Radial sections of severely deformed NiTi shape memory alloys studied by TEM and HRTEM

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Shape memory properties are of great interest for various applications. The shape memory effect is caused by a martensitic phase transformation that can be significantly affected by a small grain size [1]. Refining of the microstructure can lead to enhanced shape memory properties. Severe plastic deformation and subsequent annealing is a promising route for achieving bulk nanocrystalline shape memory alloys. Severe plastic deformation by high-pressure torsion (HPT) of NiTi shape memory alloys can lead to an amorphous phase. Nanocrystals surviving the amorphization significantly impact the crystallization of the amorphous phase upon annealing [2]. In this study, radial section of the HPT samples were studied to analyze the nanocrystals embedded in the amorphous phase by transmission electron microscopy (TEM) including high-resolution TEM (HRTEM).

Using pure components, a Ni-50.1at.%Ti alloy was made that shows a martensitic phase transformation from the cubic B2 phase to a monoclinic B19′ structure (martensite) upon cooling that is completed at room temperature. Discs with a diameter of 8 mm and a thickness of 0.7 mm were deformed by HPT applying 12 turns at a pressure of 4 GPa. Radial section TEM specimens with a foil normal along the radius of the HPT discs were obtained by a special procedure involving cutting, grinding and final ion beam thinning at a rather low glancing angle (10°) and low energy using a Bal-Tec RES 101. The specimens were investigated using a CM 200 and a CM 30ST operating at 200 and 300 kV, respectively.

TEM bright-field images show band shaped regions of amorphous and nanocrystalline phases (cf. Fig. 1). The interfaces between them are rather sharp, straight and almost perfectly orientated along the shear direction. The selected area diffraction pattern (SADP) of the amorphous bands consist of a rather broad and diffuse rings; superimposed weak reflections indicate that tiny crystals survived within the amorphous phase (cf. Fig. 1b). In the nanocrystalline bands, a texture is encountered since the rings of the SADP show a heterogeneous distribution of the diffracted intensity (cf. Fig. 1c). Fig. 2 shows elongated nanocrystals embedded in the amorphous phase that are almost parallel to each other and to the shear direction. Frequently, constrictions are observed along the length of the nanocrystals leading to their fragmentation. As analyzed by TEM dark-field images almost the same crystalline orientation is observed along the length of the crystals that can be several micrometers long (cf. Fig. 2b). Fig 2c shows a HRTEM image of a fragment of the elongated nanocrystals that contains B19′ martensite. The interface between crystalline and amorphous phase is almost parallel to (001) and sharp even at the atomic scale.

Based on the TEM results, it is concluded that the mixture of amorphous and nanocrystalline phases (cf. Fig. 1) arises by a heterogeneous deformation occurring via the formation of shear bands. Caused by the shear deformation, grains become thin, elongated and are gradually rotated towards the shear direction finally leading to the debris structure observed in Fig. 2.

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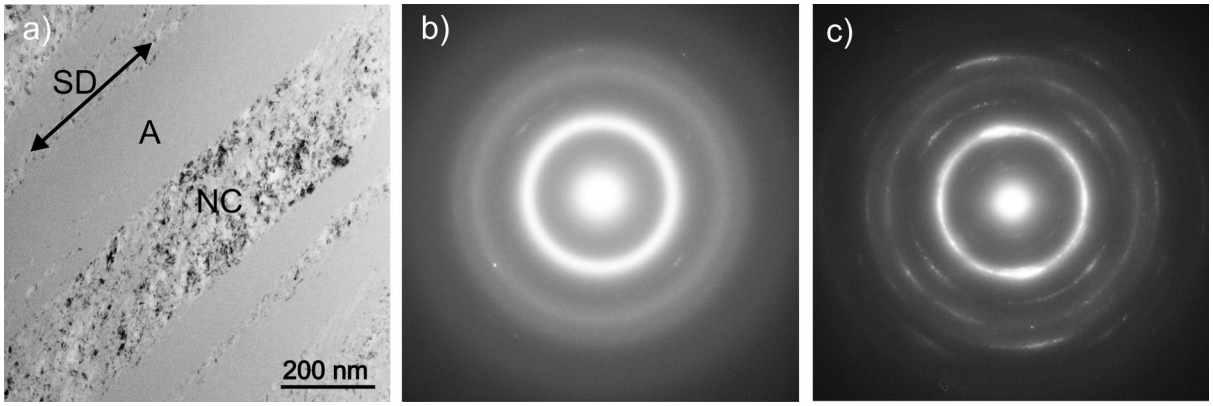

Figure 1. HPT deformed NiTi. Radial section. a) TEM bright field micrograph. Band shaped regions containing amorphous and nanocrystalline phase are marked by A and NC, respectively. A and NC are separated by rather sharp and straight interfaces. The shear direction SD is indicated. b) SDAP of A. Weak spots superimposing diffuse diffraction rings arise by nanocrystals embedded in the amorphous matrix. c) SDAP of NC. A texture of the nanocrystals leads to diffraction rings that show a heterogeneous distribution of the intensity.

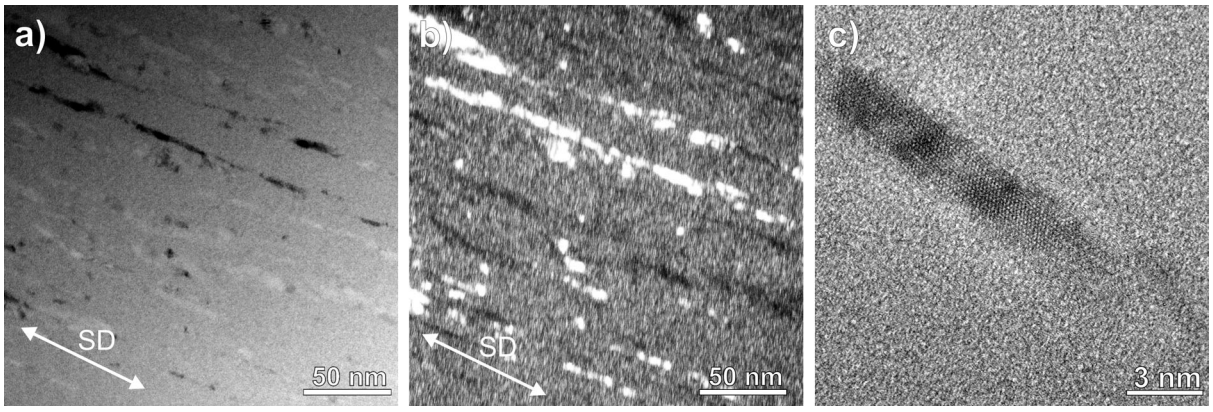

Figure 2. HPT deformed NiTi. Radial section. a) TEM bright-field image. Thin and elongated crystallites are running almost parallel to each other and to the shear direction SD. b) TEM dark-field image of the same area as in a). Along their length, the crystals show a rather homogeneous contrast indicating the almost constant crystalline orientation. c) HRTEM image. Even on the atomic scale the nanocrystals have straight and sharp interfaces.

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