TEM investigations of severely deformed and annealed NiTiHf high temperature shape memory alloys

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NiTi based shape memory alloys show a martensitic phase transformation from a cubic high temperature phase (B2 austenite) to a monoclinic structure (B19´ martensite). In NiTi, a crystalline to amorphous phase transformation can be obtained by methods of severe plastic deformation such as cold rolling and high pressure torsion (HPT) [1,2]. Devitrification of the amorphous phase can lead to a nanocrystalline structure. It is aim of the present work to study the microstructure of a NiTiHf high temperature shape memory alloy (martensite finish M_f and austenite finish A_f temperature of 170 and 260°C, respectively) after HPT and annealing.

A NiTiHf alloy that is martensitic at room temperature (RT) was subjected to HPT deformation at RT (discs 8 mm diameter and 0.8 mm thick were deformed applying 12 turns at 4 GPa). Specimens for transmission electron microscopy (TEM) were punched out at a distance of 2.7 mm from the centre of the HPT discs corresponding to a deformation of 25000%. In addition to HPT deformed samples, annealed ones at temperatures of 450 and 550°C for 30 min were investigated by TEM operating at 200 kV. The phase structure was analysed using selected area diffraction patterns (SADP).

After HPT, the TEM bright field image (cf. Fig. 1a) shows a martensitic microstructure containing rather large grains and some nanocrystalline areas. A stripe like contrast is frequently observed arises by twins of the martensite. A weak diffuse ring is encountered in the SADP indicating a small volume fraction of amorphous phase; whereas, B2 austenite is hardly observed (cf. Fig. 1b).

After annealing at 450°C, the TEM bright field image of Fig. 2a shows a drastic change since a nanocrystalline structure is observed. The average grain size is about 20 nm. The corresponding SADP shows that B2 occurs in the nanocrystals (cf. Fig. 2b). It should be noted that a minor volume fraction of grains > 50 nm are observed that contain B19´. Annealing at 550°C for 30 min leads to larger grains (average grain size of about 100 nm; cf. Fig. 3a). B2 and twinned B19´ is observed by SADP (cf. Fig. 3b).

Based on the experimental results (cf. Figs. 1 and 2), it is concluded that annealing of HPT deformed NiTiHf leads to grain refinement. Therefore, in NiTiHf a new pathway leading to the formation of a nanocrystalline structure is observed that is not based on the nanocrystallization of an intermediate amorphous phase as in NiTi. Possible mechanisms leading to formation of the nanograins might involve recovery and recrystallization affected by a concomitant reverse transformation (B19´ to B2). In the nanograins, the B2 austenite is stabilized at RT (i.e. even 150°C below M_f) caused by a grain size effect [1]. After annealing at 550°C, a heterogeneous phase structure of (cf. Fig. 3) occurs since grains already grown to larger grain sizes transform to B19´ upon cooling to RT whereas still some of the B2 nanocrystals survive the annealing.

1. T.Waitz et al., J. Mech. Phys. Sol. 55 (2007) p419.
2. Nakayama et al., Mat. Trans. 42 (2001), p1987

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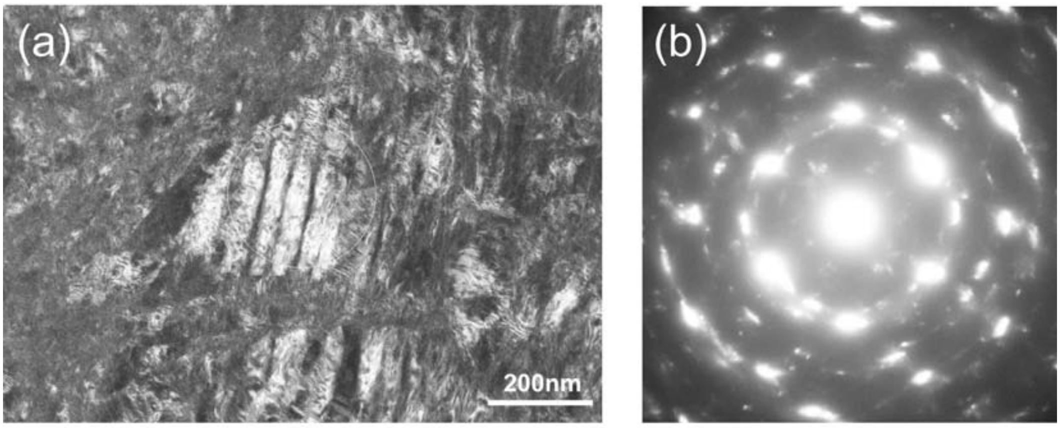

