CONF-870862--2

DE87 008776

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

A TEM STUDY OF AMORPHIZATION IN NiTi IRRADIATED WITH Ni^{2+} IONS AT ROOM TEMPERATURE

P. J. Maziasz, N. H. Packan, D. F. Pedraza, and E. H. Lee

Oak Ridge National Laboratory, P.O. Box X, Oak Ridge, TN 37831-6376

Irradiation of a crystalline solid solution can cause the decomposition of the matrix into enhanced, modified thermal, or irradiation-induced precipitate phases. Irradiation can also transform the matrix into a new crystalline phase. Alternatively, the heavily irradiated matrix can become amorphous. Alloys of NiTi are intermetallic compounds with shape-memory applications whose amorphization is of both technological and basic scientific interest. Our purpose is to observe some details of the intermediate stages of amorphization to provide further insight into the mechanisms of the phenomenon.[1]

An alloy of 50.5 at.% Ni/49.5% Ti was obtained from Raychem Co. as 0.76-mm-thick sheet and then rolled to 0.5 mm sheet. Disks 3 mm in diameter were punched and annealed for 0.5 h at 850°C. This results in an ordered austenitic (B2) structure, which partially transforms to martensite after air quenching. Disks in this condition were then irradiated with 3 MeV Ni^{2+} ions at room temperature (26–28°C) to peak damage levels calculated to be 0.01 to 0.35 dpa. Samples were then back-thinned to the peak damage region and observed in either JEOL 100CX or Philips EM400T/FEG (100 to 120 kV) and EM430T (300 kV) analytical electron microscopes (AEMs).

The peak damage region, at a section depth of about 600 nm beneath the bombarded surface, was observed to be partially amorphized (about 50%) after doses of 0.01 to 0.02 dpa, and to be almost completely amorphized (90% or more) after 0.05 dpa. Figure 1(a,b) shows the complicated martensite microstructure and diffraction pattern of the unirradiated starting material. Figure 1(c,d) shows the partially amorphized structure after irradiation to 0.018 dpa. Figure 1(c,d) shows the crystalline portions of the irradiated sample appear to be highly damaged, but the nature of the damage has not yet been determined. The regions with only a few defects are mostly amorphous. This can be seen in the dark-field series [Fig. 1(c–h)] in which these regions are dark for images with the matrix reflection, but show uniform bright contrast in images made from two different portions of the diffuse amorphous ring. Small crystallites in some portions of the microstructure appear to be causing the weak spots seen in the ring. Several details are immediately apparent. The amorphous regions appear to have faceted shapes with fairly sharp interfaces separating them from the surrounding crystalline regions. This can be seen both at high magnification [Fig. 1(c)] and in a dark-field (DF) series at lower magnification [Fig. 1(e–h)]. This observation is in contrast to the formation of irregular amorphous regions without any definite shape reported by others for in situ irradiations.[2,3] The diffraction pattern of the irradiated material shows much less evidence for martensite relative to the starting structure [Fig. 1(d)]. This suggests that much of the martensite converts to austenite prior to amorphization.[4] Similar behavior is noted during in situ irradiation with high energy electrons,[3] but

Irradiation of a crystalline solid solution can cause the decomposition of the matrix into enhanced, modified thermal, or irradiation-induced precipitate phases. Irradiation can also transform the matrix into a new crystalline phase. Alternatively, the heavily irradiated matrix can become amorphous. Alloys of NiTi are intermetallic compounds with shape-memory applications whose amorphization is of both technological and basic scientific interest. Our purpose is to observe some details of the intermediate stages of amorphization to provide further insight into the mechanisms of the phenomenon.[1]

An alloy of 50.5 at.% Ni/49.5% Ti was obtained from Raychem Co. as 0.76-mm-thick sheet and then rolled to 0.5 mm sheet. Disks 3 mm in diameter were punched and annealed for 0.5 h at 850°C. This results in an ordered austenitic (B2) structure, which partially transforms to martensite after air quenching. Disks in this condition were then irradiated with 3 MeV Ni^{2+} ions at room temperature (26–28°C) to peak damage levels calculated to be 0.01 to 0.35 dpa. Samples were then back-thinned to the peak damage region and observed in either JEOL 100CX or Philips EM400T/FEG (100 to 120 kV) and EM430T (300 kV) analytical electron microscopes (AEMs).

The peak damage region, at a section depth of about 600 nm beneath the bombarded surface, was observed to be partially amorphized (about 50%) after doses of 0.01 to 0.02 dpa, and to be almost completely amorphized (90% or more) after 0.05 dpa. Figure 1(a,b) shows the complicated martensite microstructure and diffraction pattern of the unirradiated starting material. Figure 1(c,d) shows the partially amorphized structure after irradiation to 0.018 dpa. Figure 1(c,d) shows the crystalline portions of the irradiated sample appear to be highly damaged, but the nature of the damage has not yet been determined. The regions with only a few defects are mostly amorphous. This can be seen in the dark-field series [Fig. 1(c–h)] in which these regions are dark for images with the matrix reflection, but show uniform bright contrast in images made from two different portions of the diffuse amorphous ring. Small crystallites in some portions of the microstructure appear to be causing the weak spots seen in the ring. Several details are immediately apparent. The amorphous regions appear to have faceted shapes with fairly sharp interfaces separating them from the surrounding crystalline regions. This can be seen both at high magnification [Fig. 1(c)] and in a dark-field (DF) series at lower magnification [Fig. 1(e–h)]. This observation is in contrast to the formation of irregular amorphous regions without any definite shape reported by others for in situ irradiations.[2,3] The diffraction pattern of the irradiated material shows much less evidence for martensite relative to the starting structure [Fig. 1(d)]. This suggests that much of the martensite converts to austenite prior to amorphization.[4] Similar behavior is noted during in situ irradiation with high energy electrons,[3] but not during in situ heavy-ion irradiation.[2,3]

