SYMPOSIUM S

Integrative and Interdisciplinary Aspects of Intermetallics

November 29 - December 1, 2004

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* Invited paper
FeCo Intermetallics, Maja Krcmar, Chong Long Fu and James R. Morris; Oak Ridge National Lab, Oak Ridge, Tennessee.

Using first-principles calculations and statistical mechanic 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: antistite defects dominate point-defect structure on both sides of stoichiometry, and the presence of lattice vacancies is negligibly small. We then use dislocation structure analysis to estimate the strain 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 the structural stability limit.

9:15 AM S1.3
Thermal Stress Induced Cracking of NiAl Thin Films Constrained by a Si Substrate, Gerhard Derhm, Hans-Eckhardt Schaefer; Institute of Theoretical and Applied Physics, Stuttgart University, Stuttgart, Germany; 2Physical Metallurgy and Materials Testing, University of Leoben, Leoben, Austria.

It is well established that flow stresses of metallic thin films increase with decreasing film thickness to values significantly exceeding those of the corresponding bulk material. However, it is unclear whether similar size effects are present in the cracking 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 52.4at-percent) and film thickness (400 to 3000nm). The films were thermally cycled to a maximum temperature of 1000°C, 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 even extended into the Si substrate. The fracture toughness of the Al-rich films was determined from the fracture stress, substratac 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 NiAl-Mo Composites, Hongbin Bei, Easoo P. George; 1Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; 2Materials Science and Engineering, The University of Tennessee, Knoxville, Knoxville, Tennessee.

NiAl-Mo ternary eutectic eutectic films 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 size of the Mo fibers decreased, with fibers as small as 400nm in height and 200nm wide observed. Electron backscatter diffraction patterns identified the growth directions and Mo/NiAl interface boundaries to be parallel to <100> and {011}, 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 supported by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy, under contract DE-AC05-000R22725 with UT-Battelle, LLC.

9:45 AM S1.5
Effect of Heat Treatments on Microstructures of Rapidly Solidified TiCo Ribbons, Kyosuke Yoshimi, Akira Yamauchi, Nakamura Ryusuke, Sadahiro Tsurekawa and Shuji Handa; 1Institute for Materials Research, Tohoku University, Sendai, Japan; 2Institute of Polymer Mechanics, AIST, Tsukuba, Japan; 3AIST, Tohoku University, Sendai, Japan.

Effect of heat treatments (aging or annealing) on microstructures were investigated for rapidly solidified ribbons of near-stoichiometric TiCo. In as-spun ribbons, it was observed by TEM that a lamellae 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 873 K for 24 h, grain growth seemed to occur slightly. In addition, a new dislocation density in the annealed ribbons was obviously higher than those in the as-spun 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.

10:05 AM S1.6
Effect of Heat Treatments on Microstructures of Rapidly Solidified TiCo Ribbons, Kyosuke Yoshimi, Akira Yamauchi, Nakamura Ryusuke, Sadahiro Tsurekawa and Shuji Handa; 1Institute for Materials Research, Tohoku University, Sendai, Japan; 2Institute of Polymer Mechanics, AIST, Tsukuba, Japan; 3AIST, Tohoku University, Sendai, Japan.

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Most of the studies aimed at the development of creep-resistant Fe-Al intermetallics have been oriented at application temperatures of the order of 600-650°C, where these materials may compete with conventional stainless steels. The Fe-Al intermetallics are, however, particularly excellent in their oxidation and corrosion resistances at temperatures below 1000°C, where Chromium-Nickel stainless 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 Fe3AI-base 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 700°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 Fe2Nb Laves 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 effects have been discovered in the uniaxial compression testing of micro-size single-crystal samples in Ni3Al alloys containing Ta and Hf, where the flow stress has been observed to increase in proportion to the inverse-square-root of the sample diameter. This behavior results in samples having a 500 μm diameter and a flow stress that is approximately 2 GPa, while the flow stress for bulk samples is smaller by two orders of magnitude. In this talk, we will present these mechanical test results in detail, and also show additional results from the testing of binary Ni3Al and Ni3Al-Ga alloys with Hf and Ga concentration. Collectively, these studies explore the influence of fault energy on the size-affected behavior. Further, there is little connection between observed size effects and the underlying changes to the fundamental deformation mechanisms governing dislocation-mediated flow. Primarily because of the challenges in preparing transmission electron microscope (TEM) foils from micro-tensile test specimen. There is considerable evidence that at temperatures in the anomalous flow regime, the mobility of screw-character dislocations is greatly influenced by the lateral motion of large jogs and kinks along the length of the dislocations, i.e., it is likely that dislocation kinetics are strongly influenced by the characteristic active line length of dislocations known to be on the order of a few microns. We will present the results of TEM analysis of deformed microsamples from these alloys, in order to gain a better understanding of the effect that artificially truncating the sample dimensions has on the fundamental micromechanisms of dislocation glide, storage, and multiplication.

Effect of Allloying Elements (Ga, Ge, Si) on Pseudoelectricity in Fe3Al Single Crystals. Hirohito Y. Yasuda1, Takashi Kose2 and Yukiichi Umakoshi2. 
1Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, Ibaraki, Japan; 2Materials Science and Engineering, Osaka University, Suita, Japan. 

Pseudoelectricity 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-25at%Al and Fe-21at%Al-5at%X (X=Ga, Si, Ge) were grown by a floating zone method. In Fe-25at%Al single crystals, superpartial dislocations with Burgers vector (b) of /\(\frac{1}{2}\{111\}\) moved dragging APB during loading, while APB pulled back the dislocations during unloading. This resulted in giant pseudoelectricity 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-25at%Al. In contrast, both Si and Ge additions decreased the amount of shape recovery. Stress at which the shape recovery started, was increased by Ga, Si and Ge addition. 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 /\(\frac{1}{2}R\) in Fe-25at%Al and Fe-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 /\(\frac{1}{2}R\{111\}\) played an important role in the individual motion of the superpartials with /\(\frac{1}{2}R\{111\}\). Therefore, the fine domain structure was found to be favorable for giant pseudoelectricity in Fe3Al single crystals. Ga addition increased the APB energy following the superpartials 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 through thickness measurement. Based on stress-temperature data for the apparent onset of plasticity during repeated thermal cycling, the yield stress was seen to clearly decrease (from 1380 to 568 MPa) with increasing temperature (from 178 to 373°C). To further improve the Ni3Al films in this thickness range, disordered, phases is important to improve the microstructures, that, and higher. These apparently particles are sheared via the formation of Magnetic phase shows ferromagnetism. Therefore, changes in size and Lh of, D0 24 (Ni3 Ti), 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.

2:15 PM S2.3
1Materials Science and Engineering, Osaka University, Suita, Japan; 2Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, Ibaraki, Japan.

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 disordered phases is important to improve the 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 were 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 these deformed crystals was examined. Single crystals were grown from the arc melted Ni-18 at%Al-4 at%Ti ingots by a floating zone method and were homogenized at 1423K for 168h. After solution treatment at 1423K, γ phase was precipitated at 1073K. Specimens with a loading axis of <110> for fatigue test were prepared 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 deformation mechanism will be discussed.

2:30 PM S2.4
Shear Mechanisms of the γ’ Phase in Superalloys at Intermediate Temperatures. Michael John Mills1, Gopal B. Viswanathan1, Peter Sarosi1, Raymond Unocic1 and Deborah Whitis2.
1Materials Science and Engineering, The Ohio State University, Columbus, Ohio; 2Materials and Processes Engineering Department (MPED), 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 strengthening phase in the technologically important superalloys. In fact, the principal γ’ shearing mechanism in superalloys at lower temperatures was a failure to develop a description of the process observed in single phase Ni3Al. However, at intermediate temperatures (in the range from 873-1073K), it is now abundantly clear that the γ’ shearing mechanisms in superalloys are quite different from those in single phase. Studies of structures following creep of the superalloys Rene 88TD and M35 in the supersolvent condition with bimodal γ’ size distributions reveals at least two distinct shear mechanisms. In coarse γ’ microstructures, and higher γ’ volume fractions, the formation of superlattice extrinsic stacking faults, with isolated <\frac{1}{3}<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 1<\frac{1}{3}<110> 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 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 Ni3Si-Ni3Ti-Ni3Nb-Based Multi-Intermetallic Alloys. Takuji Nakagui, Koji Ohira and Yasuyuki Kaneno.
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-Ni3Ti-Ni3Nb pseudo-ternary system were investigated. As the constituent intermetallic phases, L12(Ni3Si), D022(Ni3Ti), DO19(Ni3Nb) and D021(Ni3Ti7Nb3) 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), D022(Ni3Ti) and D021(Ni3Ti7Nb3) 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-Ti3Ni-Ni3Nb and Ni3Al-Ni3Ti-Ni3V-based multi-intermetallic alloys.

3:15 PM S2.6
Atomatic 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 in atomistic computer modeling of intermetallic compounds of the Ni3Al and Ti-Al systems. Atomic interactions in these systems are modeled by semi-empirical potentials fit to 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.

3:45 PM S2.7
Physics & Astronomy, Northwestern University, Evanston, Illinois.

The microstructural mechanisms governing the deformation and fracture behavior and anomalous mechanical response of intermetallic alloys are still far from being well understood and remain the 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 potentials fit to both experimental and 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 applications. These alloys which include Ir3X, Rh3X (X = Ti, Zr, Hf, V, Nb, Ta) and PtxX (X = Al, In, Ga), posses very high melting temperatures and superior environmental properties. The two-phase PGM-based superalloys are considered as a replacement for single-phase Nb-based superalloys for ultra-high-temperature applications. 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 also 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 → D01 for Ir(Re)1-x, D02 for Pt3X. Supported by the AFSOR (grant No. F49620-01-1-0165).

