9-fold, 10-fold and 18-fold stacking sequences. The stacking configuration of close-packed planes in those phases was examined by a direct observation using high resolution TEM, and we determined their crystal structures. The formation of those long-period ordered structures can be explained by the periodic insertion of hexagonal-type stacking faults into the D$_{024}$-Ni$_3$Ti phase. Plastic deformation behavior of those long-period ordered phases was examined by compression tests between RT and 1200°C using single crystals. Yield stress anomaly (YSA) was observed in all the crystals by slip on the close-packed plane, which was caused by the Kear-Wilsdorf locking of screw dislocations, similarly to those observed in other binary Ni$_3$X-type compounds. The extent of YSA is, however, different depending on their crystal structures. The relation between the crystal structure and YSA behavior is discussed especially focusing on the anisotropy of APB energy in those long-period ordered phases.

**S5.28**

**Formation and Migration of Thermal Vacancies in Ni$_{3}$Ti$_{2}$Ge$_{1-x}$Fe$_{x}$ Intermetallic Compounds, Ewa Partyka**, Rafal Kozubski, Wolfgang Pueschel and Hans-Eckhardt Schaefer;

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Chemical ordering in intermetallic compounds is predominantly controlled by atomic jumps to nearest neighbor vacancies. An important factor controlling the dynamics of the vacancy formation is the concentration of thermal vacancies. In the present contribution, the intermetallic Ni$_{3}$Ti$_{2}$Ge$_{1-x}$Fe$_{x}$ system (0≤x≤0.29) with a L1$_2$ structure is studied. The admixture of Fe destabilizes the ordered phase and the "order-disorder" transition temperature decreases. This is accompanied by the decrease of the activation energy $E_A$ for the ordering kinetics. By isochronal residual resistometry at 77 K and the analysis based on the Schulze - Lücke formalism we arrived at a good estimation of the formation ($E_F$) and migration ($E_M$) energies for vacancies which contribute to the activation energy $E_A$ for the ordering process. These data suggest that $E_F$ in Ni$_3$Al is lowered when alloying the compound with Fe. More specific information on the variation of the activation energies for vacancy formation and migration upon alloying Ni$_3$Al with Fe is expected from postion lifetime and coincident Doppler broadening studies of the postron-electron annihilation radiation.
nearest and next nearest neighbour atoms, starting from a perfectly ordered structure. Some of the tendencies observed can be made plausible by arguments of atom size, diffusion barriers generally becoming lower as larger atoms are replaced by smaller ones. A modification of the common Böröcz-Lebowitz (residence-time) kinetic Monte-Carlo algorithm is proposed so as to account for varying barrier heights and atom surroundings of the jumping atom. The problems of detailed balance and of obtaining the correct thermodynamic equilibrium state are discussed, as is a kinetic ansatz for the cooperative movement of two atoms which seems to play a role in the generation of antisite pairs in a disordering step.

**S5.39**
**Modeling of Creep in Retrograde Metal/Intermetallic Composites**

William A. Curtin1, Nobuaki Sekido2, Akihito Yamauchi3, Kenji Ohkubo and Tetsuo Mohri; Mat. Sci. and Eng., Hokkaido University, Sapporo, Japan; 2Materials Science and Engineering, University of Wisconsin-Madison, Madison, Wisconsin.

High-temperature creep response is a critical technological requirement for advanced intermetallic systems aimed at replacing superalloys. These advanced systems are typically composed of a refractory metal (e.g. Nb) and a metal-silicide (e.g. Nb5Si3), with a complex microstructure. Previous work has established the creep behavior of the basic constituents (MRS Symp. Proc. 753, 321 (2003)). However, correlation of the composite creep to the constituent creep behavior needs to be improved. Here, realistic Nb/Nb5-Si microstructures are mapped onto a 2d computational mesh and creep is modeled explicitly using Finite Elements. The dependence of creep on microstructure and second-phase volume fraction and on the state of metastable phases, such as a microstructure-reconstruction technique is then used to generate 3d microstructures using statistical correlation functions extracted from 2d micrographs of the real materials. Creep of the 3d microstructures is then studied and compared to experiments on the 2d models. Stress distributions and hot spots under quasistatic loading are also analyzed. Directions for microstructure optimization to enhance creep and avoid excessive local stress concentrations under quasistatic loading at low temperatures are discussed.

**S5.50**
**Phase Equilibria Predictions in Nb-Si-Based Composites**

Yang Yang1, Bernard P. Bewlay2, Melvin Jackson3 and Austin Chang2; 1General Electric Global Research, Schenectady, New York; 2Materials Science and Engineering, University of Wisconsin-Madison, Madison, Wisconsin.

Nb-based in-situ composites are promising materials for future high-temperature structural applications. Nb-Si alloys are typically alloyed with Hf, Ti, Cr, and Al to provide a balance of mechanical and environmental properties. In order to develop an improved understanding of phase equilibria in Nb-Hf-Ti-Si quaternary system, a methodology coupling the CALPHAD-type computational thermodynamics with experimental measurement of phase equilibria was used in this paper. This paper first presents the phase equilibria in the Hf-Ti-Si ternary system determined by experiments. Microstructural and microchemical evidence provided a clear definition of the Hf-Ti-Si liquidus surface and indicated that the metal-rich end of the ternary phase diagram possesses one transition reaction. This data was then used to develop a thermodynamic description of the Hf-Ti-Si system. A thermodynamic description of the Nb-Ti-Hf-Si quaternary system was then obtained by extrapolating the thermodynamic descriptions of Hf-Ti-Si, Nb-Hf-Ti, Nb-Ti-Si and Nb-Hf-Si into the quaternary space. The phase equilibria and solidification paths predicted from the currently obtained Nb-Ti-Hf-Si quaternary thermodynamic description are compared with experimental results.

**S5.51**
**Effect of Temperature and Ternary Additives on the Creep/Recrystallization Rivalry among Phases Related to the Eutectoid Decomposition of Nb5Si2.**

Seiji Miura, Kenji Ohkubo and Tetsuo Mohri; Mat. Sci. and Eng., Hokkaido Univ., Sapporo, Japan.

Authors have reported in the previous study that the sluggish decomposition of Nb5Si2 phase is effectively accelerated by Zr addition [1]. This effect is obvious at lower temperature range than the nose temperature of the TTT curve. The crystallographic orientation relationships among phases, such as eutectoid Nb and product phases of eutectoid decomposition of Nb5Si2 (eutectoid Nb and Nb3Si phases) in the Zr-containing samples heat treated at various temperature ranges were investigated by FESEM/EBSD to understand the decomposition process. Also the effects of additives other than Zr on both decomposition rate and crystallographic orientation relationship were studied. [1] S. Miura, M. Aoki, Y. Saeki, K. Ohkubo, Y. Mishima and T. Mohri, submitted to Met. Mat. Trans. A (2004).

An outstanding issue in the processing of Mo-Mo5Si x intermetallic composites is that microcracks develop after annealing at high temperatures, which significantly degrade the mechanical properties of the composite materials. The formation of microcracks is difficult to understand from the view point of differential thermal stresses that develop during cooling, which have been estimated to be 10 MPa for both phases. It is unlikely such a level of residual stress would cause microcracking at any stage during cooling. In order to determine the nature of microcracking in Mo-Mo5Si, we have conducted a systematic study of Mo-Mo5Si composite using a combination of in-situ neutron diffraction, composition analysis, and scanning electron microscopy. In-situ neutron diffraction measurements at 1300°C revealed a significant increase of the lattice parameter in the α-Mo phase as a function of annealing time, whereas the lattice parameter of the Mo5Si phase shows a corresponding decrease. At the same time, the diffraction peak widths of both phases increased, unexpectedly, with increasing annealing time, giving evidence that plastic deformation occurred at high temperature. X-ray and neutron diffraction measurements made before and after annealing confirmed that the changes in lattice parameters are irreversible, i.e., due to plastic deformation. Because the α-Mo5Si phases were obtained off-equilibrium by fast cooling, the α-Mo phase is known to be super-saturated with Si. The in-situ neutron diffraction data therefore suggest that high-temperature plastic deformation during annealing was due to diffusion of Si atoms from α-Mo to α-Mo5Si. This finding was corroborated by microscopy studies which demonstrated that the microcracks almost always started at the interface of α-Mo and Mo5Si grains and grew into the Mo5Si phase. Furthermore, crack density measurements via microscopy indicated that as the annealing time increased more cracks were formed. Based on these experimental observations, it is clear that microcracking in Mo-Mo5Si occurred at high temperature. Quite possibly, the diffusion of Si during high temperature annealing is a highly influenced by the stress distribution at the interface where cracks were initiated when the plastic energy exceeded the tensile limit of the Mo5Si phase. This research was supported by the Division of Materials Science and Engineering, U.S. Department of Energy under contract DE-AC05-000R22725 with UT-Battelle, LLC.

