Alloys and Intermetallics

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Devitrification of Co_3Ti studied by in-situ heating in the TEM

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The investigation of amorphous metallic alloys is of special interest from both, a scientific point of view and because of their specific potential applications. It is the aim of this work to study the transition from the amorphous phase to the crystalline one. Therefore the stability and the phase transition of intermetallic Co_3Ti, made amorphous by severe plastic deformation (SPD), are investigated by in-situ heating in the transmission electron microscope (TEM).

Intermetallic Co_3Ti with the nominal composition of Co-23at.%Ti was made by melting and mixing Co and Ti of 99.995% purity in an induction furnace. The alloy was annealed at 950°C for ~100h under a static Ar overpressure to achieve the $L1_2$ long range ordered phase. Disc shaped specimens were cut out of the rod formed alloy by spark erosion and deformed by SPD using the method of high pressure torsion (HPT) at a low deformation speed to avoid heating of the specimen. The deformation was performed using 3 different settings: 20 turns at a quasi-hydrostatic pressure of 4GPa, 80 turns at 4GPa and 20 turns at 8GPa.

The analysis in the scanning electron microscope of cross sections of the HPT deformed discs showed that only the samples deformed by 20 turns at 8GPa were amorphous in the outer regions, while all others, deformed at 4GPa, showed still a largely crystalline structure.

The TEM micrographs show that there are a few nanocrystals present in the amorphous matrix of the specimen deformed at 20 turns at 8GPa (cf. Fig. 1). These crystals have bright or dark contrast in bright field images, depending on their orientation respective to the incoming electron beam. Crystals orientated near Bragg condition scatter strongly and therefore they have a dark contrast relative to the amorphous matrix. Other crystals orientated in such a way, that they scatter the beam stronger in the forward direction show a brighter contrast in the image. The amorphous regions scatter mostly diffuse because of the lack of a periodic structure and therefore produce a contrast of medium intensity.

A differential scanning calorimetry (DSC) measurement was done to determine the temperature of the phase change from the amorphous to the crystalline phase. In an in-situ experiment the specimen was heated inside the TEM stepwise using a heating holder and images were taken for each temperature step at different times of the isothermal. Devitrification is occurring especially near the rim of the TEM foil. Fig. 2 shows a sequence of bright field images taken from the same area at 300°C, 400°C and 430°C. With increasing temperature the volume fraction of the crystalline phase increases; still the size of the crystals remains 10-50 nm due to the high nucleation rate. The nanocrystals contain planar defects as shown in Fig. 3(a), indicated by the arrows. In addition selected area (SA) diffraction patterns were taken to analyse the crystalline structure of the devitrified areas, cf. Fig. 3(b). The profile analysis with PASAD [1] shows clearly the occurrence of hcp Co crystals. The fact that it is not so easy to identify the nanocrystals containing the Ti rich phases (Co_2Ti, $Co_{2.1}Ti_{0.9}$) from the integrated profiles can be caused by their faults, small size, and complicated Laves structures, that all lead to a reduction of intensity and broadening of the lines. It is interesting to note, that the temperature at which the crystals start to grow is about 80K below the crystallization temperature of the bulk amorphous alloy as deduced from the DSC measurement. Therefore our results are in agreement with a surface induced phase transition, as known from surface melting. In addition, it seems that the nanocrystals already present in the amorphous matrix before heating, do not act as nucleation centers. This is concluded since the newly formed nanocrystals do not concentrate near the already existing ones, which themselves do not grow. The results of this in-situ study will be compared with those of bulk annealed samples.

1. C. Gammer, C. Mangler, C. Rentenberger and H.P. Karnthaler. Scripta Materialia 63, (2010), 312. doi:10.1016/j.scriptamat.2010.04.019
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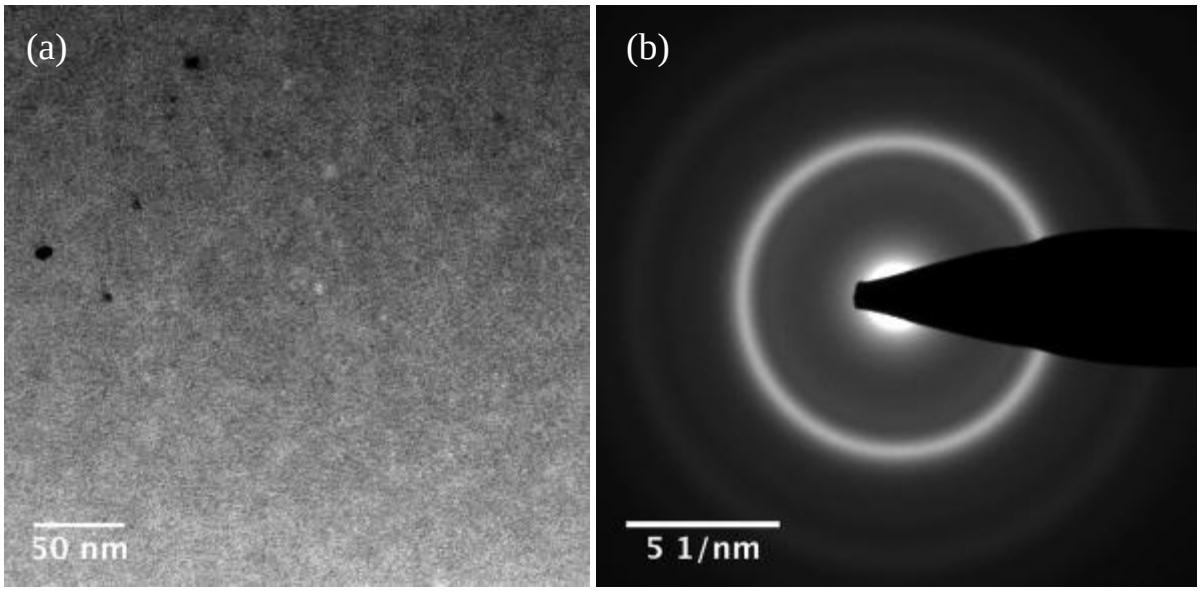