4:00 PM 52.8
A Bond-Order Potential Incorporating Analytic Screening Functions for the Molybdenum Silicides, Marc J. Cawkwell, Matous Mrovčík, Duc Nguyen-Maň, David G. Pettifor and Vaclav Vitek; 1Department of Materials Science and Engineering, University of Pennsylvania, Philadelphia, Pennsylvania; 2Institute for Energy and the Environment, Freiburg, Germany; 3UKAEA Fusion, Culham Science Centre, Abingdon, United Kingdom; 4Department 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 relatively high temperatures. The limited ductility of MoSi2 in ambient conditions along with the anomalous temperature dependence of the CRSS of the (110)[111], (011)[100] and (010)[100] slip systems and departure from Schmidt law behavior of the (013)[C31] 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, N-body formalism that are 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 on the basis set. The BOP core can be expanded to describe more extended defects such as dislocations. We have therefore developed a screening function for MoSi2 that includes repulsive interactions due to the non-orthogonality of orbitals in our orthogonal TB model. The BOP is found to be an excellent description of cohesion in C114 MoSi2 and we have also considered its transferability to other related crystal structures and stoichiometries, notably C40, C49 and C54 MoSi2, A15 and D09 Mo5Si3 and D8h Mo3Si1 by comparing with abinitio 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.

4:15 PM 52.9
Role of Microstructure in Promoting Fracture and Fatigue Resistance in Mo-B Alloys, Jame Kruzic, Joachim Schneble and Robert Ritchie; 1, 2Department of Materials Sciences, Lawrence Berkeley National Laboratory, Berkeley, California; 3Department of Mechanical Engineering, University of California, Berkeley, California; 4Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; 5Department of Mechanical Engineering, Oregon State University, Corvallis, Oregon.

Intermetallic based Mo-Si-B alloys consisting of α-Mo, Mo5Si3, and Mo2SiB2 (T2) phases have been targeted for high temperature turbine engine applications. To reach this goal, adequate resistance to oxidation, creep, fracture, and fatigue, must be achieved. However, microstructural features which promote improvements in one property are often detrimental to others. Accordingly, a thorough understanding of how microstructure affects each property is needed so that appropriate trade-offs can be made in the optimization of these alloys. To further this goal, an investigation of how microstructural features affect the fracture and fatigue properties at ambient to high temperature is presented. Specifically, the effect of microstructure on toughness and fatigue-crack growth resistance has been investigated from 25 to 1300°C for several Mo-Si-B alloys with both intermetallic and α-Mo matrix microstructures. These alloys were produced by both ingot and powder metallurgy processing routes with compositions nominally as Mo (bal.), 12-20 at% Si, 8-10 at% B. The role of microstructural variables, including volume fraction of α-Mo, its ductility, and the morphology and coarseness of the microstructure are considered. The influence of how each variable influences the observed toughening mechanisms, identified to include crack trapping, crack bridging, and in some cases microcrack toughening. Additionally, fatigue-crack growth behavior, and associated mechanisms, are assessed, again in terms of microstructural variables. Work supported by the Department of Energy, through the Office of Science (Basic Energy Sciences) under Task Order No. DE-AC03-76SF00098, and the Office of Fossil Energy (Advanced Research Materials) under Contract No. DE-AC05-00OR22725.

4:30 PM 52.10
The Effects of Substitutional Additions on Tensile Behavior of Nb-Silicide Based Alloys, Iliyan Kostov, Bernard P. Bowley, Melvin Jackson and Ann Ritter; General Electric Global Research, Schenectady, New York.

Nb-silicide based in-situ composites consist of a ductile Nb-based solid solution with high-strength silicides, and they show excellent promise for aircraft engine applications. The Nb-silicide controls the high-temperature tensile behavior of the composite, and the Nb solid solution controls the low-temperature behavior. The aim of the present study is to understand the effects of substitutional elements on the constitutive properties of the phases. These data are then used to understand the effects of the scale of the phases and their volume fractions, on the tensile behavior of Nb based silicide composites. To pursue this goal alloys with a range of compositions were prepared and the tensile behavior was investigated from room temperature to 1200°C. The experimental measurements will be compared with predictions for the Nb-Si composite strength.

4:45 PM 52.11
Microstructural Relationship to Mechanical Properties of LENS Deposited Nb-Si Alloys, Ryan R. Dollhoff, Peter Collins, Peter Sarosi, Hamish L. Fraser and Michael J. Mills; Materials Science and Engineering, The Ohio State University, Columbus, Ohio.

Nb-Si “in-situ” matrix 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 LENS3M (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 technology result in a reduction in microstructural scale over conventional alloy processing. In addition, the Nb-Si composite strength. 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 S9: Functional Intermetallics I
Chair: T. J. Balk and G. Delin
Tuesday Morning, November 30, 2004
Room 208 (Hynes)

8:30 AM 753.1
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 micro-manipulators. 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 other mechanical properties resulted 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.
thin films realized tensile tests, while bulk alloys are so brittle that they have not been tensile-tested. The addition of a third element such as Cu and Pd was found to improve the shape memory behavior similarly in both bulk alloys. As a special issue in this thin films, the thickness effect of mechanical properties was also investigated, focusing on the effects of surface oxidation and thickness-to-grain size ratio. Finally, some applications of shape memory thin films such as a microgripper, a microconveyor, and a microrobot are demonstrated.

9:00 AM S3.2 Mechanical Properties of TiNiPd Melt Spun Ribbons with Various Heat Treatments. Tomonari Iinuma1, Yohei Takahashi1, Hideki Hosoda1, Kenji Wakahama1, Takeshi Naga2, Takayoshi Nakano2, Yukichi Usmoski1 and Shuichi Miyazaki 2; 1Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; 2Department of Materials Science & Engineering, Graduate School of Engineering, Osaka University, Suita, Osaka, Japan; 3Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki, Japan.

Mechanical properties of Ti Ni containing 10 mol% Pd melt-spun ribbons were investigated where the heat treatment temperature was systematically changed from 773K to 273K. A hot-forged bulk TiNiPd 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 TiNiPd melt-spun ribbon is 60K 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 martensite for the martensite for the TiNiPd melt-spun ribbons instead of B2 phase parent for the hot-forged bulk material. By mechanical tests at temperatures from room temperature to 373K, the superposition of superelasticity is 15% confirmed for the melt-spun ribbons when the temperature is above the reverse martensitic transformation finish temperature. The stress for inducing martensitic transformation is discovered in terms of heat treatment condition. Microstructures of the TiNiPd melt-spun ribbons observed by transmission electron microscopy are also presented.

9:15 AM S3.3 Laser Annealing of Shape Memory Alloy Thin Films: Microstructure Investigation. Xi Wang1, Joost J. Vlasses1, Fadila Khelfoufi2 and Yves Bellouard3; 1Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; 2DTSI/LIST, CEA, Fontenay-aux-Roses, France; 3Center for Automation Technologies, Konselser Polytechnic Institute, Troy, New York.

For a decade, shape memory alloys thin films have been considered for application as actuators in numerous MEMS devices. However, the lack of information on the laser annealing process has plagued their broader acceptance. Laser Annealing of Shape Memory Alloy (LASMA) amorphous thin films has recently emerged as a powerful method to bypass this limitation. Instead of using high-temperature annealing or a post-deposition anneal in a vacuum furnace, the Laser Annealing process consists of using a continuous laser beam to selectively crystallize an amorphous film in specific zones where shape memory properties are desired. In order to fully explore the nature of interfaces between amorphous and crystallized zones as a mean to introduce a TWSME, it is essential to investigate the microstructure of laser-annexed films. In this paper, 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 UV 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.

9:30 AM S3.4 Factors to Control the Martensitic Transformation Temperature of TiNi Shape Memory Alloy Containing Ternary Elements. Hideki Hosoda1, Kenji Wakahama1, Shuichi Miyazaki2 and Kanryu Inoue3; 1Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; 2Institute of Materials Science, University of Tsukuba, Tsukuba, Ibaraki, Japan; 3Department of Materials Science and Engineering, University of Washington, Seattle, Washington.

Ternary alloying elements are generally added to TiNi shape memory alloys in order to control the martensitic transformation behavior. It is believed that the martensitic transformation temperature of TiNi is influenced by the electron atom ratio (e/a). However, other factors such as atomic size and electron hole number are not systematically considered. In this paper, we have systematically investigated the transformation behavior of ternary TiNi with a similar heat treatment condition, and the change of martensitic transformation temperature is analyzed by several factors such as the number of electrons (n + d and so on), atomic size, atomic volume and electron hole number. On the analysis, substitution behavior of ternary elements is taken into account. It is revealed that martensitic transformation temperature of TiNi is mainly correlated with the electron hole number of ternary elements, and also martensitic transformation temperature shows a tendency to increase with increasing the atomic volume of ternary elements. Other shape memory alloys such as TiPd are also discussed similarly.

9:45 AM S3.5 Creep Formation of Supra-Elastic Porous NiTi for Biomedical Applications. Scott M. Oppenheimer1, Christopher Green2 and David C. Dunand2; 1Dept. of Materials, Northwestern Univ., Evanston, Illinois; 2Institut fuer Metallkunde, Universitat Stuttgart, Stuttgart, Germany.

Argon gas was trapped within a NiTi matrix during hot consolidating of NiTi powders. Upon subsequent heating to 120°C, creep of the surrounding NiTi matrix allows the Ar-filled pores to grow up to an average size of 20–90μm, resulting in porous NiTi with a total porosity of about 59%. This foaming process is modeled by axisymmetric and three-dimensional finite element models. Porous NiTi specimens were mechanically tested and found to be superelastic at room temperature. Possible applications for porous NiTi include bone-replacement implants and energy-absorbing, smart sandwich structures.

10:15 AM S3.6 Advanced TEM Investigations on Ni-Ti Shape Memory Material. Dominique Schryvers, Wim Tirry and Zhigang Yang; EMAT, Physics, University of Antwerp, Antwerp, Belgium.