**S5.53**
**Abstract Withdrawn**

**S5.54**
**High Temperature Oxidation Behavior of Al added Mo5SiB2 in - s.i.tu composites.**

Yoshimi and Shuji Hanada; Institute for Materials Research, Tohoku Univ., Sendai, Japan.

Isothermal oxidation behavior of Al added Mo5SiB2 ~ s.i.t.u composites was investigated in an Ar-20%O2 atmosphere over the temperature range of 1073-1773 K. The Al added Mo5SiB2 ~ s.i.t.u composites were prepared by arc-melting, and homogenized at 2073 K for 24 h in an Ar flow atmosphere. The ternary Mo5SiB2 ~ s.i.t.u composite exhibited a rapid mass loss at the initial oxidation stage and then the passive oxidation after the substrates were sealed with boronicate glass in the temperature range of 1173-1473 K, whereas it exhibited a rapid mass gain around 1773 K. On the other hand, the Al addition, especially of 1mol%, significantly improved the oxidation resistance of Mo5SiB2 ~ s.i.t.u composites at temperatures from 1073 to 1773 K. The results of high temperature oxidation resistances are considered to be due to the rapid formation of a continuous, dense scale of Al2O3 complex oxides. The protective oxide scale partially contained crystalline oxides, and the amounts of the crystalline oxides obviously increased with Al concentration.

**S5.55**
**Nucleation of (Mo) Precipitates on Dislocations during Annealing of a Mo-rich Mo5SiB2 Phase.**

Nobuaki Sekido, Ridwan Sakdja and John H. Perepezko; Department of Materials Science and Engineering, University of Wisconsin-Madison, Madison, Wisconsin.

Ever since the discovery of the significant improvement in oxidation resistance of the Mo5SiB2 phase by small boron additions, the intermetallic compound Mo5SiB2 (T2) has received considerable attention for an ultra-high temperature application. In the present study of particular interest were phases the nature of which is not well understood.
Mo-10Si-20B alloy where the T2 phase with a non-stoichiometric composition is in equilibrium with Mo solid solution (A2) phase. Previous studies have demonstrated that atomic size mismatched elements precipitates are formed in the T2 matrix upon prolonged annealing at 1600°C. The present study was carried out to elucidate heterogeneous nucleation of the A2 phase on dislocations in the T2 matrix. For the examination of Fe2Nb alloy with hexagonal structure, five, 20 and 100 hours, followed by furnace cooling. The dislocations formed in the T2 phase were characterized by TEM using the thickness fringe method, invisibility criterion and stereo observations. The lattice parameters for the dislocations derived from these observations have revealed that some dislocations have developed during annealing in the T2 phase, though few dislocations and no precipitates are present in the T2 phase of the as-cast alloy. It is found that the dislocation density in the T2 phase qualitatively increases with annealing time. The dislocation network is mainly composed of the edge dislocations with Burgers vectors of <100> and <110>. XRD measurements have demonstrated that the lattice volume of T2 phase increases by about 0.9% after the annealing at 1500°C for 100 hours. Judging from the following results; (i) dislocations are developed only by heat treatments, and (ii) the lattice volume of T2 phase increases after the annealing, it is concluded that some amount of structural vacancies are introduced during solidification, and the vacancies in excess of the equilibrium concentration are annealed out to form dislocations within the T2 matrix. It is therefore possible that the character and mobility of dislocations are strongly influenced by the composition of the T2 phase. In addition the A2 particles have been preferentially precipitated on these dislocations. This suggests that these dislocations are the preferential nucleation sites for the A2 phase. The support of Air Force Office of Scientific Research (F40620-03-1-0008) is gratefully acknowledged.

S5.36

The ferromagnetic L10-ordered intermetallic phase MnAI appears near equiatomic compositions after appropriate heat treatment. This metastable phase has high uniaxial magnetic anisotropy, is the basis for attractive magnetic properties in these materials. The technologically important properties of these alloys are highly structure-sensitive, depending strongly on the microstructure and defect structure produced by transformation from the high-temperature disordered epsilon-phase (A3, hcp) to the ordered L10-ordered manganese-aluminum intermetallic phase. The support of the National Science Foundation (DMR) is gratefully acknowledged.

S5.37
Phase Equilibria and Lattice Parameters of Fe2Nb Laves Phase in Fe-Ni-Nb Ternary System at Elevated Temperatures. Nobuyuki Gomi, Sumio Morita, Takashi Matsuo and Masao Takeyama; Dept. of Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo, Japan.

Fe2M TCP compounds are potential strengtheners in austenitic heat-resistant steels to be used above 973 K for steam turbine component beyond USC power plant. Among the Laves phases, Fe2Nb(E) with hexagonal structure is the most attractive because high congruent melting temperature (1914K) and relatively large anisotropy, is the basis for attractive magnetic properties in these materials. High strength and brittleness is found at room temperature. A quasi-superplastic behavior is found at temperatures above 973 K. SPS between mechanically alloyed powders and the corresponding supporting material, has been used to produce coatings with a thickness varying from 0.1 to 0.3 mm. Steel, Co and Al are used as substrate. Characterization of the coating includes chemical analysis of the interface between coating and substrate. High hardness across the interface with the support is found. The chemical reaction with the supporting material creates different phases that have been identified.

S5.40
Compressive Creep Behavior in Coarse Grained Polycrystals of Ti3Al and its Dependence on Binary Alloy Compositions. Tohru Takahashi1, Yuki Sakaim02 and Shunzi Song2; 1Department of Mechanical Systems Engineering, Tokyo University of Agriculture and Technology, Koganei, Tokyo, Japan; 2Graduate Student, Tokyo University of Agriculture and Technology, Koganei, Tokyo, Japan.

Compressive creep behavior has been investigated on coarse grained Ti3Al alloys with aluminum contents ranging from 23at.%Al to 42at.%Al, in order to obtain basic experimental information concerning the chemical composition effect on creep of Ti3Al. Pure aluminum and titanium of 99.995% purity were arc-melted into small ingots weighing about 10gms under an argon atmosphere. Small pieces were hot deformed at 1300K by about 50% reduction in height in air and subsequently vacuum annealed at 1400K for 100ks. The resulting microstructures contained equiaxed grains of alpha 2 single phase whose average diameter ranged from 125 to 192 micrometers.
except alloys containing 40 and 42 at.\% aluminum. The microstructures in 40Al and 42Al materials contained small amounts of gamma phase. Microscopic creep tests were performed in specimens on parallelepiped specimens with dimensions of 2mmx2mmx3mm. The amount of creep deformation was measured by an LVDT sensor attached straight upon the load-conveying rod above the specimen. Based on the recorded contraction in height of the specimen, the applied load was intermittently adjusted in order to keep the true stress constant within about 1\% error. The applied compressive stress was mainly 150MPa, and the test temperature ranged from 1100K to 1200K. The primary transient and minimum creep rates were utilized as a representing parameter to illustrate the chemical composition effects upon creep strength, there seems to be 3 or 4 domains in the composition dependence of minimum creep rates upon aluminum contents of the tested alloys. Around 25at.\% aluminum the minimum creep rate showed a peak where the creep rate decreased as the aluminum content further from the stoichiometric composition. The minimum creep rate in the materials containing 27-32at.\% aluminum hardly showed a significant effect. Increasing creep rate was observed from 32at.\% up to 35at.\% aluminum. And, finally, the minimum creep rate decreased as the aluminum content exceeded 40at.\% where the gamma phase formed.

S5.41 TRANSFERRED TO S7.9

S5.42 Microstructure of Bimetallic Joints of Orthorhombic Titanium Aluminide and Titanium Alloy. Bella A. Greenberg and Valery V. Rybin. 1 Institute of Metal Physics, Ural Division of the Russian Academy of Sciences, Ekaterinburg, Sverdlovsk region, Russian Federation; 2CRM Prometey, St.-Petersburg, Leningrad region, Russian Federation.

