MS.6.P170
Statistical analysis of nanocrystals embedded in the amorphous phase of a Co-Ti alloy studied by TEM

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Keywords: nanocrystals, amorphous phase, severe plastic deformation

Amorphous alloys combine desirable properties of both conventional crystalline alloys and oxide glasses. They exhibit e.g.: the maintenance of dimensional accuracy, room temperature strength and a high elastic strain as compared with their crystalline counterparts. Their big disadvantage is a very low tensile ductility. Nevertheless, most amorphous alloys are not intrinsically brittle and possess mechanisms for plastic flow [1]. In the present study we show that in an amorphous alloy small crystals exists and it is thought that they could have a positive influence on the macroscopic properties. A Co-23at.%Ti alloy is studied for crystallisation in its amorphous phase with a transmission electron microscope (TEM) using 200 kV. The aim is to determine the size and quantity of crystals in the amorphous phase. The amorphous phase is accomplished by severe plastic deformation using high pressure torsion. A crystalline Co-Ti specimen is compressed at 4 GPa between two anvils which are then turned against each other for 20 rotations. This induces lattice defects and increases the free enthalpy until it is higher than the free enthalpy of the amorphous state and the crystal turns into the amorphous phase. An area of approx. 530 nm² with a thickness of approx. 40 nm is considered in the amorphous phase of the Co-Ti specimen. Fig. 1 shows the considered area and a large crystalline area C on the left side due to the inhomogeneous deformation of the specimen. The local thickness is determined by local intensity measurements at different tilt angles of the specimen. A functional relation between the specimen thickness and the measured intensity in an amorphous specimen is given in [2]. One can obtain the local thickness as a fit parameter of the intensity - tilt angle relation. The nanocrystals embedded in the amorphous phase are studied by bright field and dark field images captured with the CCD-camera of the TEM (see Fig. 2 and 3). The contrast theories for the amorphous phase and that for the crystalline phase are quite different. The amorphous phase can be described by diffuse scattering [2] while crystals are described by diffraction. It is interesting to note that there are bright areas in the bright field images and also dark areas in the dark field images (cf. Fig. 2 and 3). If a crystal is far off a Bragg condition, it will cause a bright contrast in a bright field image because of the intensity loss of the incident electron beam in the surrounding amorphous phase due to diffuse scattering. Likewise the amorphous phase has a certain amount of intensity in the dark field image. Thus crystals which do not satisfy the selected diffraction conditions for the dark field image will cause a dark contrast compared to the surrounding amorphous phase.
In order to recognize all crystals in the amorphous phase the specimen is tilted in little steps for the bright field images. Dark field images are taken with an objective aperture (20 µm) set on the first diffuse ring in the diffraction pattern as shown in Fig. 4 (There also lie diffraction spots of the nanocrystals.) By shifting the diffraction pattern along the ring all crystals with reflections included in the objective aperture are contained in tilted dark field images. A main task is to find a procedure for the evaluation of the images. There are about 15 images to evaluate for both imaging methods. The crystalline areas are segmented manually for the bright field images while the evaluation of the dark field images is done partly automatised by the application of digital image processing. Thereby the bright and dark areas in the dark field images are segmented separately. The procedure must allow manual changes and corrections because of overlapping crystalline areas in some images and differences of the image positions in the specimen. A combined mask image of bright field and dark field images is given in Fig. 5. The present results show that the nanocrystals have an average diameter of 7 nm. The value is obtained by a measurement of the crystal areas and the assumption of their circular forms. This is a valid approximation according to the observed morphology. The crystals cover approx. 1-2% of the considered volume of the specimen. Results for the dark field images exhibit smaller values than those for the bright field images which could be related to the different segmentation methods. Fig. 6 shows a comparison of the crystal diameters for both the bright field and dark field evaluation.

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2. L. Reimer, H. Kohl in "Transmission Electron Microscopy", 5. ed. , (Springer, New York) (2008), p.196.