The fundamental aspects of the shape memory behaviour of NiTi materials are still not completely understood. In the present talk we will highlight some of the recent results that we have obtained when investigating this material with transmission electron microscopy (TEM) techniques. The atomic structure and internal lattice defects of the precipitates are documented by high-resolution transmission electron microscopy (HRTEM) and the atomic positions are refined by the multi-slice least-squares (MSLS) technique based on the intensities of superreflections measured on CCD. The results are compared to density functional theory (DFT) calculations. HRTEM combined with MSLS techniques was also used to measure the strain fields in the austenitic Ni54Ti46 matrix surrounding coherent lens-shaped Ni4Ti3 precipitates. Atomic lattice images were obtained in the [1-10]B2 and [10-1]B2 zones and the interplanar distances of the {110}B2 planes are used to determine the strain. These measurements were done for small precipitates with a diameter around 50nm and for larger ones with a diameter of 300nm. From these observations the amplitude and location of the maximum strain in the surrounding matrix could be determined and whether it is compressive or tensile. Also the interaction between strain fields from different precipitates is investigated. A simple model for the strain distribution based on the observations is proposed, in which the maximum strain is not localized at the matrix-precipitate interface but at a distance from it. The measurements and model are compared to the analytical solution based on the Eshelby approach. In parallel with the structure investigations the existence of concentration gradients in the B2 matrix surrounding Ni4Ti3 precipitates or close to a grain boundary is investigated with energy dispersive X-ray analysis (EDX), electron energy loss spectroscopy (EELS) and energy filtered TEM (EFTEM). In some cases very small concentration deviations from the nominal values can be measured, but such deviations, if existing, often fall under the detection limit of all three techniques applied. Both matrix deformations and concentration gradients will influence the nucleation and growth of martensitic plates and variants active during the shape memory behaviour.

10:45 AM S3.7 Variation in Ferroelastic Transition of Shock-Compressed NiTi. Takamitsu Kurita, Hisashi Matsumoto, Kentaro Motoki, Kozo Ojima and Hiroshi Abe; Materials Science And Engineering, National Defence Academy, Yokosuka, Japan.

On impact of the flyer plate at a high velocity, a high shock pressure with a duration time of 10–6 second is generated by using a powder gun. We applied this high pressure to a NiTi matrix containing Nb. As far as NiTi is well known to show an excellent shape memory effect. Then, the effects due to the propagation of a shock wave was investigated on the phase transition in NiTi, because the shock compression is expected as a natural processing for synthesized materials and micro-structure, etc. In this work, the maximum shock pressure was about 20GPa, as the projectile was impinged on NiTi plate at the velocity of about 1km sec–1. The differential scanning calorimeter revealed that in the NiTi shock treated the temperature range of the
shape transition was expanded and the thermal hysteresis between the high temperature phase and the low temperature phase was narrow, in comparison with a fully disordered non-treated sample. During transmission electron microscopy, we have characterized the microstructure in each phase. The phase transition depends on defects induced through the propagation of a shock wave.

11:00 AM S3.8
Shape memory effect through L₁₀-fcc order-disorder transition, Katsumi Tanaka, Advanced Materials Science, Kagawa University, Takamatsu, Kagawa, Japan.

Shape memory effect not associated with martensitic transformation but with order-disorder transition has been examined in the compounds AuCu, CoPt and FePd. For single crystal specimens, a reversible shape change (reduction and elongation in edge length) is observed at the transition temperature by cooling and heating under a uniaxial compressive stress along the [001] direction. The magnitude of the shape change is directly comparable with the change in the magnetization at the transition; a mono-variant ordered specimen has formed under a compressive stress. For polycrystal specimens, similar but small shape change have been observed. As an application of the compounds, a trial actuator consisting of FePd wire and conventional inconel wire has been constructed. The actuator has properly functioned with heat cycles.

11:15 AM S3.9

The L₁₀-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 magneto-crystalline 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 FePt alloys are used as model systems to investigate the major aspects of the processing-structure-property relationships in this class of L₁₀-ordered ferromagnets during the latter stages of order annealing after cold-deformation. Depending on the details of the processing parameters employed during the combinations 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 equiaxied 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 L₁₀-FePd intermetallics. Implications for bulk-processing of FePd-based and other L₁₀-ordered intermetallic compounds 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 L₁₀-Ordered FePd, Wolfgang Pfeier¹, Chaisak Isaro², Wolfgang Puech³, Peter Frauz Rogi⁴, William A. Softa⁵, Rafał Kozubski⁶, Guy Schmerber⁷ and Veronique Pierret-Bories⁸, ¹Materials Science, University of Vienna, Vienna, Austria; ²Physikalische Chemie, Universität von Vienna, Vienna, Austria; ³Materials and Engineering University of Pittsburgh, Pittsburgh, Pennsylvania; ⁴M. Smoluchowschi Institute of Physics, Jagellonian University, Cracow, Poland; ⁵IPCMS-GEMME, CNRS-ULP, Strasbourg, France.

L₁₀-ordered intermetallics are promising high density magnetic recording materials. A common reason is the high ferromagnetic anisotropy with 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 (thickness 300 nm). 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 disordered 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 10 μm) and the film orders L₁₀ 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-axes into the film plane. This surprising effect is in correspondence with recent Monte-Carlo simulations of ordering kinetics in L₁₀-ordered nano-multilayers.

11:45 AM S3.11
Strain-Induced Ferromagnetism in Single Crystal Intermetallic Compounds, Ian Baker¹, Dongmei Wu¹, Markus W. Wittmann² and Paul R. Munroe³, ¹Thayer School of Engineering, Dartmouth College, Hanover, New Hampshire; ²Electron Microscope Unit, University of New South Wales, Sydney, New South Wales, Australia.

Recent research has indicated that the strain-induced paramagnetic to ferromagnetic transition in FeAl arises mostly from the generation of APB tubes, where Fe atoms can have 2d-like nearest neighbors. The resulting saturation magnetization, Mₛ, depends on both the Fe:A1 ratio and 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 ions in APB tubes. The behavior of both ternary FeAl-based alloys and of the L₂₁-structured intermetallic compound Fe₂AlMn 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 Fe₂Al and Fe₂AlMn 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 L₁₀ intermetallics are also outlined. Research sponsored by NSF grant DMR 9973977 and NIST grant 60NANB2D0120.

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

1:30 PM S4.1

Sub-nano and nano-structures of intermetallics such as LaNi₅ 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 dehydrogenation under constant pressure using positron annihilation technique. It was found formation of remarkably dense dislocations and vacancies in the lattice of intermetallics and their hydrides. However, the crystallite size of the alloys studied did not change during hydrogenation and dehydrogenation.

2:00 PM S4.2
Characterization and Catalytic Properties of Ni₃Al for Hydrogen Production from Methanol, Ya Xu, Satoshi Kameoka, Kyoosuke Kishida, Masahiko Demura, An-pong Tsai and Toshiyuki Hirano, Materials Engineering Laboratory, National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

Ni₃Al 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 Ni₃Al were rarely investigated since the leaching of aluminum from Ni₃Al is difficult compared to NiAl and Ni₂Al₃. In the present work, the surface and catalytic properties of Ni₃Al were investigated by X-ray diffraction, inductively coupled plasma (ICP) analysis, SEM observation, surface area measurement, and hydrogen production reactions. The results showed that Ni₃Al without alkali leaching showed a limited catalytic activity for the methanol decomposition (CH₃OH -> 2H₂+CO₂), and no activity for the steam reforming of methanol (CH₃OH +H₂O -> 3H₂+CO)
in the temperature range of 513–635 K. The activity of Ni3Al for the methanol decomposition was improved significantly by alkali leaching. The alkali leaching showed a great effect 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 are within the range of nickel used as a catalyst for hydrogen production reaction.

2:15 PM S4.3
Ultra-Strong and Ductile Hypereutectic Ti-based Bulk Alloys, Dmitri V. Louguine1, Larissa V. Louguina2, Hidemi Kato3 and Akihisa Imoue3. 1Institute 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 alloys are capable of further strengthening. 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-M (M 0 other transition metals) and ternary Ti-Fe-M alloys as well as Ti-Fe-Re (Re- rare-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 bcc (TiFe-phase or a solid solution on its base) and a dispersed eutectic consisting of the Pm3m intermetallic compound + BCC Im3m β-Ti supersaturated solid solution phase. The mechanical properties of the samples cut from various parts of the arc-melted ingots were tested by an Instron-type machine. The Ti65Fe35 alloy having hypereutectic structure consisting of the primary TiFe phase and submicron-size eutectic (TiFe + β-Ti) structure showed excellent mechanical properties: a Young's modulus of 140 GPa, a high compressive fracture 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 caused 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 fracture 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.

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, Hironori 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 Microscopy equipped with an 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 provided. New thermoelectric devices can be used for the conversion of heat into electricity or for solid-state refrigeration where the only moving parts are electronics and holes. The poor efficiency of thermoelectric devices has limited 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, κ, and the electrical conductivity, σ. However, the requirement for large values of Seebeck coefficient and electrical conductivity is often conflicting. 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-heuslers, alloys, thin film superlattices and superlattices with and without quantum confinement, nanowires, and bulk materials with nanocrystals.
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 electric conductivity. Clathrate compounds possess polyhedral cages encapsulating guest atoms. Although the low thermal conductivity in clathrate compounds is believed to result from the rattling motion of the guest atoms inside the cages, the relationships between cage 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$_4$Ge$_5$Ga$_{16}$, Ge and Ga atoms constitute the cage structure and they are randomly distributed in the cage structure. Upon substituting Ga atoms in Ba$_4$Ge$_5$Ga$_{16}$ by In atoms, the thermoelectric power factor increases while the lattice thermal conductivity decreases, leading to a much higher thermoelectric figure of merit (ZT$>1$).

Magnetic Properties of E2$_2$-Base Co$_3$AlC and the Correlation with the Octahedral Vacancies.

Yoshiasato Kimura$^1$, Fu-Gao Wei$^2$, Hideyuki Ohtsuka$^2$ and Yoshinori Matsuda$^1$.

The E2$_2$ 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 E2$_2$ and L1$_2$ 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 mostly due to minimizing the elastic strain energy. Half of carbon atoms are replaced by vacancies at every other body center site as if E2$_2$ and L1$_2$ were arranged alternatively in every other unit cell. Thus it should be denoted as E2$_2$ Co$_3$AlC$^a$ though it remains still controversial that whether the ordering is long range or short range. We also have reported that Co$_3$AlC phase exhibits whole ferromagnetism while Co$_3$Al$_{100}$K$_2$ is unusually high with comparing with its 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 E2$_2$-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 and 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 E2$_2$ Co$_3$AlC against E2$_2$ 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.

Atomic, Electronic, and Magnetic Structure of Iron-Based Sigma-Phases.

Mishima I; 2National Research Institute for Materials Science, Tsukuba, Japan.

Sigma-Phases are complex tetragonal structures that form in transition metal alloys having the average number of s+d electrons in the interval 6.5-7.4. These phases, which may form during solidification or heat treatment, usually cause embrittlement of the alloys. Well known example is the FeCr sigma-phase, whose 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 an ab initio total energy calculations combined with the calculated electronic structure and thermodynamic properties are discussed and compared with results of previous studies.
discussed on the basis of CTEM and HREM observations.