An excellent set of strength properties, which is inherent in orthorhombic titanium aluminides, makes one believe they can be beneficial for high performance of bimetallic joints. One more favorable factor is that orthorhombic aluminides (unlike other aluminides) contain a disordered phase, which has a relatively high plasticity. Bimetallic joints of orthorhombic titanium aluminide and a titanium alloy were produced (diffusion and explosion welding). The phase composition and the microstructure of the joints were studied by the methods of X-ray diffraction analysis, X-ray spectrum microanalysis, metallography, scanning and transmission electron microscopy. In the case of explosion welding the phase composition differed slightly from the initial composition. However, the structure changed drastically. Structural elements, which are typical of strongly deformed materials, were observed. The concurrent presence of a cellular structure and a band structure with a high density of dislocations and new grains pointed to different stages of fragmentation, which were due to an inhomogeneous plastic deformation. Different variants of the interrelation between recrystallization and phase transformations in the bimetallic system under study were considered. In the case of diffusion welding a multilayered structure of bimetallic joint was found. Layer-forming phases were identified. It was found that the titanium alloy mostly regained its initial state, while the aluminide turned into a disordered BCC phase. The corresponding BCC phases appeared on approaching to the contact surface on both sides. Their compositions differed from compositions of the initial alloys because Nb and Al diffused to the titanium alloy and Ti diffused to the orthorhombic aluminiide. The mutual adjustment of the BCC lattices, which took place during diffusion welding, was beneficial to high quality of the bimetallic joint. One more important factor was the absence of a continuous intermetallic layer, which could cause embrittlement, near the contact surface. The above two factors were revealed earlier in the study of a bimetallic joint of a titanium alloy of a similar composition and a stainless steel (diffusion welding). Thus, two techniques, which were used for welding together one and the same orthorhombic aluminide and a titanium matrix, provided bimetallic joints having absolutely different structures. After diffusion welding, the structures near the contact surface contained BCC \( \alpha \) and BCC \( \gamma \)) phases. The corresponding compressive and plane-strain fracture toughness values of \( \alpha \) and \( \gamma \) phases were determined. Fracture surface observations indicate that these variations are attributable to differences in local lamellar orientations ahead of the notch. These fracture toughness values are also lower than those having been previously reported in conventional samples. This may be due the absence of significant extrinsic toughening mechanisms in these micro-sized specimens. Fracture mechanisms of these alloys are also considered on the micrometer scale. The results obtained in this investigation give important and fundamental information on the development of TiAl based alloys with high fracture toughness.

S5.43 Micro-Fracture Toughness Testing of TiAl Based Alloys with a Gamma Lamellar Structure. Kazuki Takahashi, Daisuke Rudinal, Timothy Halford, Yakihi Higo and Masao Takeyamm. 1 National Institute of Advanced Industrial Science and Technology, Tokyo, Japan; 2Fusion Technology - PSI, Ecole Polytechnique Federale de Lausanne, Villigen PSI, Switzerland; 3Materials Science and Technology, Kyoto University, Kyoto, Japan.

Two phase materials containing the gamma phase showed small primary transient probably due to the constraint from the harder gamma phase. The minimum creep rates were utilized as a representative parameter to illustrate the chemical composition effects upon creep strength. In this investigation, a micro-sized testing technique is applied to investigate the fracture properties of lamellar colonies in a TiAl based alloy. A fully lamellar, Ti-46Al-5Nb-1W, alloy and a Ti-48Al two-phase single crystal (PST) were used in this work. Micro-sized cantilever specimens with a size \( 10 \times 15 \times 50 \mu m^2 \) were prepared from one lamellar colony by focused ion beam machining. Notches with a width of 0.5 \( \mu m \) and a depth of 5 \( \mu m \) were also introduced into the micro-sized specimens by focused ion beam machining. Fracture tests were successfully completed using a mechanical testing machine for micro-sized specimens (MFT-2000) at a constant temperature. The true stress values were obtained in the range 0.2 - 6 MPa1/2. Fracture surface observations indicate that these variations are attributable to differences in local lamellar orientations ahead of the notch. These fracture toughness values are also lower than those having been previously reported in conventional samples. This may be due to the absence of significant extrinsic toughening mechanisms in these micro-sized specimens. Fracture mechanisms of these alloys are also considered on the micrometer scale. The results obtained in this investigation give important and fundamental information on the development of TiAl based alloys with high fracture toughness.
and perpendicular to the lamellar interfaces. Abnormal deformation behaviors were observed to occur in both orientations. When the compression axis is parallel to the lamellar interfaces, the a2 and alpha lamellae deform primarily by shear in planes inclined with the lamellar interface, while the shear vectors lie in the interface. However, in-plane shear, shears in slip planes parallel to the lamellar interfaces, also influence the lamellar interface. When the loading axis is perpendicular to the lamellar interface, in-plane shear was found to be dominant at the beginning stage of plastic deformation and contributes more to the macroscopic strain. These behaviors are controversial at the macroscopic scale since the applied resolved shear stress for these deformation systems is zero. The abnormal phenomenon was explained by the large coherency stresses along the lamellar interfaces.

**S5.47**

**Experimental Studies and Thermodynamic Modelling on the Phase Transformations in γ-TiAl Based Alloys.**


Current γ-TiAl based alloys are complex multi-phase materials. Effective alloy development, hot working, and subsequent heat treatments require the knowledge of the constituent phases and their transformation kinetics. For casting the solidification path has to be known, whereas for thermal mechanical processes the role of transus temperatures, which sensitively depend on alloy composition, are of particular importance. The knowledge of the influence of alloying elements on the amount and the thermodynamical stability of the phases present in the microstructure is important for heat treatments for optimization of mechanical properties. For example, β/2 phase stabilizing elements can be harmful for creep resistance. The present study combines light-optical and scanning electron microscopy, X-ray diffraction and differential scanning calorimetry for the characterization of phase transformations in low alloyed γ-TiAl alloys (so-called 2nd generation alloys showing total alloying concentrations < 5 at%) and high Nb containing γ-TiAl based alloys. In particular, these high Nb containing alloys with chemical composition Ti-42-45Al-5-10Nb+X (at%) have attracted a lot of attention because they combine high creep strength, good ductility at room temperature and excellent oxidation resistance. The term X stands for small amounts of metallic and non-metallic alloying elements. Here, carbon is of particular interest because of its applicability to precipitation hardening. In addition to the experimental investigations, thermodynamic modelling based on the CALPHAD method was used for phase prediction. The results derived from experiments conducted on a variety of γ-TiAl based alloys are compared to thermodynamic calculations. Finally, the influence of carbon on the transition temperatures will be presented. Carbon is currently not available in the commercial TiAl database.

**S5.48**

**Massive Transformation in High Niobium Containing TiAl-Alloys.**

Arno Bartels, Slawomir Bystrozanowski, Harald Chludil, Holmut Clemens, Rainer Gerling, Harald Leitner and Frank-Peter Schimschak. Department of Physical Metallurgy and Materials Testing, University of Leoben, Leoben, Austria; *Materials Science and Technology, TU Hamburg-Harburg, Hamburg, Germany; 1Institute for Materials Research, GKSS Research Centre, Geesthacht, Germany.

Two high Nb containing γ-TiAl-based alloys with chemical composition of Ti-35Al-7.5Nb (at%) and Ti-46Al-5Nb (at%) were massively transformed to single phase γ-TiAl by rapid cooling from the α-phase field, i.e. from temperatures above 1300°C. Using a quenching dilatometer the influence of cooling rate and starting temperature of the massive transformation was studied. In addition, the required undercooling to start massive transformation could be determined by means of dilatometer measurements. With a high cooling rate from a high starting temperature, for example, an incomplete massive transformation is obtained, characterized by small transformed areas situated at the grain-boundary triple points of the parent α-grains. The orientation map of these embedded massively transformed γ-Islands and of the surrounding α-grains was determined by electron back-scatter diffraction (EBSD). The cube phase was found to be cubic and the α-phase as hexagonal α. The analyses of these measurements resulted in the orientation relation between the close-packed planes and directions of the parent α-grains and those of the massively transformed γ-Islands. The textural and thermal stability of the microstructure, massively transformed material was annealed for one hour at temperatures, increased in steps of 100°C, between 400-1200°C. After the various annealing treatments the microstructural changes caused by recovery, recrystallization and precipitation of α/α2-phase were analyzed by hardness tests and X-ray diffraction measurements. The onset of these processes is reflected in a drop of hardness and X-ray diffraction measurements. The complete analysis of these phenomena is prevented by the high density of transformation twins, high interparticle stresses and the low density of new phases. Furthermore, the occurrence of the γ/α2-ratio, is attributed to the relaxation of internal stresses and formation of α2-Ti3Al. The appearance of α2-phase is associated with an increase of the Al-content in the γ-TiAl phase.

