SEM and TEM cross-section study of inhomogeneities in Zr_3Al deformed by high pressure torsion

D. Geist[1], A. Thaller[1], C. Rentenberger[1], J. Bernardi[2] and H. P. Karnthaler[1]

1. Physics of Nanostructured Materials, Faculty of Physics, University of Vienna
Boltzmanngasse 5, 1090 Vienna, Austria
2. USTEM, Vienna University of Technology
Wiedner Hauptstrasse 8-10, 1040 Vienna, Austria

david.geist@univie.ac.at
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High Pressure Torsion (HPT) is a modern technique to render bulk materials nanocrystalline or amorphous by severe plastic deformation. In the present work, we show by SEM and TEM methods that the deformation of $L1_2$-ordered intermetallic compound Zr_3Al is unexpectedly strongly inhomogeneous in areas of constant shear strain.

Zr_3Al cannot be produced as a single-phase alloy, so discs consisting of 90% Zr_3Al, the rest being residual α-Zr and Zr_2Al, were cut by spark erosion. They were deformed by HPT at a pressure of 4 GPa at room temperature to a shear deformation of 27000% (10 revolutions). SEM cross-sections of the deformed samples were analysed using backscattered electron detection. In addition, TEM samples were prepared by focused ion beam and analysed using an acceleration voltage of 200 kV.

Due to the multiphase structure of the sample, morphological changes are visible in the SEM images by the change of shape and distribution of the embedded phases: α-Zr (bright) and Zr_2Al (dark). The SEM studies show that HPT deformation leads to two different structures depending on the pre-HPT surface treatment. Surfaces damaged by the preparation process (e.g. spark cutting) became amorphous by HPT deformation (Fig. 1a). Microhardness tests showed that for Zr_3Al the amorphous material is softer than the deformed crystalline material. Therefore, further deformation is carried by the amorphous surface layer, leaving the morphology of the different phases in the sample interior almost unchanged. When the damaged surface layer was removed prior to HPT, no amorphous surface layer was formed during HPT deformation, but amorphous deformation bands arose within the sample (Fig. 1b).

Fig. 2a shows a TEM bright-field image of a transition region between amorphous and crystalline material (cf. white box in fig. 1b). A sharp interface can be seen. Selected area diffraction patterns were taken from different regions: a crystalline one (Fig. 2b), an amorphous one with embedded nanocrystals in the vicinity of the crystalline region (Fig 2c) and an amorphous one about 10µm away from the interface (Fig. 2d). The number of nanocrystals decreases with distance from the phase boundary. The images indicate that accumulation of lattice defects leads to deformation induced amorphization via the formation of nanocrystals.

It is concluded that the surface treatment of the samples prior to deformation determines whether an amorphous layer is formed at the sample surface or in the sample interior. If the amorphous layer forms at the surface, its thickness is roughly constant for different shear strains, whereas in the other case, the thickness of the amorphous bands forming in the sample interior is strongly dependent on the shear strain.

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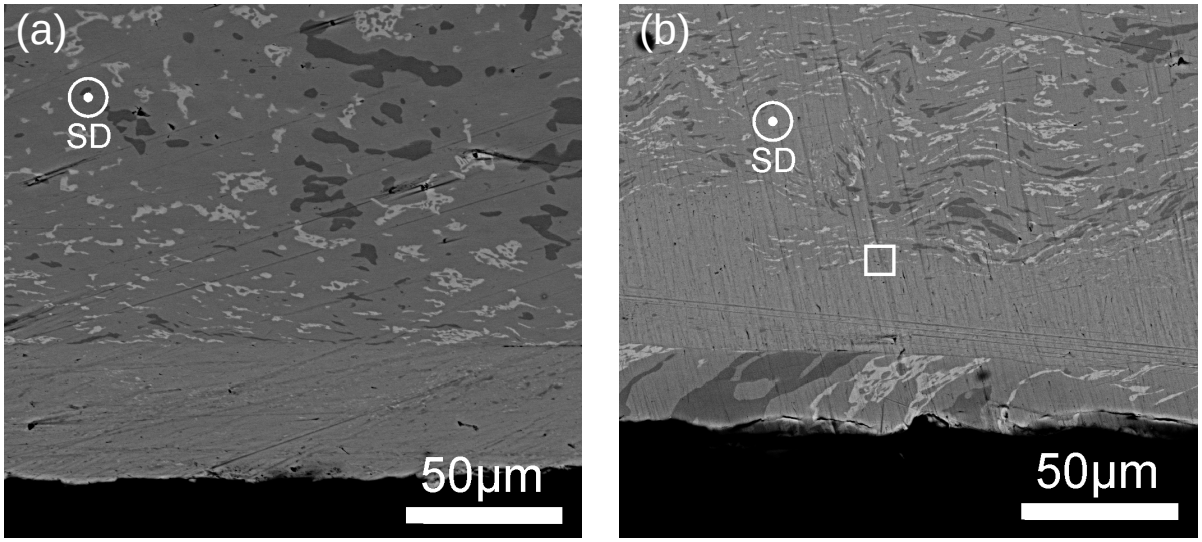