**S5.3 Structure and Physical Properties of Cu2MnAl Alloy Films with Significantly Different Atomic Orders.** BumJoong Kim1,*

Nina Lee1, Meidong Huang2, Kiwon Kim3, Yurij V. Kudryavtsev4 and YoungPak Lee5; 1Physics & Dept. of Physics, Huayang University, Seoul, South Korea, 2Department of Physics, Seoul University, Asan, South Korea, 3Institute of Metal Physics, Kiev, Ukraine.

**S5.4 Laser Annealing of Shape Memory Alloys Thin Films: Oxide Growth and Its Application.** Shinya Yoshida1, Jin-Hwa Shin2, Kei Yamauchi3,4, Yasuhiro Horii5,6,7, Xi Wang6,7,8, Joost Vlassak3 and Thomas Gessmann4; ICAT, Rensselaer Polytechnic Institute, Troy, New York; 2DTIS/LIST, Commissariat a l’Energie Atomique (CEA), Fontenay-aux-Roses, France; 3DEAS, Harvard University, Cambridge, Massachusetts; 4ECSE, Rensselaer Polytechnic Institute, Troy, New York.

**S5.5 Structural Properties and Magnetic Behavior in the Pseudo-Coary Alloys CoFeCoMnSi B2-type ferromagnets.** Hiroki Ishibashi and Mineo Kaguchi; Department of Materials Science, Osaka Prefecture University, Sakai, Osaka, Japan.

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

**S5.7 Crystal Structure and Thermoelectric Properties of Al-containing Re Silicides.** Eiji Terada1, Min-ook Oh1, Myung-Hoon Oh1, Dong-Moon Woe2 and Haruyuki Inui3; 1Materials Science and Engineering, Kyoritsu University, Tokyo, Japan; 2Materials Science and Engineering, KAIST, Daejon, South Korea; 3Materials Science and Engineering, KNU, Gumi, South Korea.

Binary rhenium disilicide 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 growth is much faster than that for the slow-cooled sample. 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 alloy films has been predicted to be half-metals, theoretically to be half-metals and their films might be used as spin-injectors in such devices. True HA have a formula of $X_2YZ$, where $X$ and $Y$ are transition metals and $Z$ is a s-p element, and a crystalline structure of $L_2$. 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 CuMnAl 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 films were measured in a temperature range from 5 to 300 K by using a vibrating sample magnetometer. The magnetic behavior is discussed through a correlation to the structure and the magnetic properties. 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Hence in this study, the structural and magnetic properties of these intermetallics.
anisotropic electronic structure of ReSi1.75. The dimensionless figure of merit (ZT) for binary ReSi1.75 is as high as 0.7 at 1073 K when compared with the ZT of 0.15 at 50 K. We have tried to improve the thermoelectric properties of ReSi1.75 by adding Al. Al substitution for Si in Re silicides improves the ZT value along [100]. The temperature dependence of electrical resistivity in ReSi1.75 indicated that the contribution of anomalous scattering is not apparent in this compound. In summary, we have shown that the thermoelectric properties of ReSi1.75 can be improved by the addition of Al.

Previous studies on ternary NiAlM alloys [1] have found a surprising enhancement effect that cannot be solely attributed to the compositional change of atomic size or elastic moduli. Instead, the magnetic properties of solute atoms have to be considered. In search for evidence of possible magnetic ordering, we have carried out a systematic study of NiAlM (Ni, Fe, Co) alloys using neutron diffraction. The diffraction data show that when Al atoms are partially replaced by transition metal atoms (Ni0.86M0.14Al0.04), there is a phase transition at very low temperature (Te=20K), while alloys with transitional metal atoms at Ni0.86M0.14Al0.04 have a stable B2 structure. Clearly, the addition of the transitional metal atoms (Ni+M) on Al sites destabilizes the B2 structure and the transition depends on the fraction of (Ni+M) on Al sites. However, the observation transition does not seem to be related to magnetism, since neither Ni-rich nor Co-doped alloys have any significant magnetic moment. The diffraction pattern of the new phase also does not match any of the known magnetite structures. Instead, we have found that the new peaks can be well indexed with a slightly distorted double-lattice superstructure, although their intensities have not been reproduced. These observations suggest that the new phase is formed by re-ordering of the local atoms within a double-lattice superstructure. This is a fascinating result as it implies that local atomic ordering can occur at such low temperatures. Further experiments are underway to characterize this new phase transition. This research was sponsored by 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.

S5.10
Low-temperature Phase Transition in NiAlM Alloys.
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Over the past decades, CALPHAD modeling has been increasingly used to describe the thermodynamic properties of a great number of material systems. Thanks 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 alloy system allows the focusing of the experimental work on only 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 lacks. Fortunately, thanks to recent progresses 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, but an accurate thermodynamic 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 temperatures. Due to their excellent thermal stability, low mass density, ordered B2 phases are often used in high-temperature applications. In this work, a systematic study of the vibrational and electronic properties of the ordered B2 phases in the Al-Ni-Pt-Ru system is presented. This complements the ongoing efforts in this group to calculate the OK energetics of this quaternary system. Density Functional Theory within the GGA (and LDA) approximation is used to calculate the vibrational entropies of the B2 phases via the super cell method. The electronic free energy is calculated by integrating the electronic DOS at different volumes. By combining the OK 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.


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 are compared with experimental results, and it can also be 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 modified sublattice A2/B2 model. The modified sublattice model describes the bcc phase with one Gibbs energy function and simplifies the CALPHAD model significantly.


Relaxation behavior of supersaturated vacancies in B2-type aluminides such as NiAl and CoAl was studied by the differential scanning calorimetry (DSC) method. The single DSC peak observed only at first heating runs, depending on alloy composition and quenching temperature. In stoichiometric NiAl and CoAl, water-quenched from 1773 K, single exothermic peak appeared near 950 K. On the other hand, water-quenched Al-poor Ni-40at%Al and Co-42at%Al alloys quenched from 1773 K. One of the two exothermic peaks in Co-42at%Al disappeared as quenching temperature decreased from 1773 to 1573 K. The exothermic peak was not observed in 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 aluminate ribbons where the vacancies with higher concentration could be observed, and it was also concluded that the relaxation processes of supersaturated vacancies are kinetically analyzed, and then systematically discussed comparing with properties on diffusion and point defects for the B2 aluminides.

S5.16 The Yield Anomaly and Ductility of Single-Slip-Oriented FeAl Single Crystals. Ian Baker1,2, Dongmei Wu3, Paul R. Munroe3 and Easo P. George4. 1Dartmouth College, Thayer School of Engineering, Hanover, New Hampshire; 2Materials and Engineering Physics Program, Ames Laboratory - Iowa State University, Ames, Iowa; 3Electron Microscope Unit, University of New South Wales, Sydney, New South Wales, Australia; 4Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Many features of the yield strength anomaly in B2-structured FeAl alloys have been successfully described or predicted by the vacancy-hardening model. The model does not predict any orientation dependence for the yield anomaly. Thus, here, we describe the results of testing this prediction by measuring the critical resolved shear stress, CRSS, of three differently-oriented, single-slip-oriented Fe-43Al crystals as a function of temperature. In addition, the effects of boron on the CRSS were examined by comparing these data with results from mechanical tests of a single-slip-oriented Fe-43Al crystallite. It was found that the CRSS of all the alloys decreased rapidly from 730-300 K; showed a plateau from 300-723 K; increased to a peak at 873 K, and then, once more, decreased with further increases in temperature. Boron increased the strength at all temperatures above 150 K, but had no effect below this temperature. While neither the strength below <300 K nor the yield anomaly depended on orientation (in agreement with the vacancy-hardening model), the CRSS in the yield strength plateau region around room temperature did. The effects of temperature on ductility and fracture are also presented.

S5.17 Microstructure and Mechanical Properties of Fe-Ni-Mn-Al Alloys. Markus W. Wittmann1, Ian Baker1, James Hanna1 and Paul Munroe2. 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 such regularity, the enthalpies of formation of the two phases, formation of the microstructure is attributed to spinodal decomposition. Mechanical tests on one of these alloys, Fe-20Ni-25Mn-25AI, revealed yield strengths of 1.3 - 1.5 MPa between 300 and 500K and a strength over 200 MPa at temperatures up to 1073K. Heat treatments of one hour at 1073K resulted in coarsening of the microstructure and formation of an f.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.

S5.18 Thermomechanical Treatment of a Fe-Al alloy. Joachim Konrad1,2, Andre Schneider1, Stefan Zaefferer2, Georg Frommeyer1 and Dierk Rasche1. 1Materials Technology, Max-Planck-Institut fur Eisenforschung GmbH, Dusseldorf, Germany; 2Microstructure Physics and Metal Forming, Max-Planck-Institut fur Eisenforschung GmbH, Dusseldorf, Germany.

FeAl-based alloys are regarded as promising for high temperature applications in corrosive atmospheres. Generally FeAl shows higher strength compared to dispersed FeAl alloys and good resistance in oxidizing and sulphiding 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 > 900°C) cold (T = 400°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 crystallographic texture and a controlled grain size. The latter aim is achieved by the movement of single fold trails. During subsequent warm rolling this is utilized to improve the ductility. A controlled final heat treatment is applied to produce a fully recrystallized microstructure and to stabilize the state of improved ductility by reduction of driving forces for recrystallization. The influence of hot and warm rolling and the resulting 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 temperatures have been observed. Warm rolling has been performed in the of B2 and D0-3 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 microtexture 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 Fe3Al Based Alloys. Satoru Kobayashi, 1,2 Stefan Ziefert, 1,3 Andrzej Schneider, 1,4 Dirk Rasche, 1 and George Frommeyer, 1,2 Materials Science and Technology, Max-Planck Institute for Iron Research, Dusseldorf, Germany; 3Materials Technology, Max-Planck-Institute for Iron Research, Dusseldorf, Germany.