References

1. D. F. Pedraza and L. K. Mansur, Nucl. Instr. & Meth. Physics Research, (1986) B16, 203.
2. J. L. Brimhall, H. E. Kissinger and A. R. Pelton, Radiation Effects (1985) 90, 241.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

"The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. DE-AC05-84OR21400. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government

3. P. Moine et al., Nucl. Instr. Meth. Phys. Research, (1985) B7/8, 20.
4. Research sponsored by the Division of Materials Sciences, U.S. Department of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

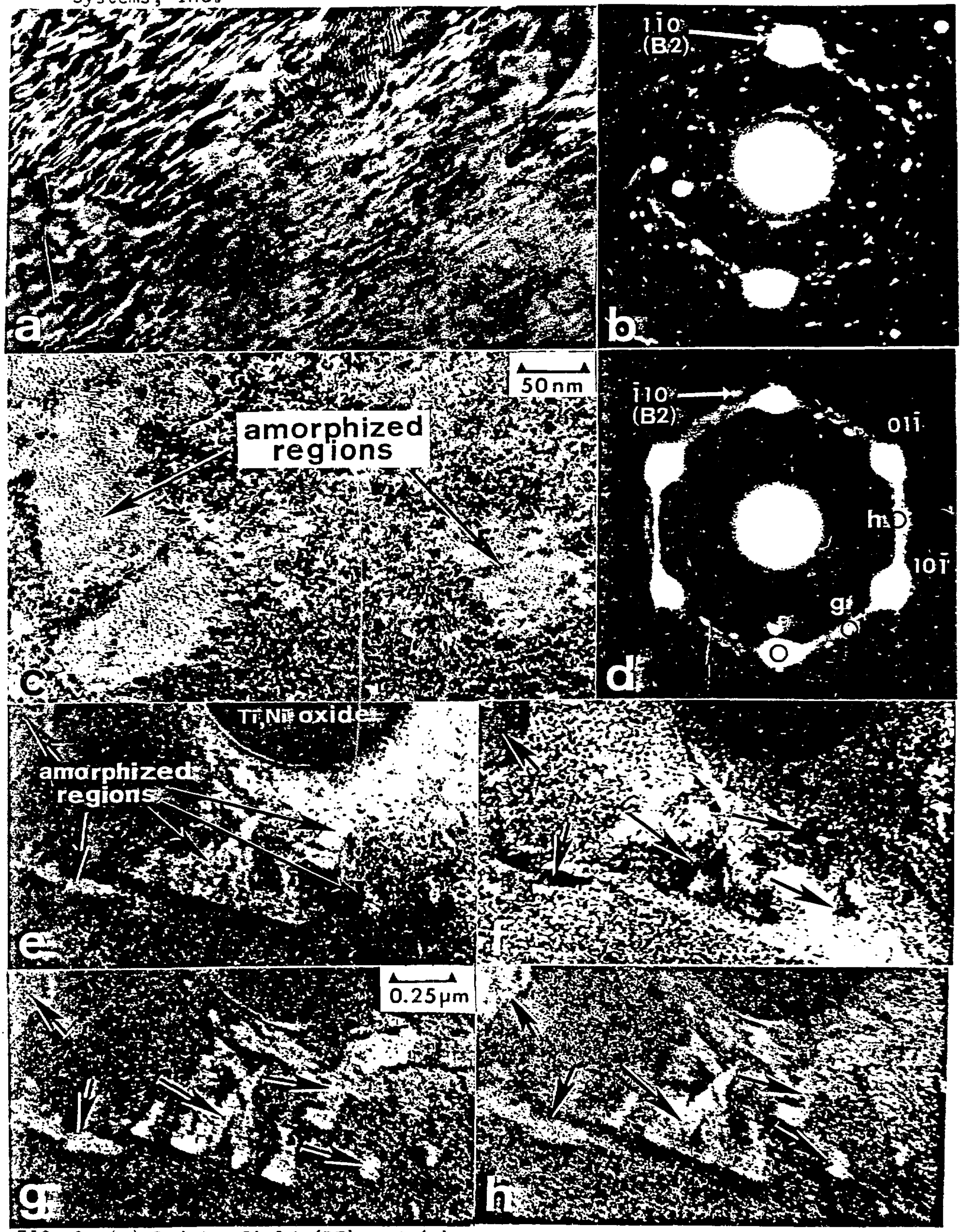

FIG. 1--(a) Bright-field (BF) and (b) selected area diffraction (SAD), showing martensite and austenite of unirradiated NiTi. (c) BF near Z = [111] showing damaged crystalline and partially amorphized regions after irradiation to 0.018 dpa, (d) SAD from same area. (e) BF, and (f), (g) and (h) dark-field (DF) images from same region at lower magnification. Aperture placement for (f)—(g) indicated in (d).