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 this 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 (BF) and dark field (DF) 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 BF images and also dark areas in the DF images (cf. Fig. 2 and 3). If a crystal is far off a Bragg condition, it will cause a bright contrast in a BF 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 DF image. Thus crystals which do not satisfy the selected diffraction conditions for the DF image will cause a dark contrast compared to the surrounding amorphous phase.

Crystals with a contrast similar to that of the amorphous phase are not detected. To improve this the specimen is tilted in small steps for the BF images. In contrast the DF images are taken in one tilt position only with an objective aperture (20 µm) set on the first diffuse ring as shown in Fig. 4. (On this ring there are also the diffraction spots of the nanocrystals.) By shifting the aperture along the diffraction ring the crystals were imaged in tilted DF mode.

A main task is to find a procedure for the evaluation of the images. There are 17 images to evaluate for both imaging methods. The crystalline areas are segmented manually for the BF images while the evaluation of the DF images is done partly automatised by the application of digital image processing. Thereby the bright and dark areas in the DF 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 BF and DF images is given in Fig. 5.

An average diameter (7 ± 1 nm) of the nanocrystals is deduced from measuring the areas of the crystals and by assuming that they have circular forms. This is in agreement with the observed morphology. The density of the crystals is 1-2% of the considered volume of the specimen. In the DF images less crystals are recognized than in the BF images because with DF images we consider the first diffuse ring in the diffraction pattern only and not spots lying on outer rings. Also with DF images only one specimen tilt is taken into account. Fig. 6 shows a comparison of the calculated crystal diameters deduced from both BF and DF images.

1. A. L. Greer, E. Ma, MRS Bulletin 32 (2007), p. 613.
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, I1309].

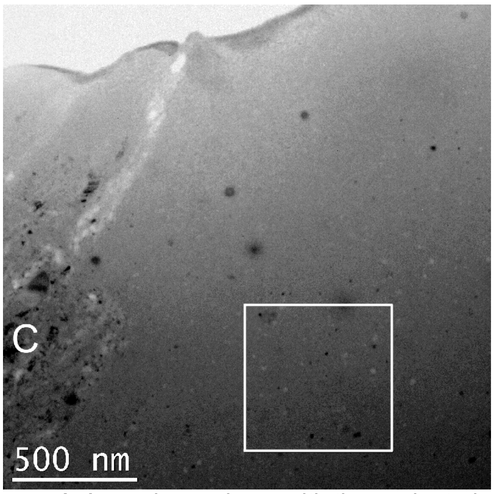

Figure 1. Amorphous phase with the evaluated area and a crystalline area (marked with C)

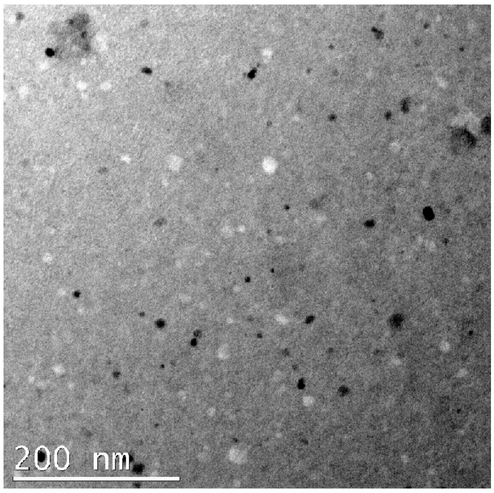

Figure 2. Bright field image of the evaluated area, showing dark but also bright nanocrystals

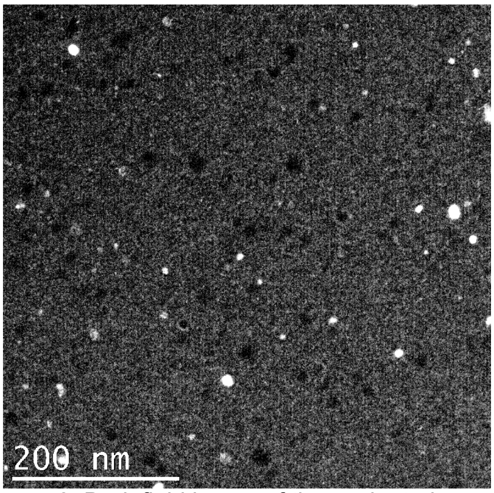

Figure 3. Dark field image of the evaluated area (same area as in Fig. 2), showing bright but also dark contrast of the nanocrystals

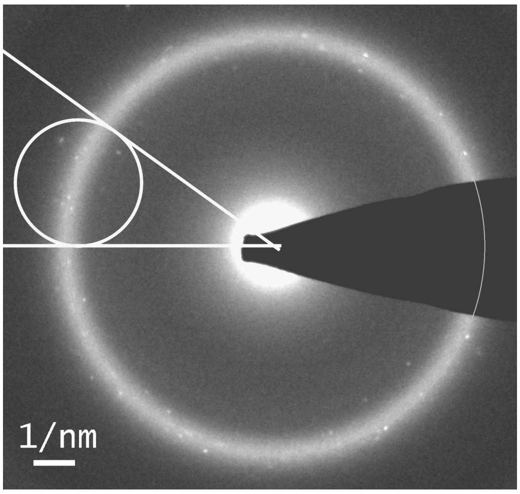

Figure 4. Diffraction pattern with marked objective aperture lying on the diffuse ring of the amorphous phase and containing spots of the nanocrystals

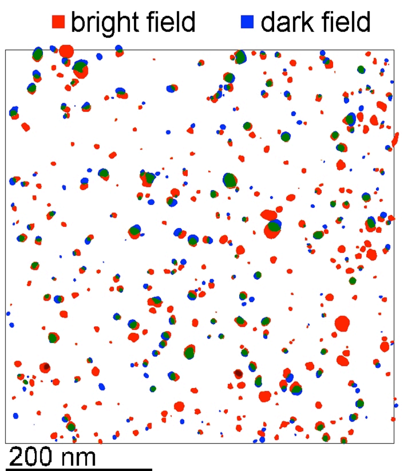

Figure 5. Combined mask image of the segmented bright field and dark field images of the nanocrystals

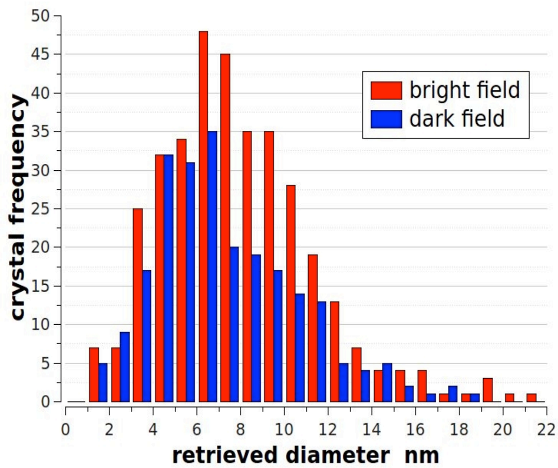

Figure 6. Frequency versus diameter of the nanocrystals taken from both bright field and dark field