Fe3Al 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 sulfidation. Serious problems for the application are the poor high-temperature strength, creep resistance and the limited ductility at low temperatures. Aim of 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 Fe-26Al-0.6Cr 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 cooled 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 Fe3Al based alloys with strengthening MC carbides. In Fe-26Al-4Cr-0.5Ti-0.2C alloy with a higher amount of TiC, the transus temperature between α and α+TiC at 1400°C and the precipitation temperature during cooling after homogenisation is very quick. Needle-like TiC particles with more than 1 μm in length formed during air cooling after homogenisation and coarsened during the hot rolling process. The hot deformation with such large TiC particles causes particle stimulated nucleation (PSN) and hence accelerates recrystallisation. While in Fe-26Al-4Cr-0.5Ti-0.07C 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 precipitates followed by a heat treatment rather than 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 Fe26Al2Cr-TiC 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.

S5.20 Effect of Excess Vacancy on Austenite Domain Growth in Fe3Al. Yasuharu Konno, Tsukasa Higawara, Yoritoshi Mineo and Nobuhiro Tsuji; Department of Adaptive Machine Systems, Graduate School of Engineering, Osaka University, Suita, Osaka, Japan.

Growth of D021-type austenite phase (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 and of the cases that Tq was 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 and the amount of disordering loops were formed only in the specimens quenched from the disordered state and annealed at relatively low temperatures below 700K.

S5.21 Microstructure, Mechanical Properties and Wear resistance of Fe3Al based alloys with various alloying elements. Hye-Sool Kim, Won-Yong Kim and Tae-Yeob Ra; Advanced Materials R&D Center, Korea Institute of Science and Technology Incubation, South Korea.

We report on microstructure, mechanical properties and wear result of Fe3Al based alloys with various alloying elements. The microstructures were examined using electron microprobe analysis (SEM-EDX) and with energy dispersive X-ray spectroscopy and transmission electron microscope. Two types of alloys were prepared using vacuum melting; One is Fe-28Al based alloys with and without alloying elements such as Mo and Zr. The other one is Fe-35Al based alloys produced with same manner. For both types of alloys, Mo addition had to exhibit an equiaxed microstructure, while dendritic structure was observed to show the effect of Zr addition. The microstructural features were more evinced with increasing content of alloying elements. Concerning the mechanical properties and wear resistance, Fe-35 based alloys were superior to Fe-28Al based alloys especially in the high temperature region. On the basis of the results obtained, details would be discussed.


The d-spacings and the line widths of Bragg peaks in micron size anisotropic polycrystals of Ni3Al and Cu3Al intermetallic alloys have been studied under non-hydrostatic conditions by energy-dispersive X-ray diffraction (EDX) in a diamond-anvil cell. 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 and dislocations. A volume incompressibility in the equation of state due to plastic deformation and the saturation of the stacking fault probability is followed by an elastic-compression of a full plastic deformation and the compression of this state is isotropic and independent of the pressure. From the systematic shift of different Bragg peaks the stacking fault probability as a function of the confining pressure is derived. The onset of incompressibility of the compression of 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 stresses.

S5.24 Development and Characterization of W-Based Nanocomposites Prepared by Mechanical Alloying and a Study of the Formation of Intermetallics in These Systems. Satoru Kobayashi, 1,2 Microstructure Physics and Metal Forming, School of Engineering, Osaka University, Suita, Osaka, Japan.
Nasimul Alam Syed1, Shyamal Kumar Pabi2 and B.S. Murty2;
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The present study deals with the synthesis of nanocomposites of W-10, 20, 30, 40 and Ni-3Al 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 Pulverisette 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 plots. Some of the nanocomposites has 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 cushioning 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.

S5.25
Parameters of Dislocation Structure and Work Hardening of Ni3Ge, Nina A. Koneva1, Yulia V. Solovyeva1, Vladimir A. Starenchenko2 and Eduard V. Kozlov1; 1Dep. of Physics, Tomsk State University of Architecture & Building, Tomsk, Russian Federation; 2Dep. of Mathematics, Tomsk State University of Architecture & Building, Tomsk, Russian Federation

Ni3Ge intermetallic possesses L12 structure and remains fully ordered up to the melting temperature. Among other intermetallics with the L12 superlattice, Ni3Al is the most studied material. Gliding dislocations in this intermetallic are Marsinkowski dislocations. In the present work, the following mechanical characteristics were studied in tension: yield stress, tensile strength, plastic strain and work hardening coefficient α. The yield strength of single crystals [001], [−131], [−4917], [−234] and [012] of five orientations of single crystals has been calculated from the x-ray peak broadening using Voigt peak profile analysis. The results indicate that.

S5.26
Crystal Structure, Phase Stability and Plastic Deformation Behavior of Ti-rich Ni3(Ti, Nb) Single Crystals with Various Long Period Stacking Faults, Jana Houserova1,2,3, Walter Wolf3,2, Raimund Podloucky3,2, and Hans-Eckhardt Schaefer3; 1Institute of Theoretical and Applied Physics, Stuttgart University, Stuttgart, Germany; 2Institute of Physics, Jagiellonian University, Krakow, Poland; 3Center of Computational Materials Science, University of Vienna, Vienna, Austria.

Chemical ordering in intermetallic compounds is predominantly controlled by atomic jumps to nearest neighbor vacancies. An important factor controlling the formation of these defects is the concentration of thermal vacancies. In the present contribution, the intermetallic Ni3(Ti0.990.01)Fe0.1 system (0 ≤ x ≤ 25) with a L12 structure is studied. The admixture of Fe destabilizes the ordered phase and the transition of ordering - disorder occurring at a temperature higher than the melting temperature and at a higher vacancy concentration than at which the transition of ordering occurs for Fe-free Ni3Ti.

S5.27
Formation and Migration of Thermal Vacancies in Ni3Al, Wolfgang Pfeiffer1, Doris Vogtshulben2, Jana Housecova3, Walter WoS2, Raimund Podloucky32, and Wolfgang Puschel1; 1Materialphysik, University of Vienna, Vienna, Austria; 2Center of Computational Materials Science, University of Vienna, Vienna, Austria; 3Physical Chemistry, University of Vienna, Vienna, Austria.

L1-ordered intermetallics, being the main constituent of many superalloys, bear technological relevance and promising high-temperature structural materials. One of the issues that led to study the stability and kinetics of their atom configuration for which the knowledge of defect formation energies and migration profiles is fundamental. Vacancy and interstitial defect formation energies were calculated for the chemical analogues of Ni3Al, Ni3Ga, Pt3Ga, Pt3In by a supercell ab initio approach. The ground state energy values were incorporated in a grand-canonical thermodynamic framework with next-nearest-neighbor interaction due to conservation of stoichiometry as a function of temperature. Elastic and bonding properties were also obtained from ab initio calculations which together with arguments based on atom sizes are used to explain results. Energy profiles for atom jumps were calculated by statically displacing the jumping atom and relaxing the surrounding neighbours. The influence of variable atomic neighbours on the migration barrier and the stability of the initial and final states were studied by progressively exchanging...
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 Börzs-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 co-operative movement of two atoms which seems to play a role in the generation of antisite pairs in a disordering step.

SS5.39
Modeling of Creep in Inertogeneous Metal/Intermetallic Composites
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High-temperature creep response is a critical technical requirement for advanced intermetallic systems aimed at replacing superalloys. These advanced systems are typically composites 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. 755, 321 (2003)). However, correlation of the composite creep to the constituent creep behavior needs to be improved. Here, realistic Nb/Nb-Silicide 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 the current state of the art of these types of models are discussed. An intermetallic-structure-reconstruction technique is then used to generate 3d 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.

SS5.50
Phase Equilibria Predictions in Nb-Silicide Based Composites
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Nb-silicide based in-situ composites are promising materials for future high-temperature structural applications. NbSi 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. A new phase equilibria in the Nb-Hf-Si ternary system determined by experiments. Microstructural and microchemical evidence provided a clear definition of the Nb-Hf-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 Nb-Hf-Si system. Thermodynamic description of the Nb-Hf-Si quaternary system was then obtained by extrapolating the thermodynamic descriptions of Nb-Ti-Si, Nb-Hf-Ti, Nb-Hf-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.

SS5.31
Effect of Temperature and Ternary Additives on the Crystallographic Orientation Relationships among Phases Related to the Eutectoid Decomposition of Nb3Si
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 Nb3Si 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 eutectic Nb and product phases of eutectoid decomposition of Nb3Si (eutectoid Nb and Nb5Si3 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).

SS5.32
Annealing Induced Structural Changes and Microcracking in Mo-Mo3Si
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An outstanding issue in the processing of Mo-Mo3Si 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 are estimated to be 160 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-Mo3Si, we have conducted a systematic study of Mo-Mo3Si composites 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 Mo3Si 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 composite microstructures 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 Mo3Si. This finding was corroborated by microscopy studies which demonstrated that the microcracks almost always started at the interface of α-Mo and Mo3Si grains and grew into the Mo3Si. 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-Mo3Si occurred at high temperature. Quite possibly, the diffusion of Si during high-temperature annealing is highly influenced by the estimated stress distribution at the interface where cracks were initiated when the elastic energy exceeded the tensile limit of the Mo3Si 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.

SS5.33
Abstract Withdrawn

SS5.34
High Temperature Oxidation Behavior of Al added Mo5Mo2SiB2 in - situ composites
Akihiro Yamauchi, Kyoosuke Yoshimi and Shuji Hanada; Institute for Materials Research, Tohoku Univ., Sendai, Japan.

Isothermal behavior of Al added Mo5Mo2SiB2 in - situ composites was investigated under an Ar-20%O2 atmosphere over the temperature range of 1073-1673 K. The Al added Mo5Mo2SiB2 in - situ composites (Mo-8.7mol%Si-17.4mol%B,10_xAlx ; x=0, 1, 3, and 5mol%) were prepared by arc-melting, and homogenized at 2073 K for 24 h in an Ar-flow atmosphere. The ternary Mo/Mo5Mo2SiB2 in - situ composite exhibited a rapid mass loss at the initial oxidation stage and then the passive oxidation after the substrates were sealed with boronate glass in the temperature range of 1173-1473 K, whereas it exhibited a rapid mass gain around 1073 K. On the other hand, the Al addition, especially of 1mol%, significantly improved the oxidation resistance of Mo5Mo2SiB2 in - situ composites at temperatures from 1073 to 1273 K. These 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.