Figure 1. Cross-section images of deformed Zr_3Al. SD indicates the shear direction. (a) HPT-deformed sample prepared by spark-cutting showing an amorphous surface layer. (b) Sample with as-prepared surface layer removed prior to deformation. The white box indicates a transition region as analyzed in Fig. 2.

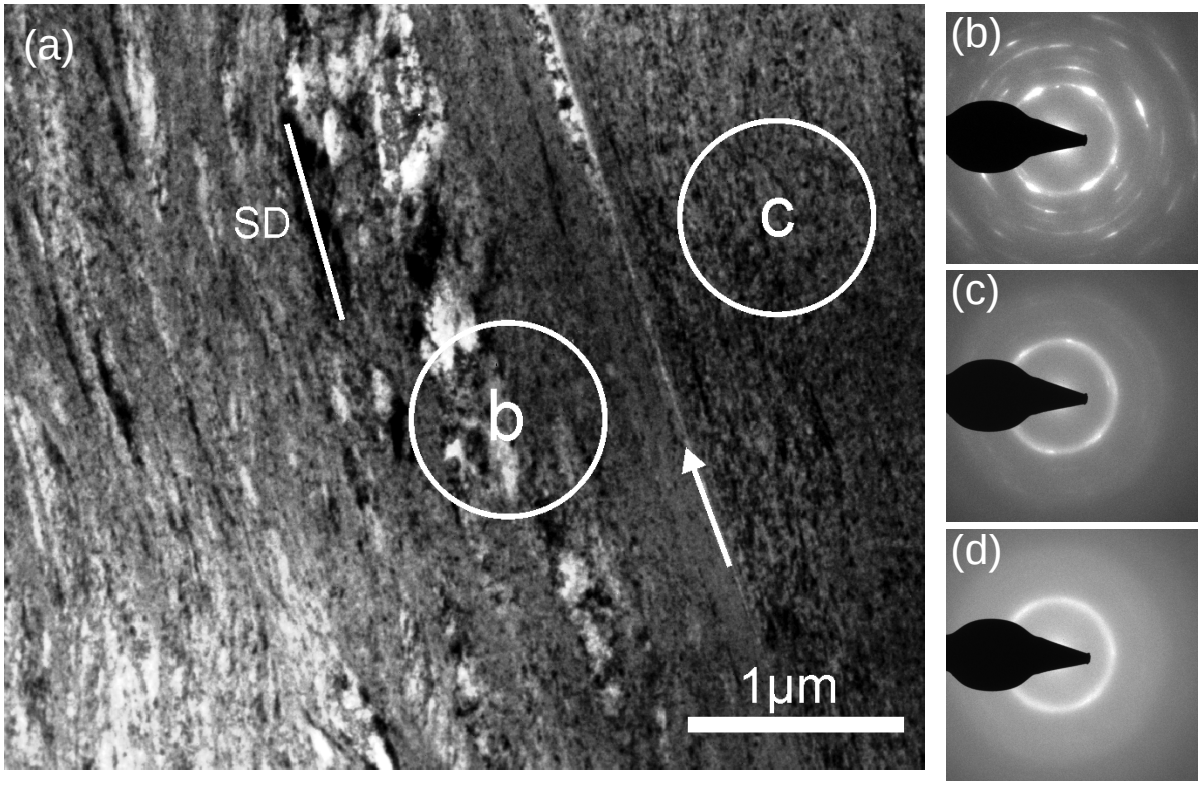

Figure 2. TEM bright-field image and diffraction patterns of Zr_3Al. SD indicates the shear direction. The interface is marked by an arrow. The diffraction patterns are taken from the crystalline region (b), an amorphous region near the phase boundary with embedded nanocrystals (c), an amorphous region ~10µm away from the phase boundary (d).