

Nanostructures in metals and intermetallics studied by TEM

C. Rentenberger¹, C. Mangler¹, C. Gammer¹, J. Rajagopalan², and H. P. Karnthaler¹

1. Physics of Nanostructured Materials, Univ. of Vienna, Boltzmanng. 5, 1090 Wien, Austria.

2. Mechanical Science and Engineering Department, University of Illinois at Urbana-Champaign, Urbana, IL-61801, USA.

christian.rentenberger@univie.ac.at

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Nanostructures of materials with structural units in the nanometer range can be divided into different categories according to the dimensions of the structural unit: (A) thin films, (B) rod-like structures and (C) nanocrystals. Transmission electron microscopy (TEM) including high-resolution TEM is appropriate to study these structures. In this report recent TEM results of nanostructures belonging to all three categories are given.

Ad A. Intermetallic $L1_0$ long-range ordered FePt is an interesting candidate for high density recording devices. According to the $L1_0$ structure that is based on the fcc structure with alternating Fe and Pt planes along the c-direction a magnetic anisotropy is observed. Using TEM the local degree of ordering and of epitaxy was studied on FePt thin films grown by molecular beam epitaxy on MgO(001) without any buffer layer [1]. Fig. 1(a) and (b) show a lattice plane image and the corresponding selected area diffraction pattern, respectively, revealing ordering domains with the c-axis perpendicular to the (001) plane of MgO. Domains of a different type with c-axis parallel to MgO(001) have not been encountered. Atomic resolution TEM images (cf. Fig. 1(c)) show that the lattice misfit between FePt and MgO is accommodated by an array of interfacial dislocations.

Ad B. Long-range ordered intermetallics show extensive chemical disordering by severe plastic deformation (SPD) [2, 3]. TEM studies of $L1_2$ ordered Cu_3Au reveal the local destruction of chemical order by the formation of antiphase boundaries (APB) forming tubes [3]. Fig. 2 shows a dark-field TEM image using superlattice reflection $\mathbf{g}=[1-10]$. The dark lines and bands observed in Fig. 2 (parallel to the dashed line) correspond to deformation-induced nanostructures in the form of nano-sized tubes composed by APB faults on different planes (cf. inset of Fig. 2).

Ad C. Intermetallic long-range ordered alloys with high ordering energies (FeAl, Ni_3Al) can be rendered chemically disordered by SPD and not by quenching. In this case chemical disordering is accompanied with the formation of a nanocrystalline structure [2]. Thermal annealing of the disordered nanocrystalline structure can be used to study the restoration of the ordered structure of nanocrystals. By the dark-field images using superlattice reflection (100) as shown in Fig. 3, B2 long-range ordered nanodomains within FeAl nanograins can be revealed after annealing (up to 220°C). The intensity profile (cf. Fig. 3(b)) of the corresponding diffraction pattern (using software program PASAD [4]) shows the effect of re-ordering by the emergence of superlattice reflections.

Ad A+C. In this example, recent results on freestanding thin films (160 nm thick) of gold consisting of nanocrystals are presented. Due to the special design of the sample (cf. Fig. 4(a)) a structural TEM study concomitant with quantitative stress measurements can be carried out during the in-situ deformation in the TEM. Fig. 4(b) shows a bright-field TEM image of a grain containing dislocations (indicated by arrows) that become glissile at stresses corresponding to the microplastic regime. From the in-situ TEM study the extended microplastic regime of nanocrystalline samples can be directly correlated to the structural changes observed during the deformation.

