MS-4-P-5706 Analysis of nanocrystals embedded in Co,Ti made amorphous by severe plastic deformation

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Pure components were used to make the Li1 long range ordered intermetallic Co,Ti alloy that was homogenised for 100 hours at 950 °C to dissolve dendritic structures. To accomplish an amorphous phase the alloy was severely plastically deformed (SPD) by high pressure torsion at 4 GPa with 80 rotations. Methods of transmission electron microscopy (using a Titan operating at 300 kV) were applied to analyse the specimens. After SPD the specimens contain both, crystalline and amorphous regions. In the amorphous phase nanocrystals are encountered. Fig.1 shows an annular dark field (ADF) image with nanocrystals having dark or bright contrast corresponding to the given diffraction conditions. It is the aim of this study to find out whether the nanocrystals contain retained crystalline structures or if they were formed during the SPD process.

Local chemical variations in the specimens are studied by high angle annular dark field (HAADF) images and electron energy loss spectroscopy (EELS). Fig.2 shows a HAADF image of the amorphous region with embedded nanocrystals (cf. Fig.1). The contrast variations in the HAADF image indicate chemical variations in the specimen. To analyse the chemical composition of the nanocrystals EELS line scans were acquired for the nanocrystals and the surrounding matrix. The elemental concentrations of Ti, Co and O are deduced from the integrated EELS intensities between different positions; their concentration profiles are shown in Fig.3. The evaluation leads to the result that the nanocrystals exhibit a higher ratio of Ti / Co than the surrounding amorphous phase. The concentration of Ti at the positions of the nanocrystals is about 15% higher than the one in the surrounding amorphous phase. This is an indication that the embedded nanocrystals contain Laves phases (Co,Ti$^{2.1}$ or Co,Ti) and are not retained crystalline material. Therefore, we conclude that the nanocrystals are formed in the amorphous phase during SPD by a dynamic non-polymorphic crystallisation process.

In addition, a few nanoparticles revealing pronounced dark contrast in the HAADF images were encountered. As shown in Fig.4 the concentration profiles indicate that they contain preferentially Ti and O. Their Ti concentration is about 50% higher than the one of the surrounding amorphous phase. These nanoparticles are therefore interpreted as titanium oxide particles (cf. [1]).

[1] "Fluctuation electron microscopy of an amorphous-crystalline composite material", Ebner C., Gammer C., Karnthaler H.P., Rentenberger C., this conference

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Fig. 1: Annular dark field image of a HPT deformed Co3Ti specimen showing nanocrystals embedded in an amorphous phase.

Fig. 2: HAADF image of the specimen area shown in Fig.1. The outlined regions correspond to segmented nanocrystals in the ADF image.

Fig. 3: Concentration profiles deduced from an EELS line scan across a nanocrystal showing a higher amount of titanium and a lower one of cobalt at the nanocrystal.

Fig. 4: Concentration profiles deduced from an EELS line scan across a nanoparticle showing both, a high titanium and oxygen concentration.