**S5.49**

**Abstract Withdrawn**

**S5.50**

**Different Creep Behaviors of Two Fine-Grained XD TiAl Alloys Prepared by Similar Heat Treatments.**

Hanliang Zhu, D. Y. Seo and K. Maruyama. 1Graduate School of Environmental Studies, Tohoku University, Sendai, Japan; 2Institute for Aerospace Research, National Research Council of Canada, Ottawa, Ontario, Canada.

The structure and phase transformations of polycrystalline orthorhombic Ti2AlNb alloys subjected to severe plastic deformation and creep. The phase transformations of the displacement type and those associated with a change in the degree of long-range order, namely β2-omega (B2), B2-B19 and B2- beta (BCC) (in the case of alloy with initial B2-phase structure) and O- B19(29% and O-phase structure, Ti2AlN9). Two different types of severe plastic deformation by high pressure or shock wave loading with steel plate were studied. It was found that severe plastic deformation of orthorhombic Ti2AlNb alloys caused phase transformations of the displacive type and associated with a change in the degree of long-range order, namely β2-omega (B2), B2-B19 and B2-beta (BCC) phases. In the early stages of deformation this behavior was observed for each of the phases present in the microstructure. The BCC phase is also metastable, it is absent in the equilibrium phase diagrams at room temperature. Upon severe plastic deformation, the alloy with initial O-phase, as the degree of deformation increases, the content of the B19 phase disordered for niobium decreases, whereas the content of the completely disordered.

**S5.51**

**Phase Transformation in Orthorhombic Ti2AlNb Alloys Under Severe Deformation.**

Nataliya V Kozantseva, Belia A Greenberg and Vitaliy P Pilugin. 1Ural Division RAS, Institute of Metal Physics, Ekaterinburg, Russian Federation; 2Institute of Metal Physics, Ekaterinburg, Russian Federation.

The structure and phase transformations of polycrystalline orthorhombic Ti-22%Al-26%Nb, Ti-25%Al-22%Nb (initial phase O-phase, small content of B19 phase), Ti-19%Al-8%Nb, Ti-18%Al-8%Nb (initial phase O-phase, large content of B19 phase) alloys after severe plastic deformation by shear under pressure and pulse shock loading with steel plate were studied. It was found that severe plastic deformation of orthorhombic Ti2AlNb alloys caused phase transformations of the displacive type and associated with a change in the degree of long-range order, namely β2-omega (B2), B2-B19 and B2-beta (BCC) (in the case of alloy with initial B2-phase structure) and O-B19(29% and O-phase structure, Ti2AlN9). Unlike to ordinary metallic, severe plastic deformation of the titanium aluminum intermetallics leads to decreasing of the strength of the material. Upon severe deformation by shear under pressure of the orthorhombic alloys, as the degree of deformation increases, the content of the B19 phase disordered for niobium decreases, whereas the content of the completely disordered...
A20 phase content increases. At a grain size of 20 nm, the initial orthorhombic O-phase is completely transformed into the A20 phase. Fine plate-like O-phase were formed in the magnetic intermetallic alloys with initial O-phase after shock wave loading. The ways of improving the room temperature plasticity of the titanium alloys are discussed.

S5.52 Mechanical Behavior of a Pt-Cr Jewelry Alloy Hardened by Nano-Sized Ordered Particles, Kamili Jackson, Melani Nzula and Candido Long, Chair - Materials Engineering, University of Cape Town, Cape Town, South Africa.

Annual platinum jewelry sales exceed a billion dollars but South Africa, the largest supplier of platinum, exports minimal jewelry. UCT aims to change this by increasing the scientific knowledge of platinum-rich alloys. Mechanical testing as related to microstructure and processing can provide properties useful to both research and industry. Platinum jewelry alloys are based on ordered systems that have not been well characterized. The phase diagrams are often incomplete and mechanical data is missing. The focus of this research is to provide mechanical data of a Pt-Cr alloy by small-scale tensile testing. The materials engineering of platinum jewelry is interesting because only 5wt% can be used for alloying in order to maintain hallmarking. However, pure platinum is very soft and must be alloyed in order be used effectively as jewelry. In several binary systems an increase in hardness has been found after cold working and annealing at low temperatures. The hardening in these alloys has shown to be a result of nano-sized ordered particles. In particular, the existence of the nano-sized particles has been confirmed for a Pt-Cr alloy by TEM. Extensive work has been done on the Pt-Cr alloy to understand the crystal structure and mechanisms of the ordered phase. Hardness tests were performed to measure mechanical properties after different treatments. Unusually, most platinum researchers have used hardness tests as the only measure of mechanical properties. A new small-scale tensile testing machine has made the measurement of other mechanical properties economically possible. An 8mm long specimen is used, which significantly reduces the cost of the specimens. Mechanical properties other than hardness are important to jewelers since they determine how easily the alloys will be formed, how well they can be polished and resist scratches, and how well the final piece will hold stones. It also gives basic data that scientists can understand and relate to the hands-on experience of jewelers. In addition, researchers can use mechanical data as selection criteria when choosing alloys for future work. The information gathered on the microstructure of the Pt-Cr binary alloy combined with the results of the mechanical studies will give a more complete picture of behavior to both jewelers and researchers. Preliminary tests on an annealed and cold rolled Pt-Cu alloy show a clear relationship between strength and hardness. In addition, data like strain to fracture and strain hardening was also obtained. It will be helpful to see if the hardening in the Pt-Cr alloy will affect other properties like ductility and strain hardening. It is expected that the increased strength will have little effect on the ductility of the cold rolled specimens but elastic strain energy will be improved. Considering that a finished item is cold worked but a higher hardness is useful this will be an excellent result.

S5.53 Formation of Plate-Like Tetragonal Tau Phase in MnAl-C Alloys, Alla S. Sologubenko1, Helge Heinrich2, Peter Mueller3 and Gernot Kastner2,3,1 Angewandte Physik, ETH Zurich, Zurich, Switzerland, 1AMPAC, University of Central Florida, Orlando, Florida.

The metastable tau phase of MnAl is of interest for its magnetic properties. The tau phase forms from the supercooled high-temperature epsilon phase. The epsilon-to-tau transformation has been studied by transmission electron microscopy with the goal of clarifying details of the microscopic mechanisms of nucleation and growth of the metastable tau phase in MnAl-C alloys. Small amounts of C (within the solubility limit) were added to delay the decomposition of both tau and epsilon to the equilibrium phases beta and gamma. The plate-like tau phase (L10 structure) of MnAl forms from the epsilon phase (A3 structure) in a step-like process. The displacive mode of the plate-like tau formation results in definite crystallographic relationships between the matrix epsilon and the product tetragonal tau phase. The crystallographic evidence is transmitted only by the intermediate epsilon-prime phase (B19 structure). The structural domains of epsilon-prime nucleate and grow coherently within the epsilon matrix. As epsilon-prime domains evolve, elastic strains increase, and the epsilon-prime intermediate domain makes the formation of stacking faults reduces these strains. A shuffling of the stacking faults by motion of partial dislocation within a single epsilon-prime domain diminishes the elastic strains further. As a result, polypeptide appears. The transformation field is limited to a single epsilon-prime domain. Finally, the epsilon-prime domain boundaries are overcome by the collective motion of transformation dislocations which form twinned tau plates. The epsilon-prime domain boundaries are nucleation sites for successive transformation steps and limit the spatial extension of the transformation for the intermediate steps.

SESSION S6: Other Intermetallics and Titanium Aluminides I

Chairs: F. Appel and J. M. K. Wiezorek
Wednesday, December 1, 2004
Room 208 (Hyne)

8:30 AM S6.1
Alloy Design Concept using TCP and GCP Intermetallics for Austenitic Heat Resistant Materials, Masao Takeyama1,2, Material Science and Engineering, University of Michigan, Ann Arbor, Michigan; 2Materials and Ceramics Science, Tokyo Institute of Technology, Tokyo, Japan.