1. B. Laenens, et al., *J. Appl. Phys.* **105** (2009) p. 073913.
2. C. Rentenberger, H. P. Karnthaler, *Acta mater* **53** (2005) p.3031.
3. C. Rentenberger, H. P. Karnthaler, *Acta mater* **56** (2008) p.2526.
4. C. Gammer, et al., *Proc. Microscopy Conference 2009, Graz*.
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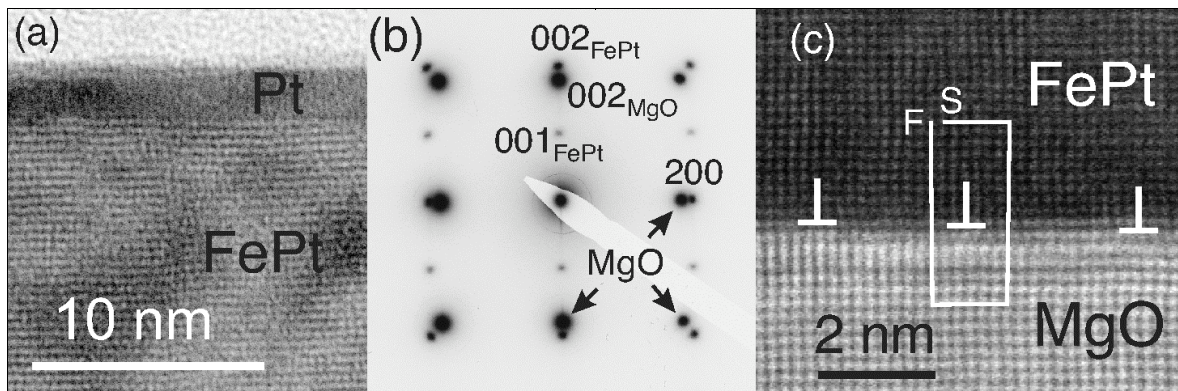


Figure 1. TEM images of $L1_0$ ordered FePt grown on MgO. (a) Lattice plane image showing the long-range periodic structure. (b) Diffraction pattern indicates the presence of ordered domains with c-axis perpendicular to the interface FePt-MgO. (c) High resolution TEM image showing an array of interfacial misfit dislocations; one Burgers circuit (S-F) is shown.

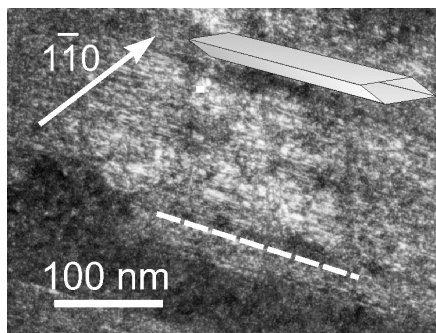


Figure 2. Cu_3Au , highly deformed. TEM dark-field image using superlattice reflections shows the presence of nano-sized tubes (cf. sketch as inset).

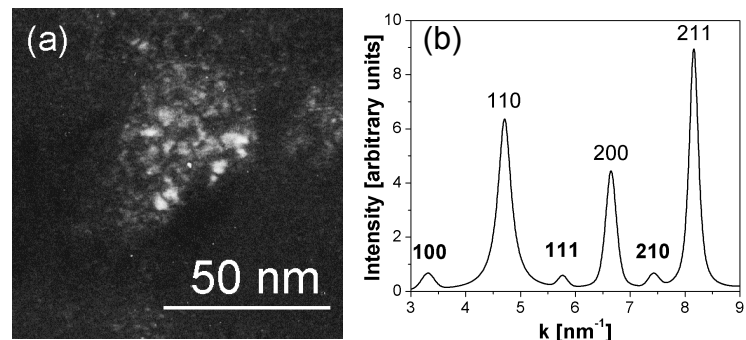


Figure 3. FeAl re-ordered at $220^\circ C$. (a) Dark-field TEM image using the superlattice reflection (100) shows tiny ordered domains within nanometer-sized grains. (b) Re-ordering leads to superlattice reflections (indicated bold) in the intensity profile of the diffraction pattern.

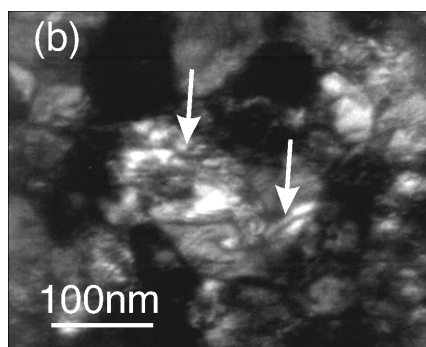
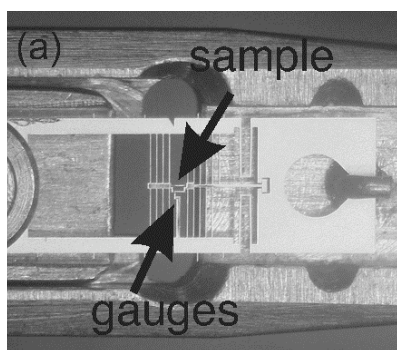


Figure 4. (a) Free-standing thin film sample of Au mounted in the in-situ deformation holder. (b) In-situ TEM bright-field image out of a movie showing the dislocation movement.