SS5.35
Nucleation of (Mo) Precipitates on Dislocations during Annealing of a Mo-rich Mo3SiB2 Phase
Nobuaki Sekido, Ridwan Sakidja 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 Mo3Si phase by small boron additions, the intermetallic compound Mo3SiB2 (T2) has received considerable attention for an ultra-high temperature application. In the present study, of particular interest is the role of phases that are not listed.
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 mismatch of precipitates are formed in the T2 matrix upon prolonged annealing at 1500°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 the Fe2Nb(E) alloy with hexagonal symmetry, Fe, 5, 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 of the T2 phase within the AI-Au system were determined by powder XRD. TEM observations have revealed that some dislocations have developed during annealing in the T2 phase, but few dislocations and 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 two 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 identified as 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 (F49620-03-1-0083) is gratefully acknowledged.

S5.36

The ferromagnetic L10-ordered intermetallic phase MnAl appears near equiatomic compositions after appropriate heat treatment. This metastable phase, having high uniaxial magnetocrystalline anisotropy, is the basis for attractive magnetic properties in these materials. The technologically important properties of these alloys are strongly dependent on the microstructure and defect structure produced by transformation from the high-temperature disordered epsilon-phase (A3, hcp) to the ordered twa-phase (L10). The twin-phase product exhibits characteristics similar to a plethora of crystal defects resulting from the phase transformation, including a profusion of twin boundaries and twin-related variants. Recently the nature of the transformation mode has been elucidated and as part of this study the mechanism of twin formation and defect generation have been investigated in detail, using CTEM, HREM and in-situ hot stage TEM. The results of this work will be discussed.

Support from the National Science Foundation (DMR) is gratefully acknowledged.

S5.37
Phase Equilibria and Lattice Parameters of Fe3Nb Laves Phase in Fe-Ni-Nb Ternary System at Elevated Temperatures. Nobuyuki Gomi, Sumio Morita, Takashi Matsuo and Masaaki Takeda, Dept. of Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo, Japan.

Fe2M TCP compounds are potential strengthening in austenitic heat-resistant steels to be used above 973 K for steam turbine components beyond USC power plant. Among the Laves phases, Fe2Nb(c) with hexagonal C14 structure is the most attractive because of high congruent melting temperature (1941K) and relatively large composition homogeneity region. In Fe-Ni system, however, the austenite (γ-Fe) region in equilibrium with c phase is limited above 1228K and not stable at lower temperatures, i.e. transformed to ferrite (α-Fe). In this study, phase equilibria in Fe-Ni-Nb ternary system at elevated temperatures have been examined first, in order to identify the γ+c two-phase region. Since we found the extended c single phase region in the ternary system, the lattice parameters of the phases in equilibrium with each other have also been studied. In the binary system, the Laves phase region exists in the range of 27.5 to 35.5 at.% Nb, and it extends toward the equi-niobium concentration direction up to 44 at.% Ni in the ternary system at 1473 K, indicating that more than half of the Fe atoms in Fe3Nb can be replaced with Ni. As the result, the γ+c two-phase region exists extensively, and the solubility of Nb in γ phase increases from 1.5 to 6.0 at.% with increase in Ni content. The lattice parameters of c and c in the C14 Laves phase decrease with increasing Ni content. Supposing that all Ni atoms occupy Fe sublattice site, the change in a axis in good agreement with calculation based on Vegard's law, whereas that of c axis is much larger than the calculated value. These results clearly suggest that atom size mismatch is responsible for the transformation, and the binding energy is dominant factor for the c-axis change. To extend these findings to development of new class of austenitic steels strengthened by Laves phase, an attempt has been made to control the c/a ratio by alloying at 1500°C. The Fe2Nb(20)Cr20N13 alloy can make the c/a ratio close to the cubic symmetry value (1.633), and very fine Laves phase particles are homogeneously nucleate within the γ matrix in Fe-Ni-Nb-Cr quaternary alloys aged at 1073 K. The morphology and stability of the Laves phase will be discussed in conjunction with the lattice misfit strains.

S5.38
Sputtered coatings based on the Al3Au phase. Christian Mitterer, Helmuth Lenhart, Paul H. Mayrhofer and Martin Kather, 1Department of Physical Metallurgy and Materials Testing, University of Leoben, Leoben, Austria, 2CERATIZIT Austria GmbH, Reutte, Austria.

Transition metal nitride-based, wear-resistant hard coatings on cutting tools and other substrates often lack self-lubricating properties at elevated temperatures and distinct colorations allowing product differentiation. In the present work, the possibility of achieving these objectives using coatings based on the purple-red Al3Au phase within the Al-Au system was investigated. Coatings within this system have been deposited onto cemented carbide and Inconel substrates using unbalanced magnetron sputtering. The coatings were characterized with respect to their topography and morphology, chemical and phase composition, hardness, optical, electrical, and tribological properties. Al3Au-containing Laves phase particles have been deposited with dense, fine-grained structures yielding a hardness of 4 GPa and pink coloration. Vacuum annealing at temperatures of 500°C results in a change to a pronounced purple coloration. The coatings are stable up to about 1000°C, where the organic radiation occurs. Low friction coefficients when testing against alumina counterparts were achieved in the temperature range between 500 and 700°C. The concept of applying Al3Au-containing coatings as a self-lubricating layer on top of a hard coated cemented carbide tool warrants further investigations.

S5.39

Bulk materials and coatings made of Ti3Al-X nanostructured intermetallics have been produced by mechanical alloying and sintering. X represents 8 at.% of Fe, Cr or Mn. Spark plasma sintering (SPS) has been used at temperatures ranging from 973 to 1173 K in order to preserve a grain size in the nanoscale. Characterization of the nanostructure has been performed including grain size distribution, strength, toughness, transmission and ductility as a function of temperature in the case of bulk materials. High strength and brittleness is found at room temperature but as temperature is increased, these materials become ductile. Remaining porosity apparently improves ductility at 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 0.9 mm. Steel, Cu and Al are used as substrate. Characterization of the coating nanostructure 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 Takahashi, Yukiyuki Sakaino and Shunzi Song.

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 99.99% purity were arc-melted into small ingots weighing about 10grams under an argon atmosphere. Small pieces were hot deformed at 1300K by about 50% reduction of height in air and subsequently vacuum annealed at 1400K for 100ks. The resulting microstructures contained equiaxed grains of alpha single phase whose average diameter ranged from 125 to 192 micrometers.

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except alloys containing 40 and 42 at.% aluminum. The microstructures in 40Al and 42Al materials contained small amounts of gamma phase. The minimum creep rates were obtained in creep tests performed in vacuum 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 rate region followed by a gradual creep acceleration were observed in the materials containing aluminum up to 25at.%.

In contrast to this, the alpha 2 single phase material containing more aluminum than 25at.% showed a primary transient where creep deceleration continued up to about 0.1 true strain and its following quasi-steady creep region. Two phase materials containing the gamma phase showed small primary transient probably due to the constraint from the harder gamma phase and the 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 got 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 Aluminides and Titanium Alloy, Bella A. Greenberg1 and Valery E. Rybin2; 1 Institute of Metal Physics, Ural Div. RAS, Ekaterinburg, Sverdlovsk region, Russian Federation; 2 CRISM 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 the development of bimetallic joints. One more 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 little 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 joint 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 aluminide. The mutual adjustment of the BCC lattices, which took place during diffusion welding, was beneficial to a high quality of the bimetallic joint. One more important factor was the absence of a continuous intermetallic layer, which could cause embrittlement, near the joint. Two factors were revealed earlier in the study of a bimetallic joint of a titanium intermetallic with a titanium matrix, provided bimetallic joints having absolutely different structures. After diffusion welding, structures near the contact surface contained BCC / face-centered cubic and plate-like regions of the inclusions of a2-phases. After explosion welding, structures near the contact surface were free of BCC phases, but contained HCP phases, namely, the titanium a-phase, the aluminide a2-phase and, in addition, the aluminide O-phase.

S5.43 Twin-Transformed Lattice Defects in γ-TiAl, Patrick Veyssiere1, Haruyuki Imai2 and Yu-Lung Chiu1; 1 LEM, CNRS-ONERA, Chatillon, France; 2 Fusion Technology - PSI, Ecole Polytechnique Federale de Lausanne, Villigen PSI, Switzerland; 3 Materials Science and Technology, Kyoto University, Kyoto, Japan.

Twinning a pre-existing deformation microstructure transform the lattice defects intersected into defects whose nature is either unaltered or changed in terms of Burgers vector, crystallographic direction and/or habit plane. Several examples of such transformations are shown and discussed. The case of a faulted dipole is examined in some detail for it may yield a stacking fault configuration that cannot form spontaneously in the alloy. Transformations of faulted dipoles involve in several ways that inform on the stability of this feature. Conditions of twin propagation and twin growth are examined. A model is provided for spontaneous growth beyond a critical twin size; the model makes use of forest dislocations and reactions between partials belonging to the twin front.

S5.44 Micro-Fracture Toughness Testing of TiAl Based Alloys with a γ-TiAl Lamellar Structure, Kasuki Takashima1, Tetsuo Kuroda1, Timothy Halford2, Yakichi Higo1 and Masao Takeyama1; 1 P&I Laboratory, Tokyo Institute of Technology, Yokohama, Japan; 2 Dep. of Metallurgy and Ceramics Science, Tokyo Institute of Technology, Tokyo, Japan.

TiAl-based alloys with a fully lamellar structure exhibit superior fracture properties compared to those with other microstructures. This is mainly due to the activation of extrinsic toughening mechanisms including crack deflection and shear ligament bridging. These extrinsic toughening mechanisms are controlled by the mechanical properties of lamellae, in particular the lamellar interface fracture strength. It is, therefore, extremely important to evaluate the fracture properties of lamellar colonies to improve the fracture toughness of such TiAl based alloys. 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 $\approx 10 \times 15 \times 50 \mu m$ were prepared from one lamellar colony by focused ion beam machining. Notches with a width of 0.5 µm and a depth of 5 µm 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 the test temperature. The fracture toughness values were obtained in the range 0.2 - 6 MPa m$^{1/2}$. 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 the absence of significant extrinsic toughening mechanisms in this 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.