Figure 1. HPT deformed NiTiHf. (a) TEM bright field image. Heterogeneous microstructure of rather coarse grains containing B19’ only (b) SADP of the area encircled in (a). Diffraction spots of B19´ are superimposed by weak diffuse rings caused by small volume fraction of amorphous phase.

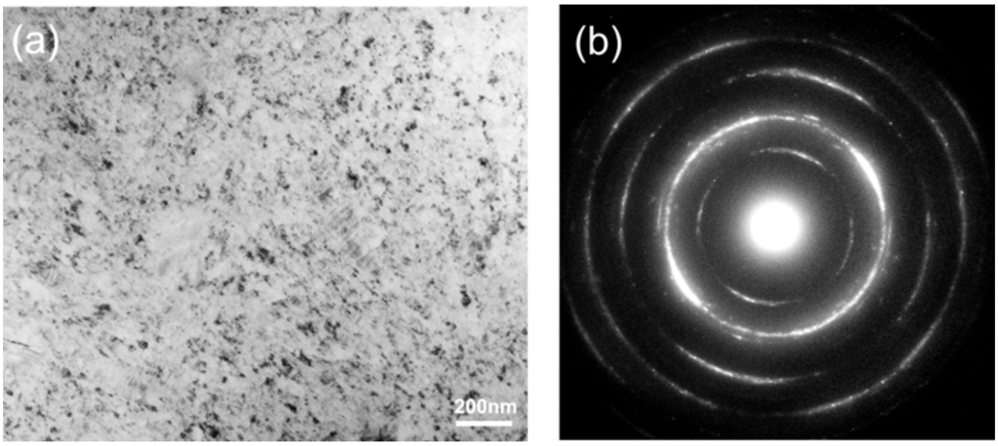

Figure 2. HPT deformed NiTiHf annealed at 450°C. (a) TEM bright field image of a nanocrystalline structure. (b) SADP. Caused by the nanograins, diffraction rings arise. Most of the diffraction spots correspond to B2. Weak rings corresponding to B19´are also observed.

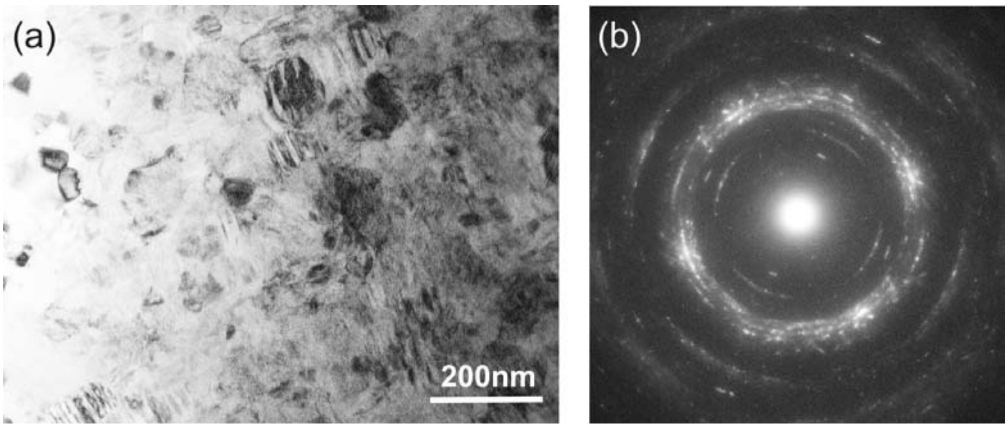

Figure 3. HPT deformed NiTiHf annealed at 550°C. (a) TEM bright field image. Grains have an average size of about 100 nm. Stripe like contrast is caused by twins of the martensite. (b) SADP. Diffraction rings correspond to both B2 and B19´.

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