Why there are no austenitic heat-resistant steels strengthened by intermetallic compounds? For energy efficiency and environmental protection, there is a strong demand for developing new austenitic steels applicable to steam turbine components beyond state-of-the-art USC power plants. This talk will provide some ideas for the alloy design how we use intermetallics as a strengthening for the development of a new class of austenitic steels to be used above 973 K. TCP and GCP compounds are of great potential to achieve this goal. Most of transition metals M form both Fe2M Laves (TCP) and Ni3M (GCP) compounds, so that the phase equilibria among these compounds together with γ-(Fe, Ni) austenite phase at elevated temperatures in Fe-Ni-M ternary systems are extremely important for design of multi-component alloys. In TCP and GCP compounds as a strengthening factor, the partition coefficient of Ni between γ and Fe2M phases is a key factor, and it has to be large enough to stabilize γ against both Fe2M and α-Fe, since M is in general a stabilizer. However, very limited studies on the phase equilibria have been reported. We reveal that the single-phase region of Fe2Nb with hexagonal C14 structure extensively penetrates into the ternary field toward equi-niobium concentration direction, and more than 40at% Ni can dissolve into it at 1473 K. This result makes it possible to disperse the Laves phase finely in the γ matrix by controlling the C/α ratio of C14 through alloying in Fe-Ni-Nb-Cr alloys. For GCP, a Kurnakov type compound of Ni3V with D022 structure is the most stable, because of relatively high disordering temperature (1350 K) as well as the lattice parameters with respect to the disorderingfcc: aD022 = aC14 (C2D022/2). The volume fraction as well as coherent morphology of the D022 phase in the two-phase alloy can widely be changed through the fcc→D022 phase transformation and misfit control. The knowledge can apply to the multi-component system by replacement of Ni with Fe and V with Nb, making it possible to design a new class of austenitic steels. The detail of the alloy design concept together with experimental results will be presented.

9:00 AM S6.2
RuAl-Ru Enticements, Todd Reynolds and David Johnson, MSE, Purdue University, W. Lafayette, Indiana.

The fracture toughness of two phase alloys of RuAl-Ru(Mo,Cr) processed by arc-melting then directionally solidifying using a cold crucible Czochralski technique or a modified Bridgman process were investigated. Due to different amounts of aluminum loss, volume fraction of phases varied. The fracture toughness values for the B2-hcp alloys ranged from 23 to 38 MPasqrtm. Increasing the volume fraction of RuAl was found to decrease the fracture toughness. The oxidation resistance was poor at 1100°C for the RuAl-Mo alloys but was considerably better for the RuAl-Mo-Cr alloys. Mechanical property data from compression and four point bend tests as well as oxidation tests will be reported for alloys of these systems.

9:15 AM S6.3
Formation and Morphology of Kurnakov Type D022 Compound in Disordered fcc γ-(Ni, Fe) Matrix Alloys, Akira Suzuki1 and Masao Takeyama2, 1Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan; 2Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo, Japan.

Ni3M type GCP compounds are attractive as strengtheners in heat-resistant austenitic steels as well as Ni base superalloys. In this study, formation and morphology of D022 compound Ni3V in γ-(Fe, Cr) alloys were investigated as a model case in order to understand fundamentals for microstructure control of new class of austenitic steels. Because of a Kurnakov type compound, Ni3V becomes disordered γ-fcc phase above 1278K (1545 K at stoichiometric composition). The formation of the D022 phase in γ matrix varies, depending on the composition and heat treatment temperature; precipitation of D022 takes place at temperature above

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Here, the topological anisotropic material l00l’m. by
C. H. Chester Lo, Y. Y. Ge, S. S. Mao, and A. Conti1, 2; 1LEM, CNRS-ONERA, Chatillon, France; 2Materials Science Division, Bhabha Atomic Research Centre, Mumbai, India; 3CEMES, CNRS, Toulouse, France.


In polycrystalline lamellar Ti-47Al-1.1C-0.2Si, compression tested at RT and 600°C, deformation is achieved by ordinary dislocations and twins. Alloys cooled down from 1400°C at about 15°C per minute exhibit one prevalent variant O accompanied by its twin-related variant OT yet under a lesser proportion (average lamellar thickness 110 nm). In such a microstructure, the propagation of strain is explained by the notion of pilot and driven orientations. In a number of situations of adjacent pairs of variants O/OT, deformation takes place along one variant of these pairs, either O or OT, to form the slip system that is the most favoured under the applied stresses. This slip plane is unique irrespective of the thickness of the lamellae; the nature of the favoured slip system is also independent of lamellar environment including adjacent alpha2 lamellae. In case of multiple slip families as is the case of the present investigation, the operating slip plane is not necessarily the slip system with lowest critical resolved shear stress. When ordinary dislocations are activated by the applied stress in variant O, then slip occurs by ordinary dislocations in variant OT too with the peculiarity that this is not necessarily the slip system with lowest resolved shear stress within that family. The variant that responds according to the Schmid law is regarded as the pilot variant, its companion twin-related variant is dubbed the driven.

The property holds true whether the operating Burgers vector is parallel or inclined to the interface. The same behaviour is encountered for twinning deformation. It is the pilot orientation in the same way. An additional property is that twinning and not ordinary slip, is activated in the driven variant even though the resolved shear stress for ordinary slip is larger than its critical resolved shear stress. Finally, a property common to both welding and twinning is that the operating slip systems are in mirror orientations in the pilot and driven variants and that includes the slip directions. In particular the orientation of the \(\langle110\rangle\) slip direction to the interface (parallel or inclined) is conserved.

Processing of titanium aluminides often starts with the break-down of the coarse-grained lamellar microstructure of the ingot. The lamellar microstructure consists of alternating layers of \(\gamma\)-TiAl and \(\alpha_2\)-TiAl. After large compressive deformation at temperatures above 1000°C samples were analyzed by means of scanning electron microscope. The lamellar colonies show, depending on their orientation, more or less marked buckling-type deformation modes according to an instability mechanism big wave length. These two deformation modes interact in such a way, which leads to a very inhomogeneous deformation pattern. In order to simulate the microstructural development during hot working we have established a finite element unit cell model, which is a representative volume element (RVE). The model describes the deformation behavior of the material on a micro-, meso- and macroscopic level. The microscopic level represents the local deformation behavior inside single lamellar colonies taking into account the anisotropic deformation behavior of the \(\gamma\)-TiAl lamellae. At the mesoscopic level six grains are considered to describe the material behavior over a length scale of 100μm. Here, the topological anisotropic material behavior is described by means of Hill-plasticity, which combines the anisotropy due to the existence of the lamellae with the anisotropy of the \(\gamma\)-TiAl lamellae. The numerical model is able to reflect the very inhomogeneously deformed microstructure and demonstrates the concentration of strain in bands of different amounts of localized shear. The expected deformation pattern can be well predicted by our numerical findings. To our knowledge, it is the first study on the impact of the imposed deformation energy on the microstructure of TiAl. The experimental and numerical results are in good agreement. Finally, we were able to verify the interaction of all these mechanisms and to quantify their specific contributions.

Finally, an analysis of the local hydrostatic stress state was conducted to calculate the mechanical driving force in addition to the still
existing chemical driving force with respect to an $\alpha_2$-$\text{Ti}_3\text{Al} \rightarrow \gamma$-$\text{TiAl}$ transformation. This phase transformation leads to a spheroidization of the lamellar colonies, this mechanism can be considered as a further softening mechanism.

11:00 AM S6.8

**Effect of B Addition on Thermal Stability of Lamellar Structure in Ti-47Al-2Cr-2Nb Alloys, Yukimori Yamamoto, Philip J. Maziasz and C. T. Liu; Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.**

Thermal stability of fully lamellar microstructure in hot-extruded T-47Al-2Cr-2Nb (at %) alloys with and without 0.15 B has been evaluated for both powder-metallurgy and ingot-metallurgy materials. The lamellar microstructure in the alloy with B becomes unstable after a short period of aging at 800 °C because gamma grains form adjacent to borides (TiB2 or TiB) within the lamellae. The formation of these grains is attributed to less alpha 2 plates around the borides because they scale Ti, resulting in coarsening of the lamellar plates during the early stages of aging. In the case of the alloy without B, however, the lamellar microstructure exhibits higher thermal stability after long time exposure (> 5000 h) at 800 °C, indicating that the existence of borides is the cause of thermal instability of the microstructure. In the presentation, the formation mechanism for gamma grains will be discussed. This research was sponsored by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:15 AM S6.9

**Fatigue Testing of Microsized Samples of $\gamma$-$\text{TiAl}$ Based Material, Timothy P. Halford, Kazuki Takashima and Yakichi Higo; Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Japan.**

High strength $\gamma$-$\text{TiAl}$ based alloys, such as Ti-46Al-5Nb-1W (Alloy7), which were originally developed for gas turbine and automotive applications are now being considered for application in Microelectromechanical Systems (MEMS). This requires the evaluation of these materials upon the microscale. As international standards do not currently exist for the evaluation of the mechanical properties of samples with dimensions equivalent to those required by MEMS devices, the development of an appropriate testing method was required. The method developed here is intended for the fatigue testing of samples measuring $\approx$ 10 mm (B) x 20 mm (W) x 40 mm (L). This is completed using a machine recently developed at Tokyo Institute of Technology to load samples of lamellar $\gamma$-$\text{TiAl}$ based material to failure in compressive bending. This method is intended to work alongside methods previously developed for the fracture toughness testing of similar microsized cantilever bend specimens. In this work sample cantilevers of Alloy 7 are Focused Ion Beam (FIB) machined from foil $\approx$ 20 mm thick and their stress – life ($S$ – $N$) fatigue behaviour evaluated. The dependence of fatigue life upon lamellar orientation for given peak stress / stress ranges is considered. The effect of the reduced scale of these samples upon the mean and scatter of these sample lifetimes is also considered through comparison with previous data obtained from the $S$ – $N$ testing of microsized samples of the same material.

