MOTION PICTURE X-RAY DIFFRACTION INVESTIGATION OF THE MARTENSITIC TRANSFORMATION IN NICKEL–TITANIUM ALLOY

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The thermoelastic martensitic transformation of an equiatomic nickel–titanium alloy is investigated by means of x-ray diffraction motion pictures using synchrotron radiation. The x-ray patterns are recorded by a parallel detector simultaneously in the range of angles $2\theta = 34^\circ - 51^\circ$ at a wavelength of $1.4879 \, \text{Å}$ during heating and cooling with good temperature resolution. The basic parameters of the attendant B2, R, and B19' phases are determined simultaneously and compared. The variations of the parameters of the martensite B19' unit cell are traced, along with the evolution of the R phase {$202\overline{2}02\overline{2}$} reflection doublet during transformation. Volumetric transformation phenomena are determined, and it is shown that martensite originates in a more unconsolidated structure than in the equilibrium state.

A great many papers have been devoted to x-ray diffraction studies of the martensitic transformation in nickel–titanium alloy. The diffraction pattern has been obtained either at a fixed temperature over a rather long time interval (30 min or more) or by means of a diffractometer with time-sequential recording of the individual reflection orders. First of all, this approach can induce additional error due to instability of the diffraction pattern as time passes and, second, recording in the static regime sacrifices information on the phase transformation kinetics.

On the other hand, there is the alternative of x-ray diffraction motion pictures using synchrotron radiation [1, 2]. The method has distinctive features and advantages in its application of a parallel multichannel detector to record the diffraction pattern, the high power of the incident radiation, a high level of automation in recording and processing the x-ray patterns, the possibility of choosing radiation wavelengths anywhere from several angstroms down to tenths of an angstrom, the fact that the radiation does not form doublets, and a high degree of monochromaticity and plane polarization of the radiation. In particular, these features enable one to obtain x-ray diffraction patterns of structural changes lasting fractions of a second in the material, where the profiles of the x-ray reflections are recorded simultaneously over a wide range of angles.

It is propitious in this connection to apply motion picture x-ray diffraction to the study of the martensitic transformation in NiTi, as it provides a means for investigating the kinetics of the transformations observed in NiTi by recording the parameters of all the attendant phases simultaneously.

The objective of the present study is to investigate simultaneously the behavior of the main structural austenite and martensite parameters with high temperature resolution directly in the thermoelastic martensitic transformation.

We used NiTi alloy with a nearly equiatomic composition. The characteristic temperatures of minimum and maximum electrical resistivity in the vicinity of the direct martensitic transformation were determined by thermal resistometry (Fig. 2a): $T_{\rho_{\text{min}}} = 90^\circ \text{C}$ and $T_{\rho_{\text{max}}} = 60^\circ \text{C}$. According to [3-6], these results indicate an alloy composition of 50.1 at. % Ti.

The final heat treatment consisted of vacuum annealing at a temperature of 800°C for one hour and cooling while left in the furnace. A surface layer of material was then removed in an aqueous solution of HF and HNO3. The sample was placed in a cell, which was equipped with a heater and attached to a goniometer. The diffraction patterns were recorded directly during the heating or cooling process after two test cycles. The average heating rate was 6°C/min, and the average cooling rate was 5°C/min. The temperature was recorded by a chromel–alumel thermocouple, and the working temperature range was

Fig. 1. Series of diffraction patterns of equiatomic NiTi. 1) In the heating cycle; 2) in cooling.

Fig. 2. Electrical resistivity (a), total intensities of x-ray reflections \{110\}_B (b), \{202\}_R (c), and \{202\}_R, half-width of the \{110\}_B reflection (e), and interplanar distances of \{110\}_B (f), \{202\}_R (g), and \{202\}_R (h) vs temperature for NiTi. 1) In the heating cycle; 2) in cooling.

20–200°C. The diffraction pattern was recorded by a single-coordinate parallel detector simultaneously in the angular range \(2\theta = 34–51^\circ\) during the exposure time of one x-ray frame (30 sec). The angular (2\(\theta\)) resolution of the detector was 0.008\(^\circ\). The goniometer was calibrated against a standard with the sample at room temperature to determine the absolute values of the angle 2\(\theta\). The x-ray wavelength was 1.4879 Å.

The experimental x-ray patterns were approximated by analytical profiles minimizing the standard deviation (which was less than 0.5%). The minimum deviation yielded an approximating profile of the form \(1/(1 + x^2)^2\) in the presence of several different reflections and of the form \(\exp(-x^2)\) in the case of undistorted austenite.

The parameters of the unit cell of monoclinic martensite B19' were calculated from five or six reflections within the investigate angular range by minimization of the functional

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\sum_{i=1}^{n} (\sin^2 \theta_i^e - \sin^2 \theta_i^f),
\]

where \(n\) is the number of indexed reflections of the given structure, and \(\theta_i^e\) and \(\theta_i^f\) are the diffraction angles for the i-th reflection, determined experimentally and calculated from the postulated unit cell parameters.