Figure 1. Amorphous Co_3Ti made by SPD. (a) TEM bright field image of the amorphous region showing a few nanocrystals that form during the deformation. (b) SA diffraction pattern of (a) showing amorphous rings.

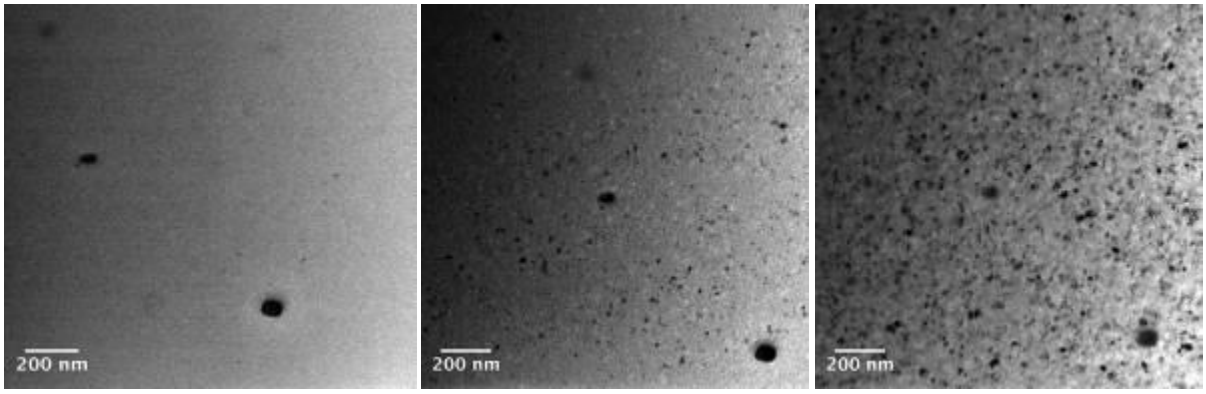

Figure 2. TEM bright field micrographs of the devitrification of amorphous Co_3Ti during in-situ heating, (a) observed at 300°C, (b) 400°C and (c) 430°C.

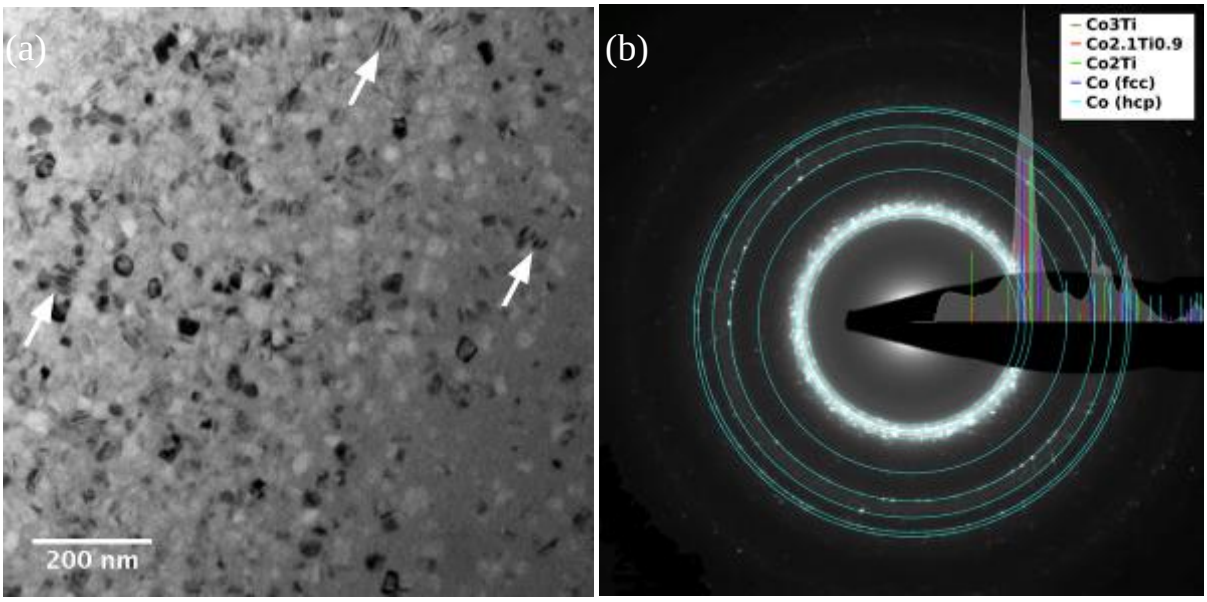

Figure 3. Devitrification of amorphous Co_3Ti after in-situ heating to 430°C observed at room temperature. (a) Bright field micrograph of the newly formed crystalline phase, arrows indicating planar defects present in the crystals. (b) SA diffraction pattern with PASAD [1] profile analysis compared with X-ray structure data.