MM 50: Topical Session Modern Atom Probe Tomography IV - Thin Films and Structural Materials

Time: Thursday 11:45–13:00 Location: H 0107

MM 50.1 Thu 11:45 H 0107

Interface sharpening in miscible Ni/Cu multilayers studied by atom probe tomography — •Zoltán Balogh, Mohammed Reda Chellali, Gerd-Hendrik Greiwe, and Guido Schmitz — Institut für Materialphysik, Westfälische Wilhelms Universität Münster; Münster (Germany)

The chemical analysis of buried interfaces is a delicate task that usually requires well conditioned specimens. The clear and gentle nature of field evaporation and the all 3D subnanometer resolution makes atom probe tomography a method with perspective in this field.

We investigated the effects of diffusional annealing in the miscible Ni/Cu system [1]. We prepared two kinds of samples. The first was produced with abrupt interfaces, while the second type revealed artificially smeared interfaces of about 3 nm in depth. After 15 min annealing at 773 K both types reveal an interface width in between the two different as-prepared values. Thus in the case of the smeared samples, the Ni/Cu interfaces sharpened even though the system is completely miscible at the annealing temperature. Another important observation is that the resulting interfaces were independent of the initial values.

Consequently even though thermodynamic equilibrium predicts infinite continous mixing at the interface, the actual kinetic process, determined by material transport properties, can require nevertheless finite sharpness in intermediate stages.

[1] Z. Balogh, M.R. Chellali, G.-H. Greiwe and Guido Schmitz, Appl. Phys. Lett. 99 (2011) 181902

MM 50.2 Thu 12:00 H 0107

Phase Separation in Immiscible Copper-Tantalum Alloy Films — •Claudia M. Mueller[1], Stephan S.A. Gerstl[2], Alla S. Sologubenko[1], and Ralph Spolenak[1] — [1]Laboratory for Nanometallurgy, Dept. of Materials, ETH Zurich, Switzerland — [2]EMEZ Center for Electron Microscopy, ETH Zurich, Switzerland

Phase separation in binary alloys of immiscible elements is investigated on the copper-tantalum system. The alloys are prepared by co-sputtering followed by annealing to induce phase separation. Local Electrode Atom Probe (LEAP) Tomography in combination with Transmission Electron Microscopy (TEM) and X-ray Diffraction (XRD) is used to study the evolution of phase separation. Results show that in the amorphous copper-tantalum alloys phase separation is coupled to the crystallization of the individual phases. Phase separation starts with the formation of Cu-rich clusters in the still amorphous matrix at temperatures lower than 400°C; the clusters contain up to 10 at% Ta and have a FCC structure with (111) texture. At 600°C the amorphous matrix crystallizes into a β-Ta structure with up to 7 at% Cu dissolved within. It is observed that GBs between Ta nanograins in this sample are enriched with Cu. The amount of Ta that is dissolved in the Cu phase decreases during annealing. The atomic distributions of Cu and Ta in the respective Ta-rich and Cu-rich phases will be discussed in terms of cluster formation and phase growth.

MM 50.3 Thu 12:15 H 0107

Spinodal decomposition in TiAlN/CrN multilayer hardcoatings studied by atom probe tomography — •Ivan Povstugar[1], Pyuck-Pa Choi[1], Jae-Pyoung Ahn[2], and Dierk Raabe[1] — [1]Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany — [2]Korea Institute of Science and Technology, Seoul, Korea

Nanoscale nitride multilayers are good candidates for protective coatings for cutting tools and machine parts owing to excellent mechanical properties and corrosion resistance. However, they possess only a limited thermal stability at operational temperatures. To understand its origin, chemical information at an atomic scale is essentially required. We exploit atom probe tomography to study the thermal evolution of Ti(0.75)Al(0.25)N/CrN multilayers prepared by the sputter deposition.

TiAlN/CrN coating shows well-resolved layered structure with single layer thickness of 4.5 nm. The multilayers are stable up to 600°C when short-range diffusion at layer interfaces begins. At 700°C TiAlN layers undergo interface-directed spinodal decomposition. As a result, each TiAlN layer evolves into a sandwich-like structure consisting of a Ti-rich sublayer confined by two Al-rich ones. With the increase of time or temperature of thermal treatment, the interface-directed mechanism passes into common isotropic spinodal decomposition accompanied by intermixing between TiAlN and CrN layers in a close-to-surface region of the coating. Conversely, Al-rich layers remain clearly distinguishable in the deep region of the coating. The difference is ascribed to the non-uniform release of residual internal stresses during heat treatment.

MM 50.4 Thu 12:30 H 0107

Atom-Probe Tomography of Grain Boundary Oxides in Stressed and Cold-Worked 304 Stainless Steel — •Karen Kruska[1], David W Saxey[2], George D W Smith[1], Takumi Terachi[3], Takuyo Yamada[3], and Sergio Lozano-Perez[1] — [1]University of Oxford, Department of Materials, Parks Road, OX1 3PH, UK — [2]School of Physics, The University of Western Australia, WA 6009, Australia — [3]2Institute of Nuclear Safety System (INSS), 64 Sata, Mihama-cho, Mikata-gun, Fukui 919-1205, Japan

Cold-worked 304 stainless steels (SS) are known to be susceptible to stress corrosion cracking (SCC). This study employs atom-probe tomography (APT) for local chemical analysis of the oxides formed. Autoclave experiments on a set of samples with/without cold-work prior to oxidation, and with/without stress applied during oxidation, were carried out under simulated pressurised water reactor (PWR) primary conditions. APT and analytical transmission electron microscopy (ATEM) were combined to investigate chemical and structural implications of surface and grain boundary oxidation in 304 SS. Focussed ion beam (FIB) milling was used to prepare specimens containing the same grain boundary for every analysis technique. Grain boundary and deformation band oxidation were observed in all but the unstressed and non-cold worked sample. Hydrogen associated to the Nickel-rich regions and cavities was found ahead of the Cr-rich oxide in some of the samples. The implications of these findings to current SCC models will be discussed.

MM 50.5 Thu 12:45 H 0107

The influence of Cu on the microstructure of AlCoCrFeNi high entropy alloys — •Anna Manzoni[1], Nelia Wanderka[1], Sheela Singh[1], Haneen Daoud[2], Rainer Völkl[2], Uwe Glatzel[2], B.S. Murty[3], and John Banhart[1] — [1]Helmholtz-Zentrum Berlin, Berlin, Germany — [2]Metallische Werkstoffe (Metals and Alloys), University Bayreuth, Bayreuth, Germany — [3]Metallurgical and Materials Engineering, Indian Institute of Technology Madras, Chennai, India

The microstructure of the five element equiatomic AlCoCrFeNi high entropy alloy is compared to the microstructure of six element equiatomic AlCoCrCuFeNi. The influence of Cu on the local microstructure as well as on the local concentration is investigated by SEM, TEM and atom probe tomography. Both as-cast alloys decompose into a dendritic and an interdendritic region. While the six elements alloy shows several Cu-precipitates in both the interdendritic and the dendritic region in addition to Al-Ni and Fe-Cr rich phases, the five elements alloy can be characterized by only two phases both in the dendritic and the interdendritic region. These Al-Ni rich and Fe-Cr rich phases in the AlCoCrFeNi show a very similar composition to those in the AlCoCrCuFeNi alloy. The influence of Cu seems to be limited to the creation of precipitates and to have no influence on the formation of the rest of the microstructure.