11:30 AM S6.10

**Texture development during hot working in gamma titanium aluminide alloys of different phase constitution.**

Michael Oehring, Fritz Appel, Heinz-Guenter Brokmeier and Uwe Lorenz; Institute for Materials Research, GKSS Research Centre, Geesthacht, Germany.

The evolution of preferred orientations during processing appears to be of significant importance for the use of gamma titanium aluminide alloys, since the desired lamellar microstructures exhibit a strong anisotropy of mechanical properties. In this work texture development has been investigated after hot extrusion and sheet rolling, which are considered to be technologically relevant working processes. As texture evolution certainly is dependent on several factors, including deformation properties, recrystallization kinetics and particularly the phase constitution, processing conditions and alloy compositions were investigated. By comparing the results it is indicated that the determined textures can be understood by considering different modes of the gamma phase. The deformation twinning is an important mode of plastic deformation in g(TiAl) based titanium aluminide alloys. The mechanism apparently compensates for the lack of independent slip systems that can operate simultaneously at given stress and, thus, plays an important role in the development of microstructure, which could at least partially explain the scatter in properties encountered for these alloys. Finally the microstructure yield stress relation will be briefly examined, and the more tenuous link between microstructure and ductility will be broached.

11:45 AM S6.11

**On the Effect of Interstitial Elements on Microstructure and Mechanical Properties of Ternary and Quaternary TiAl Based Alloys.**

Jean-Pierre Chevalier1,2, Melanie Lamirand4 and Jean-Louis Bonnenet1; 1CECM - CNRS, Vitry, France; 2Materiaux Industriels, CNAM, Paris, France.

Interstitial elements, such as carbon, nitrogen and especially oxygen are known to have a significant solubility in alpha2 TiAl, but only slight in the gamma phase. Hence, these elements have a strong influence on the microstructure. Furthermore, many titanium grades have a relatively high initial oxygen content, and due to the high reactivity of these alloys, pickup during processing may also lead to TiAl alloys with typically at least, and often more, 1000 ppm wt oxygen. Using strict clean processing (alloy preparation, melting, remelting, high temperature heat treatment) together with either ultra high purity titanium or commercial purity titanium as starting materials, the effects of interstitial elements on microstructure and mechanical properties (hardness, yield stress and ductility) have been studied systematically. Microstructures have been quantitatively analysed and particular care has been taken to assess relative ductilities of the alloys. Following from previous work on binary alloys, here Ti-Al-Cr ternary and Ti-Al-Cr-Nb quaternary alloys have been studied. The effects of interstitial content have been ascertained both for fully lamellar microstructures obtained by controlled cooling from the alpha phase and for duplex microstructures obtained by heat treatment in the alpha-gamma phase field. The results are clear and similar trends are observed in all cases: oxygen stabilises the lamellar microstructure and affects the kinetics of the alpha-gamma phase transformation, leading to a higher than equilibrium volume fraction of the alpha2 phase for the case of continuous cooling. On the other hand, the volume fractions of alpha2 and gamma are close to equilibrium values for the duplex microstructures. Both the lamellar spacing and the alpha2 volume fraction correlate with increased hardness and yield strength, but with a decreased ductility. The results for oxygen will be briefly compared to those obtained for carbon and nitrogen. Possible mechanisms linking the interstitial element content with the phase transformation kinetics will be discussed. These suggest that the interstitial elements may well play an important role in determining microstructures and that their content should be more strictly controlled in general. Fluctuations in interstitial content, together with differences in cooling rate may well give rise to variations in microstructure, which could at least partially explain the scatter in properties encountered for these alloys. Finally the microstructure yield stress relation will be briefly examined, and the more tenuous link between microstructure and ductility will be broached.
alloys after fatigue testing is very limited. In this paper we report on the TEM characterisation of fatigued Ti4Al48Nb1B with fully lamellar microstructure (with not more than 12% GB gamma grains) and near lamellar microstructure (with about 12% GB gamma grains). These underwent testing at room temperature at 600 and 500MPa respectively with a stress ratio R = 0.1 and a frequency of 8Hz. The TEM was therefore used to characterise their micro-deformation behaviour via dislocations density and slip systems in the different gamma constituent components of the lamellar microstructure. Dislocation densities and slip systems in this microstructure are correlated with the microstructure of the GB gamma in the different lamellar domains. The results are explored with a view to understanding the initial cracking of the lamellar microstructures and the possible effect of grain boundary gamma grains on the cracking behaviour.

2:00 PM S7.3
Impact Properties of Hot-Worked Gamma Alloys with BCC 2:01 Ti Phase, Koutaro Shindo1, Toshimitsu Tetsui1, Toshiro Kobayashi3, Shigeaki Morita1, Satoru Kobayashi4 and Masao Takayama1; 1Nagasaki Research & Development Center, Mitsubishi Heavy Industries, LTD., Nagasaki, Nagasaki, Japan; 2Nagasaki Research & Development Center, Mitsubishi Heavy Industries, LTD. (Present Department : Tohoku University), Nagasaki, Nagasaki, Japan; 3Department of Metallurgy and Ceramics Science, Tokyo Institute of Technology (Present Department : MPI), Meguro-ku, Tokyo, Japan; 4Department of Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo, Meguro-ku, Japan.

Impact damage behavior of a hot-forged TiAl alloy with a composition of 42 to 44 at% Al, consisting of lamellar, g and h grain of which the workability was improved by introducing b-Ti phase. The relationship between the absorbed energy and impact strain rate was investigated using an instrumented Charpy impact test. The strain rate test at high strain rate and foreign object attack test. In instrumented Charpy impact test the absorbed energies for crack initiation and propagation were measured, and the effect of microstructure on the absorbed energies was emphasized. The absorbed energy depends on the microstructure, the fraction of bcc-b-Ti phase and greater than those of the others. The energy for crack propagation is almost comparable to that of the alloy with FL structure and greater than those of the others, whereas the energy for crack propagation is the greatest among others although the value is almost equal to that of the alloy with FL structure. The energy for crack initiation of the forged alloy depends strongly on the microstructure factors. The value increases with reduction of both grain size and interlamellar spacing although the grain size effect is more obvious than that of the lamellar spacing. In summary, the value increases disproportionately in the interlamellar area fraction, and the suitable lamellar area fraction is found to be about 70%. These microstructure factors including the fraction of bcc-b-Ti phase can easily be controlled by processing route (EG). The energy for crack propagation is much larger than that for the crack propagation. The impact properties of the hot-forged TiAl alloy are superior to those of the alloys with various microstructures. The energy for crack propagation is almost comparable to that of the alloy with EG structure which has a microstructure consisting of lamellar microstructure (with not more than 12% GB gamma grains) and near lamellar microstructure (with about 12% GB gamma grains). These underwent testing at room temperature at 600 and 500MPa respectively with a stress ratio R = 0.1 and a frequency of 8Hz. The TEM was therefore used to characterise their micro-deformation behaviour via dislocations density and slip systems in the different gamma constituent components of the lamellar microstructure. Dislocation densities and slip systems in this microstructure are correlated with the microstructure of the GB gamma in the different lamellar domains. The results are explored with a view to understanding the initial cracking of the lamellar microstructures and the possible effect of grain boundary gamma grains on the cracking behaviour.

2:30 PM S7.5
TEM analysis of long-period superstructures in TiAl single crystal with composition-gradient. Satoshi Hata1, Kohjiro Shirishi1, Masaru Fukuda1, Yoishiti Tomokiyo1, Noriyuki Kiyono1, Takayoshi Nakano2 and Yukichi Umakoshi2; 1Department of Material Science and Engineering, Graduate School of Engineering, Osaka University, Osaka, Japan; 2Laboratoire d’Etude des Microstructures, CNRS-ONERA, Chatillon cedex, France.