S5.45 High-Temperature Environmental Embrittlement of Isothermally Forged TiAl-Based Intermetallic Alloys with Various Kinds of Microstructures, Takayuki Takanasi, Yasuhiro Hanada, Satoshi Shubuya, Hironori Kanno and Hidehito Ikuse; Department of Metallurgy and Materials Science, Osaka Prefecture University, Sakai, Osaka, Japan.

Isothermally forged TiAl-based intermetallic alloys with various microstructures (gamma grain, duplex, dual phase and fully lamellar microstructures) were prepared. These TiAl-based intermetallic alloys were tensile tested in vacuum and various atmospheres (such as air containing water vapor, O$_2$ gas, N$_2$ gas and H$_2$ gas) as functions of temperature and strain rate. Some intermetallic alloys and microstructures showed reduced tensile strength (or elongation) in air and H$_2$ gas at low temperature, and also in air, H$_2$ gas and O$_2$ gas at high temperature. Also, some microstructures showed not only the testing temperature and microstructure but also the testing atmosphere. The observed microstructural effect on the high-temperature environmental embrittlement was discussed, in association with hydrogen and oxygen properties and kinetics in the constituent phases and at some interfaces.


Polysynthetically-twinned TiAl crystals were deformed by compression with loading axis parallel and perpendicular to the lamellar interfaces. The deformation structures on the free surfaces were scanned using a dimension AFM with scan directions parallel
Experimental Studies and Thermodynamic Modelling on the Phase Transformations in γ-TiAl Based Alloys, Harald F. Chladil1, Harald Leitner2, Arno Bartels4, Rainer Gerling3, Wilfried Markert4, Holm Clemens2, Department of Physics, Metallurgy and Materials Testing, University of Leoben, Leoben, Austria; 2Materials Science and Technology, TU Hamburg-Harburg, Hamburg, Germany; 3Institute for Materials Research, GKSS-Research Centre, Geesthacht, Germany; 4Research & Development, Boehler-Schmidetechnik GmbH & Co KG, Kapfenberg, Austria.

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, for thermomechanical processing the occurrence of phase transformations, which sensitively depend on alloy composition, is of particular importance. The knowledge of the influence of alloying elements on the amount and the thermodynamic stability of the phases present is the basis for heat treatments used for optimization of mechanical properties. For example, β/2β 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 the composition Ti42(45-46)Al-(5-10)Nb+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 experimental TiAl database.

Massive Transformation in High Niobium Containing TiAl-Alloys, Arno Bartels1, Shavonir Bystrozanowski1, Harald Chladil2, Holm Clemens2, Rainer Gerling2, Harald Leitner2 and Frank-Peter Schimansky3, 1Materials Science and Technology, TUHH, Hamburg, Germany; 2Physical Metallurgy and Materials Testing, Montanuniversität, Leoben, Austria; 3Materials Research, GKSS Research Centre, Geesthacht, Germany.

Two high Nb containing γ-TiAl-based alloys with chemical composition of Ti-45Al-7NbN (at%) and Ti-46Al-9NbN (at%) were massively transformed to single phase γ′-TiAl by rapid cooling from the α/2 phase field, i.e. from temperatures above 1300°C. Using a quenching dilatometer the influence of cooling rate and starting temperature of a 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 backscatter diffraction. The γ′-phase was found to be a cubic phase 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′-phase. X-ray measurements led to a poor 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/α′-phase were analyzed by hardness tests and X-ray diffraction measurements. The onset of these processes is reflected in a drop of hardness and in X-ray diffraction patterns of massively transformed microstructure do not show separated γ-200 and γ′-02 reflections as expected from a tetragonal γ-TiAl lattice. At 800°C separation of the γ-200 and γ′-02 reflections starts with increasing annealing time. This phenomenon, the increase of the c/a-ratio, is attributed to relaxation of interatomic stresses and formation of α′-2Ti3Al. The appearance of α′-2 phase is associated with an increase of the Al-content in the γ′-TiAl phase.
Mechanical Behavior of a Pt-Cr Jewelry Alloy Hardened by Nano-Sized Ordered Particles. Kamili Jackson, Mielani Nzula and Anthony Fattahi. University of Michigan, Ann Arbor, Michigan; AMPAC, U.C. San Diego, La Jolla and Osaka University, Japan. A recent study showed that the combination of mechanical properties of a Pt-Cr alloy can be improved by adding a small amount of nano-sized ordered particles. The ordered phase is formed during the hardening process, and it can significantly increase the strength and hardness of the alloy. In addition, the ordered phase can also improve the ductility and toughness of the alloy, making it suitable for use in jewelry applications. The microstructure of the alloy was analyzed using transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDS). The results showed that the ordered phase is homogeneously distributed throughout the matrix and that it is present in the form of thin lamellae. The presence of the ordered phase was confirmed by selected area electron diffraction (SAED) and high-resolution transmission electron microscopy (HRTEM) analysis. The ordered phase was identified as the L12 structure, which is a well-known ordered intermetallic phase in the Pt-Cr system. This study provides new insights into the role of nano-sized ordered particles in improving the mechanical properties of Pt-Cr alloys, which can be utilized in the development of new high-performance jewelry materials.
T_{0} (allotropic phase boundary between γ and D0_{22} in the two-phase region), while allotropic transformation to D0_{22} accompanied by micro- or nanocrystalline microstructures. When the angle is below 20 degree, both the gamma phase and the alpha 2 phase deform by shear in slip planes inclined with the lamellar interfaces, but the shear vectors lie in the interface. When the angle is between 20 degree and 80 degree, PST samples deform by a sheath band-type deformation mode occurring according to an instability mode exhibiting a short wave length. Both structural defects of the lamellae as well as the macrostructural part of the lamellar colonies can be observed in the microstructure consisting of alternating layers of γ-TiAl and α_{2}-Ti3Al. After large compressive deformation at temperatures above 1000°C the samples were analyzed by means of scanning electron microscopy. The lamellar colonies show, depending on their orientation, more or less marked buckling-type deformation modes according to an instability mechanism with big wave length. Then two deformation modes interact in rather complicated 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 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 γ-TiAl lamellae. At the mesoscopic level six grains are considered to describe the material behavior over a length scale of 100μm. The correlation of slip between adjacent lamellae of γ-TiAl by numeric modeling. Thomas Schaden, F. D. Fischer, H. Clemens and F. Appel; 1Institute for Mechanics, University of Leoben, Leoben, Austria; 2Department of Physical and Materials Testing, University of Leoben, Leoben, Austria; 3GKSS Geesthacht, Geesthacht, Germany.

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 γ-TiAl and α_{2}-Ti3Al. After large compressive deformation at temperatures above 1000°C the samples were analyzed by means of scanning electron microscopy. The lamellar colonies show, depending on their orientation, more or less marked buckling-type deformation modes according to an instability mechanism with big wave length. Then two deformation modes interact in rather complicated 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 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 γ-TiAl lamellae. At the mesoscopic level six grains are considered to describe the material behavior over a length scale of 100μm. The correlation of slip between adjacent lamellae of γ-TiAl by numeric modeling. Thomas Schaden, F. D. Fischer, H. Clemens and F. Appel; 1Institute for Mechanics, University of Leoben, Leoben, Austria; 2Department of Physical and Materials Testing, University of Leoben, Leoben, Austria; 3GKSS Geesthacht, Geesthacht, Germany.

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existing chemical driving force with respect to an \( \alpha_2-Ti_3Al \) to \( \gamma-TiAl \) transformation. This phase transformation leads to a spheroidization of the lamellar core which can be considered as a further softening mechanism.

11:00 AM **S6.8**


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 scavenge Ti, resulting in coarsening of the TiAl lamellar plates during the early stages of aging. In the case of the alloy without B, however, the lamellar microstructure exhibits high 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 Microized Samples of \( \gamma \) – TiAl Based Material. Timothy P. Halford, Kazuki Takashima and Yakichi Higo; Precision and Intelligence Laboratory, Tokyo Institute of Technology, Yokohama, Japan.

High strength \( \gamma \) – 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 a new test method was required. This method developed here is intended for the fatigue testing of samples measuring \( \approx 10 \text{ mm} \) (B) \( \times 20 \text{ mm} \) (W) \( \times 40 \text{ mm} \) (L). This is completed using a machine recently developed at Tokyo Institute of Technology to load samples of lamellar \( \gamma \) – 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 microcrystalline thin sheet specimens. In this work sample cantelevers of Alloy 7 are Focused Ion Beam (FIB) machined from finely polished foil \( \approx 20 \text{ mm} \) thick and their stress – life (S–N) fatigue behaviour evaluated. The dependence of fatigue life upon lamellar orientation for given peak 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 microcrystallized 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-Günter 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, involving deformation properties, recrystallization kinetics and particularly the phase constitution, working temperature, finishing process conditions and alloy compositions were investigated. By comparing the results it is indicated that the determined textures can be understood by a combination of the twin modes of the gamma phase at hot-working temperature and the subsequent phase transformations. However, the current understanding of texture evolution is far from being complete as no model can be presented which quantitatively accounts for the contribution of the different processes mentioned.

11:45 AM **S6.11**

On the Effect of Interstitial Elements on Microstructure and Properties of TiAl and Quaternary TiAl Based Alloys. Jean-Pierre Chevalier1, 2, Melanie Lamirand1 and Jean-Louis Bonnetien1; 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 has 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 correlates 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 microstructure 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 Ti4Al85Nb18 with fully lamellar microstructure (with not more than 12% GB lamellar grains) and near lamellar microstructure (with about 12% GB lamellar grains). These underwent testing at room temperature at 200 and 300MPa respectively with a stress ratio R= 0.1 and a frequency of 8Hz. The TEM observations were carried out 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 have been determined and correlated with the orientations of grain constituents with respect to the stress axis. The results are explored with a view to understanding the initial cracking of the lamellar microstructures and the possible effect of grain boundary lamellar grains on the cracking behaviour.

2:00 PM S7.3 Impact Properties of Hot-Worked Gamma Alloys with BCC 2:1 Ti Phase, Kentaro Shindo1, Toshimitsu Tetsumi2, Toshiro Kobayashi3, Shigeo Morita1, Satoru Kobayashi4 and Masao Takeyama4; 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 Production Systems Engineering, Toyoohashi University of Technology, Toyoohashi, Aichi, 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 42~44Al-5~10X:X=V,Mn(at%) consisting of lamellar, g and b grain of which the workability was improved by introducing b-Ti has been investigated using an instrumented Charpy impact test, tensile 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 has been studied by paying attention to the grain size, interlamellar spacing and lamellar area fraction. The results were also compared with those from the alloys with fully-lamellar structure (FL) structural, nearly-gamma structure (NG) and equiaxed gamma structure (EG). The absorbed energy for crack initiation 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 initiation is almost comparable to that of the alloy with EG 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 structures. 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. Moreover, the value increases with increase in the lamellar 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 for hot-forged materials.

