Synchrotron X-ray Study of Texture in Cold-Worked Shape-Memory NiTi Wires

A. Schuster,[1] H. Voggenreiter,[1] D. K. Balch,[2] D. C. Dunand[2]

[1] *EADS, Corporate Research Center, Munich, Germany (formerly DaimlerChrysler Research and Technology)*

[2] *Dept. of Materials Science and Engineering, Northwestern University, Evanston IL, U.S.A.*

Introduction

A series of martensitic, near-equiatomic NiTi shape-memory alloy wires was deformed to strains ranging from 1 to 40% up to stresses of 920 MPa. The stress-strain curve showed the expected shape for NiTi alloys. After deformation, the wires were exposed to a monochromatic, parallel beam of high-energy x-rays oriented perpendicular to the wire axis at room temperature. The transmitted low-index diffraction rings show that martensitic texture is increasing with prestrain up to ε=15%. Unexpectedly, further deformation in the plastic range lowers the texture, suggesting that twinning texture accumulated in the first 15% of prestrain is partially cancelled by slip texture produced upon subsequent plastic deformation.

Some of the best-studied shape-memory alloys have near-equiatomic NiTi composition. The shape-memory effect (SME) relies on a reversible, diffusionless, thermoelastic transformation from a high-temperature austenite phase to a low-temperature martensite phase.[1] When the martensite is mechanically loaded, these martensitic variants can reorient in a twinning process, leading up to about 6% macroscopic strain. After unloading, the twinned structure is stable so no deformation is recovered, except for a small elastic component. It is interesting to study the phase and texture evolution in the bulk of these materials as a function of deformation. This can be achieved by synchrotron x-ray or neutron diffraction, as recently studied in superelastic and shape-memory NiTi deformed *in situ*.[2,3]

These results will be presented at the MRS Spring Meeting 2001 in San Francisco and published in the proceedings.

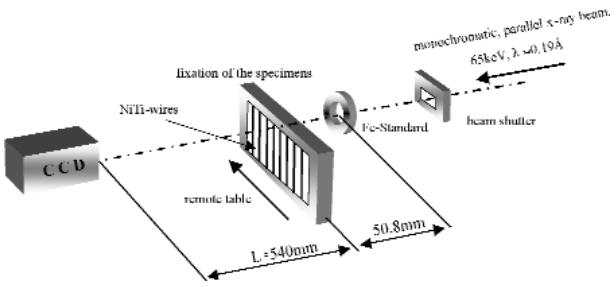

FIG. 1. Sketch of the experimental setup for x-ray diffraction experiments.

Methods and Materials

The NiTi wires used were near-equiatomic (Ni-49.7 at% Ti) in composition and exhibited a diameter of 0.8 mm. The wires were procured from Memory-Metalle GmbH (Weil am Rhein, Germany) in the "straight anneal" condition (600°C treatment) and were martensitic at room temperature.

Deformed, uncycled, NiTi wires were irradiated for 5 min. with a monochromatic, parallel beam of 65 keV photons at room temperature, in a setup similar to that described in detail in Ref. 4 and shown schematically in Fig. 1.[4] The beam had approximate dimensions of 1 x 2 mm, leading to a diffracting volume of ca. 0.5 mm^3. The transmitted diffraction patterns were recorded on a plane normal to the incident beam using a charge-coupled device camera placed at a distance of 540 mm from the sample. This allowed for recordings of all rings with diffraction angle $\theta \leq 3.48°$. An iron powder standard was also inserted into the x-ray beam for error correction purpose. All recorded diffraction rings could be assigned to the martensitic phase.

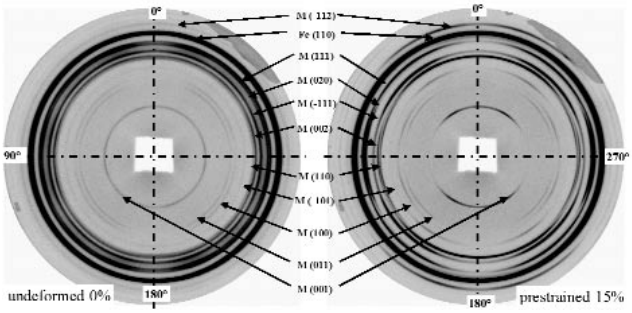

FIG. 2. Diffraction patterns of NiTi wires with 0 and 15% prestrain.