The microstructure formation in TiAl alloys with Al-rich compositions is complicated and not well understood. This is due to the fact that various long-period superstructures are formed depending on Al concentrations and annealing conditions. In order to study phase relationships and formation mechanisms of the long-period superstructures, the microstructures were investigated using TEM. The microstructure formation in TiAl alloys with Al-rich compositions is complicated and not well understood. This is due to the fact that various long-period superstructures are formed depending on Al concentrations and annealing conditions. In order to study phase relationships and formation mechanisms of the long-period superstructures, the microstructures were investigated using TEM. The results are explored with a view to understanding the initial cracking of the lamellar microstructures and the possible effect of grain boundary gamma grains on the cracking behaviour.

2:45 PM S7.6

Empirical interatomic potentials were used to simulate the atomic structure of the interface between TiAl and Ti,Al surface layers. The models consisted of a one dimensional material at the surface of the single crystal. Transmission electron microscopy (TEM) observations revealed that the following long-period superstructures were formed sequentially in the composition-gradient area: short-range order (SRO), h-Al,Ti, one-dimensional antiphase domain structure (1-APS) and Al,Ti (D022). The SRO structure transforms into Al,Ti superstructure after subsequent annealing at 973 K. All the superstructures except Al,Ti have clear orientation relationships with the L10 lattice. The observed low degree of development of the Al,Ti (D022) superstructure in Ti,Al single crystal with composition-gradient is due to the formation of a binary alloy system that is defined by a particular pair of band structure pairs. This is why the microstructures suggest that anisotropic diffusion processes and a subsequent formation of strain fields in the L10 matrix influence the domain growth of the long-period superstructures.

3:15 PM S7.7
Influence of Micro-alloying on Oxidation Behavior of TiAl. Motohiro Yoshida1 and Shiho Hanagudi; 1Mechanical Engineering and Materials Science, Yokohama National University, Yokohama, Japan; 2Materials Science and Processing, Osaka University, Suita, Japan.

TiAl-based alloys have attractive properties as light weight.
heat-resisting material. Numerous engineering alloys have been developed for structural applications in automotive and aerospace components. Several alloys have been developed containing at least several alloying elements that may optimize thermally stable microstructures and/or improved high temperature mechanical properties. However, the influence of alloying element on oxidation behavior has not been well characterized. The present study will present a wide range of elements on oxidation behavior of TiAl was investigated by micro-oxidation using ion implantation. The ion implantation was carried out with doses of 10^{17} to 10^{19} ions/cm² at acceleration voltages of 30 to 60 keV. The oxidation behavior of the implanted TiAl was investigated by weight gain measurements and X-ray analysis. The results showed the oxidation resistance of the implanted TiAl was improved compared to the unimplanted TiAl. The improvement was attributed to the formation of a protective oxide layer on the surface of the implanted TiAl.

Some of the microstructural features evaluated were grain boundary character distribution, grain size, phase volume fraction, and microstructure. The improvement is limited because of the absence of doping effect. (2) Halogen elements, F and Cl, are similarly effective to improve the oxidation resistance by forming convoluted Al₂O₃ scales, under which adherent Al₂O₃ scales remained. Preferential reaction of Ti with the halogen elements seems responsible for the results. (3) Al and Si show limited improvement, while P shows good result. The doping effect seems to be predominant for the improvement by P. (4) Implantation of Se and Ag enhances the oxidation owing to Al depletion in the modified layer. (5) B, C, N, Mg, Ar, V, Cr and Zr are ineffective for the improvement.

3:30 PM S7.8 Creep of TiAl Alloys at 750°C Under Moderate Stress. Courret Alain and Malaplate Joel; CEMES/CNRS, Toulouse Cedex4, France. The present paper presents a study of creep of two Ti-48Al-2Cr-2Nb alloys processed by cast and powder metallurgy routes at 750°C. Under 150 MPa and 80 MPa, two conditions close to the operating conditions in turbine engines and leading to identical creep rates (10^{-8} s^{-1}). Creep tests were conducted to measure the creep properties and to determine the internal stress and the activation parameters. The deformation microstructures in creep tests up to 0.5 and 2% strain were subsequently investigated in a transmission electron microscope (TEM) to determine the deformation mechanisms responsible for the creep behavior. The creep curves exhibit the usual three stages. The primary stage is more extended for the near lamellar microstructure of the as-cast alloy compared to the powder metallurgy alloy with a duplex microstructure. The secondary stage is very short. In these two alloys, stress jumps are predicted to occur during the second stage. The deformation microstructure is formed in the as-cast alloy through a mixed climb mechanism on the basis of the good consistency of the activation parameters. Furthermore, these values are constant all along the curve, namely in the primary and secondary creep. Internal back stress measured by strain change dip tests is higher in the as-cast alloy than in the powder metallurgy alloy. TEM observation of the creep microstructures shows that the creep deformation is associated with a high level of jog pairs. The deformation microstructure is formed in major by type 2 dislocations, especially after primary stage. From these experimental results, creep is found to be controlled by this mixed climb mechanism. Dislocations are generated at the boundary of the lamellae and propagate in jog pairs. The deformation microstructure is formed in major by type 2 dislocations, especially after primary stage. From these experimental results, creep is found to be controlled by this mixed climb mechanism. Dislocations are generated at the boundary of the lamellae and propagate in jog pairs.

4:00 PM S7.10 Increase in Gamma/Alpha2 Lamellar Boundary Density and its Effect on Creep Resistance of TiAl Alloys. Kouichi Maruyama, Jun Matsuda and Hanliang Zhu; Graduate School of Environmental Studies, Tohoku University, Sendai, Japan. Stability of their lamellar microstructure is crucial for creep resistance of TiAl alloys at high temperatures, but degradation of the lamellar microstructure is unavoidable. Coarsening of lamellar spacing and transformation of lamellae are the major degradation events, and both of them occur by means of lamellar boundary migration. The lamellar microstructures of TiAl alloys contain four types of boundaries. Among the four types, gamma/alpha2 boundary has the highest stability, and sustains the original lamellar microstructure for longer duration. In the present study it will be discussed how to raise density of the gamma/alpha2 boundaries for improving the microstructural stability. The volume fraction of alpha2 phase increases during heating up to temperatures in gamma phase field, and the excess alpha2 phase transforms back to gamma phase during cooling. New gamma/alpha2 boundaries may be added by the heat treatment. The driving force for the gamma to alpha transformation should be large enough for precipitation of alpha2 laths, but too large driving force may introduce alpha2 laths not parallel to the original gamma/alpha2 boundaries. A Ti-48mol%Al alloy was heated to 1550K at several heating rates. The density of gamma/alpha2 lamellar boundaries increased with increasing the heating rate, and creep resistance was confirmed to increase by the addition of the new gamma/alpha2 boundaries. On the other hand, too fast heating rate resulted in unstable lamellar microstructure due to its less aligned lamellar microstructure.

4:15 PM S7.11 Effect of Long-term Aging and Creep Exposure on the Microstructural Stability of a Cast Ti-6Al-4V. Juraj Lapin¹, Mohamed Nazmy² and Marc Staab²; ¹Division of Properties of Materials and Structures, Slovak Academy of Sciences, Institute of Materials and Machine Mechanics, Bratislava, Slovakia; ²Department of Materials Technology, ALSTOM Ltd., Baden, Switzerland. The effect of long-term ageing and creep exposure on the microstructural stability of a cast ABB-2 alloy with nominal chemical composition Ti-6Al-4V-0.5Si (wt.%) was studied. Ductility testing and creep testing the investigated investment cast components in the form of plates, bars and gas turbine blades were subjected to heat treatments. The ageing experiments were performed at temperatures ranging from 793 to 1073 K for various times from 10 to 10^6 h in air. Constant load tensile creep tests up to 25677 h were performed at applied stresses ranging from 150 to 400 MPa and at temperatures 973-1123 K. The creep microstructures of the specimens were characterized by optical microscopy, scanning electron microscopy and transmission electron microscopy. Some of the microstructural features evaluated were grain boundary character distribution, grain size, phase volume fraction, and microstructure. The improvement is limited because of the absence of doping effect. (2) Halogen elements, F and Cl, are similarly effective to improve the oxidation resistance by forming convoluted Al₂O₃ scales, under which adherent Al₂O₃ scales remained. Preferential reaction of Ti with the halogen elements seems responsible for the results. (3) Al and Si show limited improvement, while P shows good result. The doping effect seems to be predominant for the improvement by P. (4) Implantation of Se and Ag enhances the oxidation owing to Al depletion in the modified layer. (5) B, C, N, Mg, Ar, V, Cr and Zr are ineffective for the improvement.