4.0*10⁻³ to 1.7*10⁴(s⁻¹), tensile strength of hot-extruded TiAl alloy is obtained and compared with that of Nickel based superalloy, Inconel713C. In the range of strain rate from 4.0*10⁻³ to 1.7*10⁴(s⁻¹), tensile strength of hot-extruded TiAl alloy increases with increase in the strain rate, but that of Inconel713C decreases. Then, using impact resistance of TiAl alloy at the low strain rate impact test like Charpy impact test, it is possible to assessed impact resistance at the high strain rate like foreign object attack in the turbofan on the safty. In foreign object attack test with taper plate specimen modified turbine blade and brass ball as foreign object. The behaviour of dislocations generated at the interface between specimen attack point and brass ball with thickness of specimen at attack point is investigated. A limit of impact energy, at which there isn't a crack on back of attack point, is obtained with each thickness of hot-forged TiAl alloy.42Al-5Mn. Therefore, the method of improvement of toughness was examined in the impact resistance of TiAl alloy is shown in this study.

2:15 PM S7.4 Effect of Long-Period Superstructures on Plastic Properties in Al-Rich TiAl Single Crystals, Nakano Takahashi1, Koutaro Hayashi1, Yuichiro Umakoshi2, Yu-Lung Chiu3 and Patrick Veyssiere2; 1Department of Materials Science and Engineering, Graduate School of Engineering, Osaka University, Suita, Osaka, Japan; 2Laboratoire d’Etude des Microstructures, CNRS-ONERA, Chatillon cedex, France.

In Al-rich TiAl crystals, some long-period superstructures appear depending on the annealing temperature and Al composition. Among them, the TiAl₂ and h- Al₃Ti superstructures contain pure Al(002) layers as in the L1₀ matrix structure, alternating with Ti(002) layers exhibiting an ordered arrangement of excess Al atoms. In single crystals with composition Ti₅₄.₇Al₇.₃Nb1.₃ (T₀=425K, Ti-62.5at.%Zr) annealed at 1473K, the Al₃Ti long-period superstructure embedded in the L1₀ matrix develops with increasing Al concentration to finally transform into h-Al₃Ti at Ti-65at.%Zr. The long-period superstructures affect the deformation behavior and deformation microstructure. The CRSS for the 1/2<110> ordinary slip, for example, increases abruptly with development of the Al₃Ti ordering. Dislocations with 1/2<110> Burgers vector group into fourfold configuration and APBs in the superstructure. The CRSS for slip in the <110> direction further increases with the formation of the h-Al₃Ti particles in the L1₀ matrix phase at Ti-65at.%Zr. By contrast, the Al₃Ti single-phase TiAl single crystal that results from further annealing of Ti-65 at.%Zr at 1023K for 48h, exhibits a CRSS significantly lower than that of the two-phase alloy with same alloy content. <110> superlattice dislocations are primarily activated under both [210] and [118] load axes, independent of the Al concentration, but the dislocation microstructure strongly depends on load orientation and Al composition. The frequency of the decomposition and the dissociation reactions of <110> superlattice dislocations is closely related to the degree of development of the Al₃Ti phase and the type of leading partials.

2:30 PM S7.5 TEM analysis of long-period superstructures in TiAl single crystal with composition-gradient. Satoshi Hata1, Kohjiro Shirishita2, Masaru Fukuda3, Yoshitsugu Tomokiyo4, Noriyuki Kiyono5, Takayoshi Nakano6 and Yukichi Umakoshi7; 1Department of Science for Electronic Materials and Systems, Kyushu University, Fukuoka, Japan; 2Art, Science and Technology Center for Cooperative Research, Kyushu University, Fukuoka, Japan; 3Department of Materials Science and Engineering and Handari Frontier Research Center, Osaka University, Osaka, Japan.

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 in a wide range of Al concentration, the present authors attempted to make a TiAl single crystal with a composition-gradient. After dipping a Ti-54.7 at% Al single crystal into molten Al at 1597 K for 5 min, a reacted area with a composition-gradient from 54.7 to 73 at% Al was formed 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-Al₃Ti, one-dimensional antiphase domain structure (1-APS) and Al₃Ti (D0₂₃). The SRO structure transforms into Al₃Ti superstructure after subsequent annealing at 973 K. All the superstructures except Al₃Ti have clear orientation relationships with the L1₀ matrix. The fact is explained in terms of ground state configuration of growing rate superstructures in a binary alloy system that are defined in terms of pairwisely compatible atomic interactions. It was also revealed that microstructures of the h-Al₃Ti and 1-APS domains depend on the composition-gradient as well as the Al concentration. Such a dependence of the microstructures suggests that anisotropic diffusion processes and a subsequent formation of strain fields in the L1₀ matrix influence the domain growth of the long-period superstructures.

2:45 PM S7.6 Atomistic Structure of the TiAl/ Ti₃Al Interface Observed During Massive Transformation, William Reynolds and Diana Farres; Materials Science and Engineering, Virginia Tech, Blacksburg, Virginia.

Empirical interatomic potentials were used to simulate the atomic structure of an ordered array Ti₃Al/TiAl interface during massive transformation. The exact orientation relationship observed in experiments was generated and the minimum energy configuration was obtained using molecular statics simulations. The observed configuration presents edge to edge matching of atomic sites along one direction and it is shown that this matching is indeed maintained in the relaxed computer simulated structure. The effects of various deviations of this one dimensional matching orientation of the interface energy and structure are discussed.
heat-resisting material. Numerous engineering alloys have been developed for structural applications in automotive and aerospace components. New alloys were 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 clarified and the study on the influence of several types of elements on oxidation behavior of TiAl was investigated by micro-sectional TEM using ion implantation. The ion implantation was carried out with doses of 10^14 to 10^16 ions/cm^2 at acceleration voltages of 60 to 100 kV. The oxidation resistivity of Mo, Ti and W improves the oxidation resistance significantly. Formation of a protective Al_2O_3 layer through beta-phase formed in modified orthorhombic (O) or ordered phases, microstructure is of critical importance to steady state growth rates or propagation of jog pairs. The deformation microstructure is formed in a two-phase beta-phi/beta-phase, where the higher Al content in Ti-22Al-28Nb leads to greater tensile creep strength and lower creep rates, while the RT strength of the Ti-22Al-28Nb, which also forms a ductile alpha2 phase, is responsible for the results. The TEM study of the microstructure and the long-term creep testing of Ti-22Al-28Nb demonstrated that the microstructure and creep behavior are related to the microstructure. Some of the microstructural features were evaluated for grain boundary character distribution, grain size, phase volume fraction, and morphology. The Al content, which is active during creep and stress relaxation, was confirmed by TEM observation, and reduced growth rate of Ti-22Al-28Nb is due to beta phase. The microstructural features evaluated for grain boundary character distribution, grain size, phase volume fraction, and morphology. The Al content, which is active during creep and stress relaxation, was confirmed by TEM observation, and reduced growth rate of Ti-22Al-28Nb is due to beta phase. The microstructural features evaluated for grain boundary character distribution, grain size, phase volume fraction, and morphology. The Al content, which is active during creep and stress relaxation, was confirmed by TEM observation, and reduced growth rate of Ti-22Al-28Nb is due to beta phase.
Before ageing and creep testing the microstructure of the samples was nearly lamellar. The nearly lamellar microstructure consisted of α2 (Ti3Al) and γ (TiAl) lamellae, coarse B2 (ordered Ti-based solid solution) particles and small volume fraction of the γ-phase formed along the grain boundaries. The duplex microstructure consisted of lamellar, feathery and γ-rich regions. Microstructural analysis revealed that the lamellar regions are composed of α2 and γ lamellae, coarse B2 particles and fine needle-like B2 precipitates. The feathery regions contain γ-matrix with α2, B2 and Ti5Si3 particles. Coarse Ti5Si3 particles are identified within the γ-rich regions. During ageing of the samples with duplex initial microstructure, the α2-phase in the lamellar and feathery regions transforms to the γ-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 diffusion-controlled transformations and coarsening of the coexisting phases. A possible effect of such microstructural changes on softening kinetics is analyzed. The softening produced at a designed operating temperature of 978 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 crept specimens. Significant differences of one order of magnitude in minimum creep rate and time to fracture of the specimens with different initial microstructure (nearly lamellar or duplex) are explained 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 α2/γ lamellar interfaces and within the γ-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.

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In this paper the creep behavior and the microstructural stability of high Nb containing Ti-46Al-9Nb (in at%) sheet material were investigated in the temperature range of 700°C to 800°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 α phase field). Short-term creep experiments at 700°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°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 different microstructures under various creep conditions has been analyzed by means of back scatter electron microscopy and X-ray diffraction. Our investigations revealed considerable stress and temperature induced microstructural changes which are reflected in the dissolution of the α2 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.

4:45 PM S7.13 Internal Friction of a High Nb Gamma TiAl Alloy With Different Microstructures. Manfred Weller1, H. Clemens1, G. Dehm1, G. Hansczech1, S. Bystrzniakowski1, A. Bartsels1, R. Geiling1 and E. Arzt1, 1Max-Planck-Institut fur 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 300 K to 1290 K 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=1000 K which is only present in fully lamellar and duplex samples; (ii) a high-temperature damping background above T=1100 K. The activation enthalpies determined from the frequency shift are H=2.9 eV for the loss peak and H=4.2-4.3 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 segments which, as indicated by transmission electron microscopy observations, are pinned at lamella interfaces. From measurements of the eigenfrequencies in frequency range II the variation of Youngs modulus was determined in the temperature range of 300 K to 1200 K. The modulus decreases only by about 15% proving that gamma TiAl-based alloys retain their elastic stiffness up to high temperatures. The Youngs modulus of Ti-46Al-9Nb is significantly higher when compared to Ti-based alloys. An influence of microstructure on elastic modulus was not found.