3. We kindly acknowledge the support by the Austrian Science Fund (FWF): [P22440].

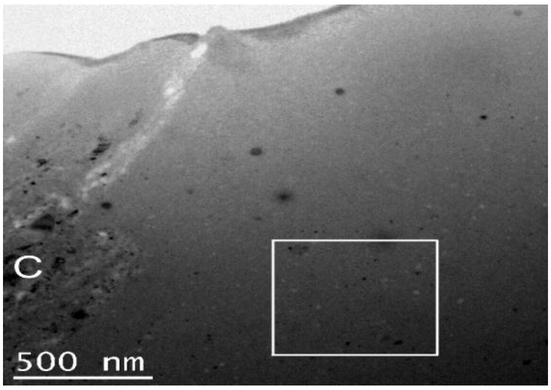

Figure 1. Amorphous phase with the evaluated area and a crystalline area C on the left side

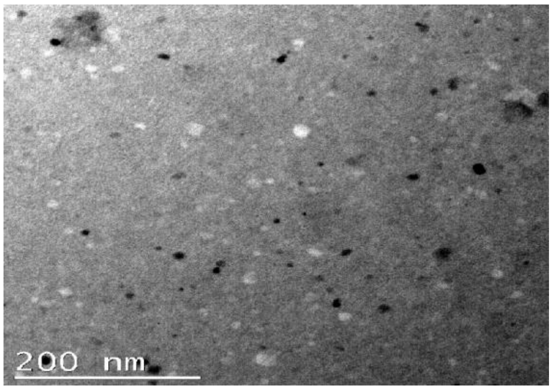

Figure 2. Bright field image of the evaluated area, showing dark but also bright nanocrystals

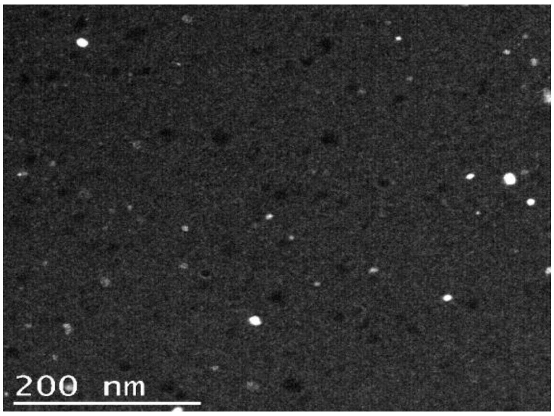

Figure 3. Dark field image of the evaluated area (same area as in Fig. 2), showing bright but also dark nanocrystals.

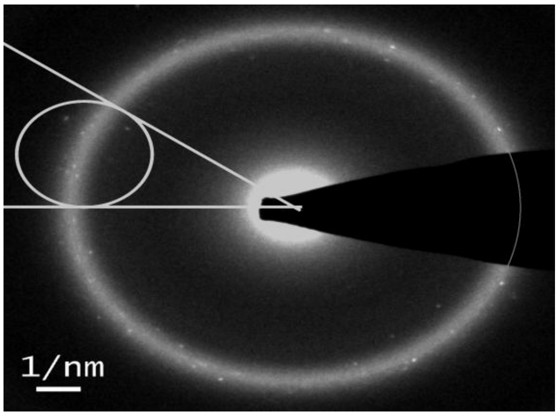

Figure 4. Diffraction pattern with marked objective aperture lying on the diffuse ring of the amorphous phase and containing spots of the nanocrystals

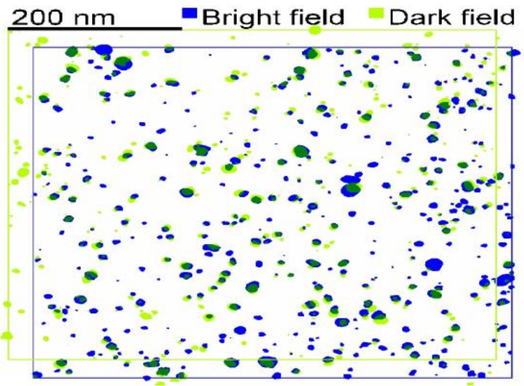

Figure 5. Combined mask image of the segmented bright field and dark field images of the nanocrystals

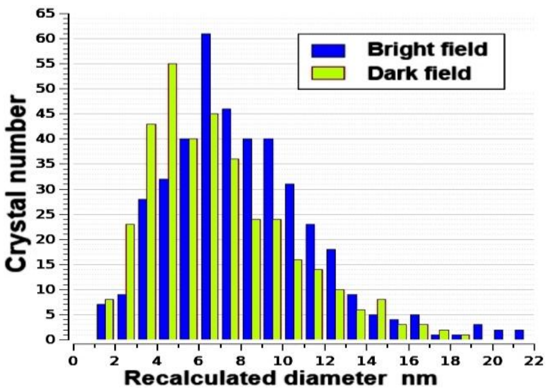

Figure 6. Frequency - diameter graph of the crystals for both the bright and the dark field evaluation