TEM investigations of bulk nanocrystalline intermetallics in 3D

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Bulk nanocrystalline intermetallics receive increasing interest since they can show novel properties as high strength combined with high ductility [1]. Methods of severe plastic deformation, e.g. high pressure torsion (HPT), are frequently used to produce nanocrystalline intermetallics. In the present work electron microscopy results of two intermetallic compounds refined by HPT will be presented.

Figure 1 shows the evolution of the nanocrystalline structure in HPT deformed B2 structured FeAl. In the beginning of the deformation nanocrystalline bands form that are embedded in a still coarse crystalline matrix (cf. Fig. 1a). During further deformation their volume fraction increases until the whole sample is nanocrystalline (cf. Fig. 1b). Grain size histograms deduced from dark-field images can be used for a grain size determination. Nevertheless, care has to be taken due to the complex moiré contrast arising in nanomaterials containing a large number of subgrains and dislocations [2]. Using diffraction, X-ray profile analysis is routinely applied for the analysis of nanomaterials. Recently it was shown that profile analysis of selected area electron diffraction patterns (PASAD) can be carried out for a quantitative analysis of nanomaterials on a local scale [3]. This allows to obtain quantitative results like the subgrain size of regions specifically selected in TEM images. The application of PASAD to SAD patterns shown in Figure 1 reveals that the subgrain size in the nanocrystalline band is the same as that of the final nanocrystalline structure.

In order to obtain 3D information usually two different sections are necessary. Figure 2a shows a cross section of nanocrystalline FeAl. The grains are elongated in the shear plane. To analyse the morphology of the subgrains using only one section (the top-view), TEM diffraction patterns showing rings were recorded with a large range of tilting angles and evaluated in different directions using PASAD (cf. Fig. 2b). Figure 2c shows a model of the elongated subgrains. Tilting the sample leads to a reduction of the size measured normal to the tilt axis allowing to determine the elongation (cf. Fig. 2c) [4].

To obtain 3D information of HPT deformed $L1_2$ structured Zr_3Al on different length scales SEM and TEM methods were combined. The SEM image (cf. Fig. 3) shows that the sample is very inhomogeneous containing a complex mixture of coarse crystalline and nanocrystalline material [5]. The transition region (cf. arrow in Fig. 3) was studied in more detail from TEM samples prepared by focused ion beam. The coarse crystalline region (cf. Fig. 4a) shows elongated grains (>1µm) containing a large number of dislocations, whereas in the refined region (cf. Fig. 4c) very small grains (<10nm) can be observed. The corresponding diffraction pattern (cf. Fig. 4d) shows no texture indicating that the deformation mechanism has changed from a dislocation to a grain boundary mediated one. This change is responsible for a softening in the refined region leading to a pronounced inhomogeneous deformation even after 100,000% of deformation. In the case of FeAl the evolution of the nanocrystalline structure occurs also inhomogeneously but leads to a homogeneous nanocrystalline structure as the refined structure is harder than the coarse crystalline one.

In summary, TEM methods are essential for the characterization of nanocrystalline materials produced by severe plastic deformation. Structural inhomogeneities or elongated

nanostructures can be studied using local 3D TEM investigations. The combination of SEM and TEM imaging as well as TEM diffraction can give additional insight into the material.

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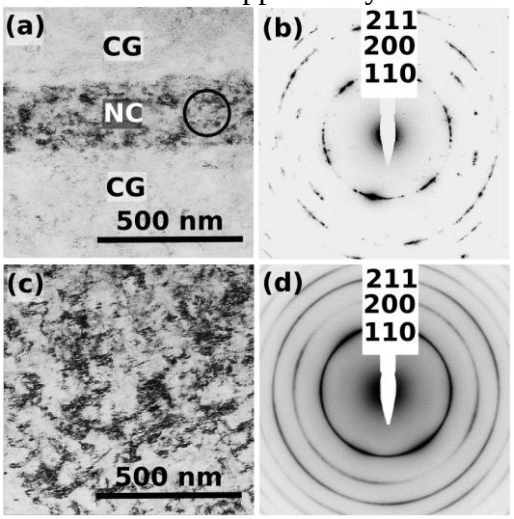

Figure 1. TEM study of the nanocrystalline (NC) structures occurring in HPT-deformed FeAl as a function of shear strain γ. (a) γ=1200%; bright-field (BF) image showing a band containing NC material embedded in coarse-grained (CG) material. (b) SAD of the area indicated in (a). (c) At γ>7100% the sample is homogeneously NC. (f) SAD of (e).