Results and Discussion

In Fig. 2, diffraction patterns are shown before wire deformation and after a prestrain of 15%. All diffraction rings are indexed with the martensitic planes, and the (110) diffraction ring of the iron powder standard is also visible. In the undeformed state, the intensities of almost all NiTi diffraction rings are constant along the ring perimeters, indicating that the annealing procedure after wire drawing removed any texture. A prestrain of 15% leads to a varying intensity along the perimeter of the diffraction rings, indicative of preferred orientation. For example, two intensity maxima are seen for planes (001) and (020), which have a multiplicity factor of M=2.

In Fig. 3, the diffracted intensity is shown as a function of d-spacing from a horizontal radial scan in Fig. 2 from the center of the detector to its outer edge. The peaks, are produced by lattice planes parallel to the wire axis. To allow direct comparison, the spectra are intensity normalized and volume corrected for change in wire diameter. The three most intense peaks (111), (020) and (-111) have about the same intensity in the undeformed state. After a prestrain of 7%, the (111) and (020) plane intensities have increased markedly, indicating that twinning produces a strong texture, as expected. A further increase in (111) and (020) intensity is visible after a prestrain of 15%, coupled with a decrease in the (-111) plane intensity. This demonstrates that twinning is occurring in the second linear elastic region, a somewhat unexpected result, since this region is usually associated with elastic deformation of the twinned martensite. Finally, for the heavily cold-worked wire subjected to a prestrain of 40%, the intensities of the (111), (020) and (-111) planes are lowered and are closer to those of the undeformed state. This result suggests that the high texture on these planes produced by twinning is decreased upon

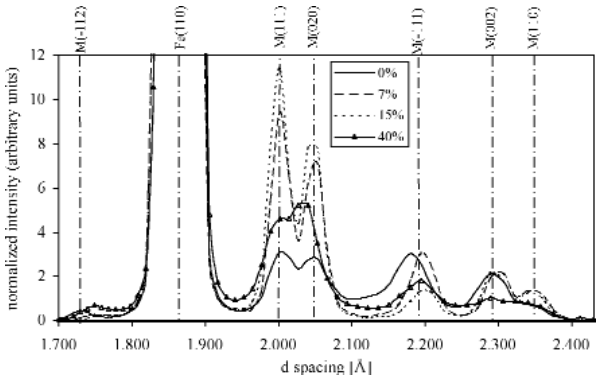

FIG. 3. Diffracted normalized intensities as a function of d-spacing from a horizontal radial scan in Fig. 2 from the center to the outer ring (corresponding to lattice planes parallel to the wire axis) for wires with different levels of prestrain. Normalized, volume-corrected intensities of the martensitic planes were plotted as a function of the azimuth angle in Fig. 2 (from 0° to 360°). The intensity fluctuations of these different planes were also analyzed.

plastic deformation. Thus, the texture induced by twinning (up to a prestrain of 15%) is partially cancelled by the texture produced by slip (from 15 to 40%). This new result illustrates that two independent mechanisms (twinning and slip) produce different types of texture, which can partially neutralize each other.

Acknowledgments

The authors acknowledge useful discussions with Prof. G. Eggeler (Ruhr-Universität Bochum, Germany) and Dr. U. Klemradt (Ludwig-Maximillian-Universität, Munich). The diffraction experiments were performed at the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) Synchrotron Research Center located at the Advanced Photon Source (Argonne National Laboratory, IL), whose staff we acknowledge for their technical support. DND-CAT is supported by the E.I. DuPont de Nemours & Co., The Dow Chemical Company, the US National Science Foundation through Grant No. DMR-9304725, and the State of Illinois through Grant No. IBHE HECA NWU 96. Use of the Advanced Photon Source was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.

References

[1] K. Otsuka and C.M. Wayman, *Shape Memory Materials* (Cambridge Univ. Press, 1999) p. 3.

[2] D.C. Dunand, D. Mari, M.A.M. Bourke, and J.A. Roberts, Metall. Mater. Trans. A **27**, 2820-2836 (1996).

[3] R. Vaidyanathan, M.A.M. Bourke, and D.C. Dunand, J. Appl. Phys. A **86**, 3020-3029 (1999).

[4] A. Wanner and D.C. Dunand, Metall. Mater. Trans. A **31**, 2949-2962 (2000).