3:30 PM S7.8 Creep of TiAl Alloys at 750°C Under Moderate Stress. Courret Alain and Malaplate Joel; CEMES/CNRS, Toulouse Cedex4, France. The present paper presents a study of creep of two Ti-48Al-2Cr-2Nb alloys processed by cast and powder metallurgy routes at 750°C. Under 150 MPa and 80 MPa, two conditions close to the operating conditions in turbine engines and leading to identical creep rates (10^{-8} s^{-1}). Creep tests were conducted to measure the creep properties and to determine the internal stress and the activation parameters. The deformation microstructures in creep tests up to 0.5 and 2% strain were subsequently investigated in a transmission electron microscope (TEM) to determine the deformation mechanisms responsible for the creep behavior. The creep curves exhibit the usual three stages. The primary stage is more extended for the near lamellar microstructure of the as-cast alloy compared to the powder metallurgy alloy with a duplex microstructure. The secondary stage is very short. In these two alloys, stress jumps are predicted to occur during the second stage. The deformation microstructure is formed in the as-cast alloy through a mixed climb mechanism on the basis of the good consistency of the activation parameters. Furthermore, these values are constant all along the curve, namely in the primary and secondary creep. Internal back stress measured by strain change dip tests is higher in the as-cast alloy than in the powder metallurgy alloy. TEM observation of the creep microstructures shows that the creep deformation is associated with a high level of jog pairs. The deformation microstructure is formed in major by type 2 dislocations, especially after primary stage. From these experimental results, creep is found to be controlled by this mixed climb mechanism. Dislocations are generated at the boundary of the lamellae and propagate in jog pairs. The deformation microstructure is formed in major by type 2 dislocations, especially after primary stage. From these experimental results, creep is found to be controlled by this mixed climb mechanism. Dislocations are generated at the boundary of the lamellae and propagate in jog pairs. The deformation microstructure is formed in major by type 2 dislocations, especially after primary stage. From these experimental results, creep is found to be controlled by this mixed climb mechanism. Dislocations are generated at the boundary of the lamellae and propagate in jog pairs.
electron microscopy before and after ageing and creep deformation. Before ageing and creep testing the microstructure of the samples was nearly lamellar. The nearly lamellar microstructure consisted of \(\alpha_2(\text{TMS3})\) and \(\gamma(\text{TIAI})\) lamellae, coarse \(\alpha_2\) (ordered Ti-based solid solution) particles and small volume fraction of the \(\gamma\)-phase formed along the grain boundaries. The duplex microstructure consisted of lamellar, franny and \(\gamma\)-rich regions. Microstructura analysis revealed that lamellar regions are composed of \(\alpha_2\) and \(\gamma\) lamellae, coarse \(\alpha_2\) B2 particles and fine needle-like B2 precipitates. The feathery regions contain \(\gamma\)-matrix with \(\alpha_2\), B2 and Ti6Si3 particles. Coarse Ti6Si3 particles are identified within the \(\gamma\)-rich regions. During ageing of the samples with duplex initial microstructure, the \(\alpha_2\)-phase in the lamellar and feathery regions transforms to the \(\gamma\)-phase and fine needle-like B2 precipitates. The microstructural instabilities lead to a softening of the alloy. The measured time exponents and activation energies for softening are discussed from the point of view controlled transformations and the coarsening of the coexisting phases. A possible effect of such microstructural changes on microstructure and time exponents is analyzed. The softening produced at designed operating temperature of \(773\,\text{K}\) is very slow and cannot affect significantly mechanical properties of the investment cast components during their long-term service. Special attention is paid to the phase analysis and deformation structure of the creep specimens. Significant differences of one order of magnitude in minimum creep rate and time to fracture of the specimens with different initial microstructure have been established on the basis of deformation structure and microstructural changes affecting the creep deformation processes of the material. The microstructural changes and softening have only negligible effect on the creep strength in the temperature range of interest for industrial applications. Fine precipitates formed due to microstructural instabilities along the \(\alpha_2/\gamma\) lamellar interfaces and within the \(\gamma\)-rich regions are effective obstacles to dislocation motion during creep, which enhance the creep strength.

4:30 PM S7.12
Creep Behavior and Microstructural Stability of Ti-46Al-9Nb Material with Different Microstructures.

Slawomir Bystrzanowski1, Arno Bartels1, Helmut Clemens2, Rainer Geiling3, Frank-Peter Schimansky3 and Gerhard Dehm4, 1Materials Science and Technology, Technical University of Hamburg-Harburg, D-21072 Hamburg, Germany; 2Department of Physical Metallurgy and Materials Testing, Montanuniversitaet Leoben, A-8700 Leoben, Austria; 3Institute for Materials Research, GKSS-Research Centre, D-21502 Geesthacht, Germany; 4Max-Planck-Institut fuer Metallforschung, D-70569 Stuttgart, Germany.

In this paper the creep behavior and the microstructural stability of high Nb containing Ti-46Al-9Nb (in wt%) sheet material were investigated in the temperature range of \(700\,\text{°C}\) to \(800\,\text{°C}\). The study involves three different types of microstructure, namely fully lamellar with narrow lamellar spacing, duplex and massively transformed (massive transformation was obtained by fast cooling from the \(\alpha\) phase field). Short-term creep experiments at \(700\,\text{°C}\) and 225 MPa confirmed that the fine lamellar microstructure with narrow lamellar spacing exhibits a much higher creep resistance when compared to massively transformed and duplex. During long-term creep tests up to 1500 hours stress exponents (in the range of 4.4 to 5.8) and activation energies (of about 4 eV) have been estimated by means of load and temperature changes, respectively. Both, stress exponents and activation energies suggest that under the applied conditions diffusion assisted climb of dislocations is the dominant deformation mechanism. Furthermore, the beneficial influence of a heat-treatment which stabilizes the fully lamellar microstructure on creep has been proven: At \(800\,\text{°C}\) and 150 to 225 MPa significantly reduced primary and secondary creep rates and prolonged time-to-failure were observed compared with samples without such an additional heat treatment. The thermal stability of the fully lamellar microstructure under various creep conditions has been analyzed by means of backscatter electron microscopy and X-ray diffraction. Our investigations revealed considerable stress and temperature induced microstructural changes which are reflected in the dissolution of the \(\alpha\) 2 phase accompanied by precipitation of new Al- as well as Ti/Nb-rich phases situated on the grain boundaries. It was shown that especially the duplex microstructure is prone to such microstructural instabilities.

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Internal Friction of a High Nb Gamma TiAl Alloy With Different Microstructures. Manfred Weiler1, H. Clemens1, G. Dehm1, G. Hansczyk2, S. Bystrzanowski3, A. Bartels1, R. Geiling5 and E. Arzt1, 1Max-Planck-Institut fuer Metallforschung, Stuttgart, Germany; 2Department of Physical Metallurgy, University of Leoben, Leoben, Austria; 3Institute of Materials Science, Silesian University, Katowice, Poland; 4Department of Materials Science, Technical University of Hamburg-Harburg, Hamburg, Germany; 5Institute of Materials Research, GKSS Research Center, Geesthacht, Germany.

An intermetallic Ti-46Al-9Nb (at%) alloy with different microstructures (fine-grained equiaxed, coarse grained fully lamellar, and duplex) was studied by internal friction measurements at 300K to 1200K using different frequency ranges: (I) 0.01 Hz to 10 Hz and (II) around 2 kHz. The loss spectra in range I show (i) a loss peak of Debye type at \(T>H\) \(1000\,\text{K}\) which is only present in fully lamellar and duplex samples; (ii) a high-temperature damping background above \(1100\,\text{K}\). The activation enthalpies determined from the frequency shift are \(H = 2.9\,\text{eV}\) for the loss peak and \(H = 4.2 - 4.3\,\text{eV}\) for the high-temperature damping background. The activation enthalpies for the viscoelastic high-temperature damping background agree well with values obtained from creep experiments and are in the range of those determined for self-diffusion of Al in TiAl. These results indicate that both properties (high-temperature damping background and creep) are controlled by volume diffusion assisted climb of dislocations. The loss peak is assigned to diffusion-controlled local glide of dislocation segment which, as indicated by transmission electron microscopy observations, are pinned at lamellar interfaces. From measurements of the eigenfrequencies in frequency range II the variation of Young's modulus was determined in the temperature range of \(300\,\text{K}\) to \(1200\,\text{K}\). The modulus decreases only by about 15% proving that gamma TiAl-based alloys retain their elastic stiffness up to high temperatures. The Young's modulus of Ti-46Al-9Nb is significantly higher when compared to Ti-based alloys. An influence of microstructure on elastic modulus was not found.