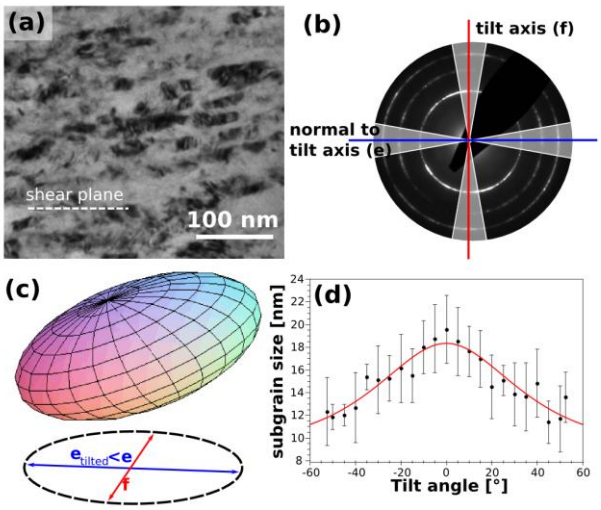

Figure 2. (a) Cross section image showing elongated grains. (b-d) To study the elongation of the crystallites from a top-view sample a large number of SAD patterns was recorded and the size of the platelet shaped crystallites was evaluated in the direction e+f for various tilt angle. When tilting the sample, the size measured in direction e decreases allowing to calculate the elongation.

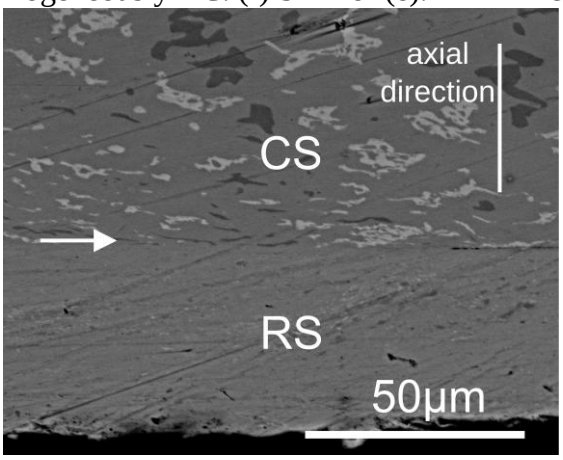

Figure 3. Cross section SEM image of a HPT disk deformed 10 turns. Different regions are clearly distinguishable and separated by a sharp line (marked by arrow): a coarse crystalline structure (CS) with different phases and a region with homogeneous contrast indicating that the structure is refined (RS).

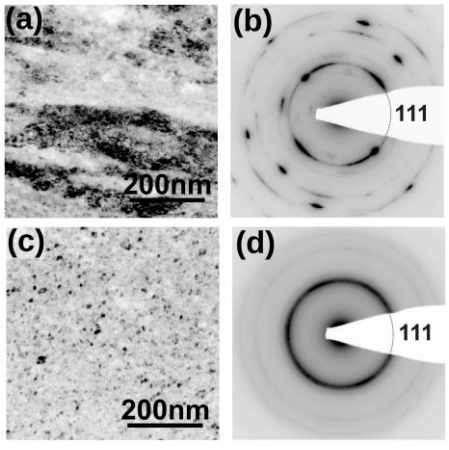

Figure 4. Cross section dark field images of the HPT disk shown in Figure 3 (N.B. black/white inverted for better visibility). (a) is taken from the coarse crystalline region and (c) from the refined structure. The grain size decreases from >1 µm to <10 nm within a few µm.