

Dislocation analysis in the B2 phase of <111>-deformed NiTi single crystals

T. Simon¹, A. Kröger, Ch. Somsen¹, A. Dlouhy², and G. Eggeler¹

1. Institute for Materials, Ruhr University Bochum, 44780 Bochum, Germany

2. Institute of Physics of Materials, ASCR, Ziskova 22, 61662 Brno, Czech Republic

Tobias.Simon@rub.de

NiTi single crystals; compression testing; dislocation analysis

NiTi shape memory alloys are widely used in engineering and medical technology because depending on alloy composition they allow to exploit the one way effect (1WE) and pseudoelasticity (PE) [1]. Shape memory components often have to withstand cyclic loading. Actuators are subjected to frequent 1WE heating and cooling cycles. Pseudoelastic stents which stabilize vessels in the human body are exposed to cyclic mechanical loading at the frequency of the heart beat. Cyclic operation is associated with functional fatigue where the properties of the material (like the martensite start temperature M_s (1WE), or the plateau stress observed during a forward transformation in a stress strain hysteresis (PE)) change during cycling. This gradual change in properties is related to dislocations, which are created during cycling and which then alter the conditions for the formation of martensite. The objective of the present study is to contribute to a better understanding of the interaction between dislocations and martensite variants using transmission electron microscopy (TEM). For this purpose we deform NiTi single crystals in compression. The Ni_{50.4}Ti_{49.6} (at.%) single crystals were grown using the Bridgman technique and homogenized for 12h at 1000°C. Compression testing was performed in the [111]-direction using small cylinders (height: 9mm, diameter: 5mm) which were fabricated using spark erosion machining followed by an annealing treatment at 1000°C for 0.5h. The test cylinders were heat-treated in quartz tubes under protective argon atmosphere [2]. Fig. 1a shows the stress-strain curve for the compression in [111]-direction up to 3.3% deformation with a significant residual strain of 2.5% after unloading. Fig. 1b shows the corresponding differential scanning calorimetry (DSC) chart with apparent one step transformations on cooling and heating. A narrow shoulder on cooling (marked by an arrow in Fig. 1b) is the macroscopic result of an underlying microscopic two step transformation from B2-phase to R-phase (first step) and from B2 to B19'-phase (second step) on cooling (not discussed here).

Figs. 2 a-d show TEM bright-field micrographs of deformed NiTi cylinders with a high dislocation density. Prior to TEM analysis, the compressed sample was heated up to 200°C to remove residual martensite and to guarantee a good dislocation contrast in B2. Similar dislocation micrographs after compression testing were reported by Hurley et al. [3]. The bright-field images of Fig. 2 were taken at different g-vectors close to the zone axis of a low index pole (arrows represent directions). In Fig. 2 a and b the dislocations are visible while in the images c and d the dislocations are effectively invisible. Gb-analysis shows that most of the dislocations have screw character with a Burgers vector of type [100]. During cooling from the high temperature regime, the martensitic R-phase was observed to grow steadily at many locations. Higher degrees of undercooling were required to form B19'. B19' formed and propagated in microscopic burst like events. Evidence for the growth of B19' in the stress-field of dislocations was found.

1. K. Otsuka et al., Prog. Mater. Sci. **50** (2005) p511.
2. G. Eggeler et al., Prakt. Metallogr. **36** (2004) p125.
3. J. Hurley et al., Z. Metallkd. **94** (2003) p547.
4. We acknowledge funding by DFG (Deutsche Forschungsgemeinschaft) through SFB459.

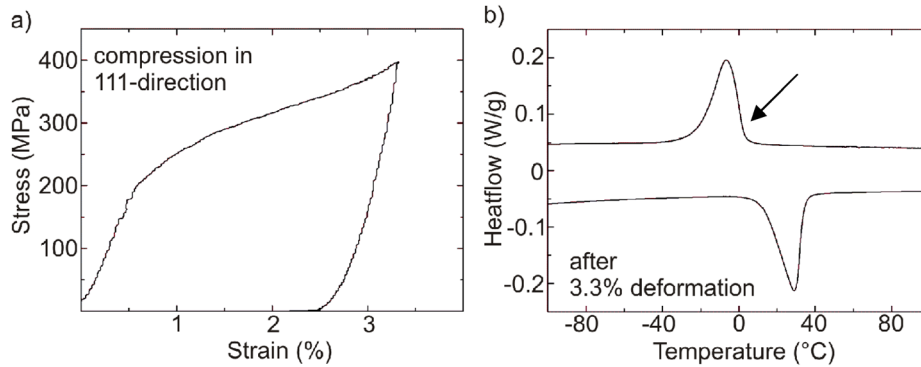


Figure 1. $\langle 111 \rangle$ compression testing and thermal analysis. (a) Stress-strain curve ($\varepsilon_{\max}=3.3\%$). (b) DSC chart.

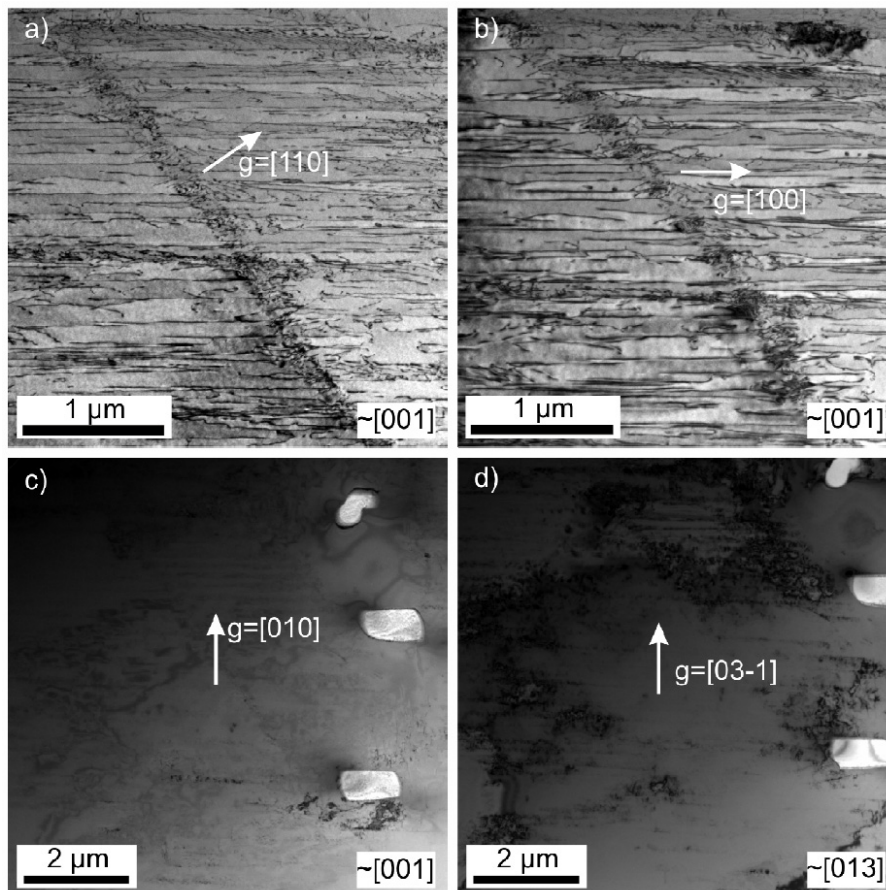


Figure 2. TEM bright field images taken in two beam contrast. (a) $g=[110]$, close to $[001]$ pole, (b) $g=[100]$, close to $[001]$ pole, (c) $g=[010]$, close to $[001]$ pole, (d) $g=[03-1]$, close to $[013]$ pole TEM-brightfield images with different g -vectors for the dislocation analysis. The arrow in each image mark the direction of the g .