ISSN 1062-8738, Bulletin of the Russian Academy of Sciences. Physics, 2014, Vol. 78, No. 12, pp. 1379–1381.

Solid State Transformations in Melt-Spun Ti_2NiCu Ribbon

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Abstract—Structural transformations in melt-spun Ti_2NiCu ribbon are studied by means of X-ray diffraction. The phase composition of a ribbon during crystallization and martensitic transformations is determined qualitatively and quantitatively. Temperatures of crystallization and the onset and finish of forward and reverse martensitic transformations are established.

DOI: 10.3103/S1062873814120351

INTRODUCTION

Alloys with thermomechanical memory effects can generally be recognized due to their unique functional properties, which include record high reversible strains under the action of temperature variation. In a number of recent studies, the martensitic transformations and strain effects in thin titanium nickelide-based alloy ribbons and films fully crystallized from the amorphous state were investigated [1], relaxation and crystallization activation energies were determined [2], and the effect of crystal grain size on the temperatures and sequence of martensitic transformations was established [3]. In addition, shape memory and reversible shape memory effects were observed in fully crystallized thin films [4, 5] and the generation of recovery stresses was established [5]; this is of primary importance in applying these materials in microsystem technology.

TiNi alloy with near-equiatomic composition contains a *B*2 austenite phase with a body-centered cubic (bcc) lattice ordered in CsCl form. Upon cooling below the martensitic transition temperature or under loading, the *B*2 phase is transformed into B19 martensite with a monoclinic lattice that ensures thermoelastic shape memory effects. Ti_2NiCu alloys have an analogous austenite phase with the *B*2 lattice in which copper atoms are located in a subblatice of nickel atoms. When the alloy is cooled below 75°C, the *B*2 phase is transformed into the orthorhombic *B*19 martensite phase [6].

The aim of this work was to investigate the phase transformations in melt-spun Ti_2NiCu ribbon samples by means of X-ray diffraction, and to determine the temperatures of crystallization and the onset and finish of forward and reverse martensitic transformations.

EXPERIMENTAL

Our object of study was a 30-μm-thick and 1.5-mm-wide Ti_2NiCu ribbon melt-spun on a drum. The ribbon was grey with one side lustrous and the other side dull. The alloy cooling rate was 10^6 °C s^{-1}. The fresh ribbon was amorphous, but with the possible formation of texture on the free surface along the normal to the drum surface due to the temperature gradient.

Sample crystallization and phase transformations were studied via X-ray diffraction on a Bruker D8 Advance unit equipped with a heating chamber in the θ–2θ geometry and the angle range $2\theta = 35°–95°$. Diffraction patterns were obtained with a pitch of 2° upon heating and cooling from room temperature to 520°C during crystallization, and from room temperature to 70°C and back during forward and reverse martensitic transformations. The ratio between the austenite and martensite phases was determined from those of the characteristic peaks in the diffraction patterns. Both sides of the ribbon were studied.

RESULTS AND DISCUSSION

The diffraction pattern from the dull side of the melt-spun ribbon at room temperature prior to annealing (line *1* in Fig. 1) displays a widely spread low-intensity peak near 42°, demonstrating the amorphous state of this side. The diffraction pattern from the lustrous side contains a set of peaks with the maximum intensity near 61° and peaks of lower intensity near 57°, 66°, and 68° (line *2* in Fig. 1). After annealing, both sides yielded sets of peaks characteristic of the *B*2 phase with the maximum intensity near diffraction angles of 41.88°, 60.73°, and 76.51° (line *3* in Fig. 1).

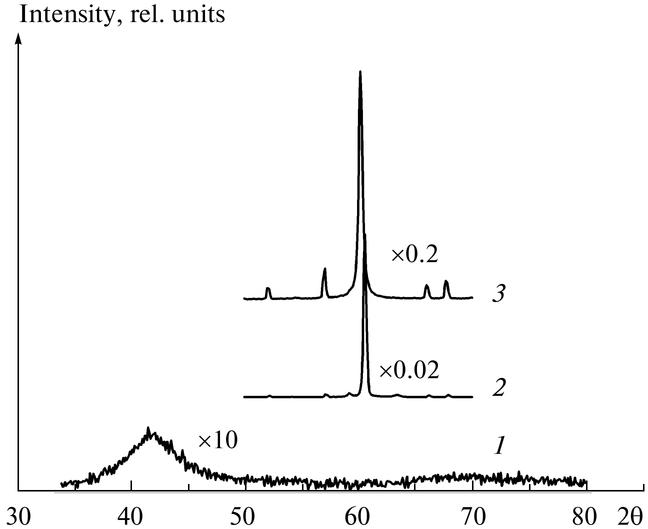

Fig. 1. Diffraction patterns of melt-spun Ti_2NiCu ribbon, obtained (*1*) from the side adjacent to the drum and (*2*) from the opposite side before annealing and (*3*) at a temperature of 520°C.

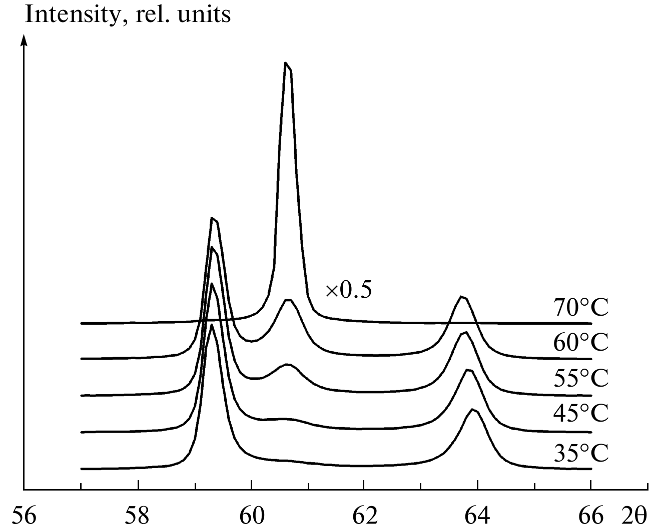

Fig. 2. Diffraction patterns of melt-spun Ti_2NiCu ribbon, obtained upon heating from 25 to 70°C.

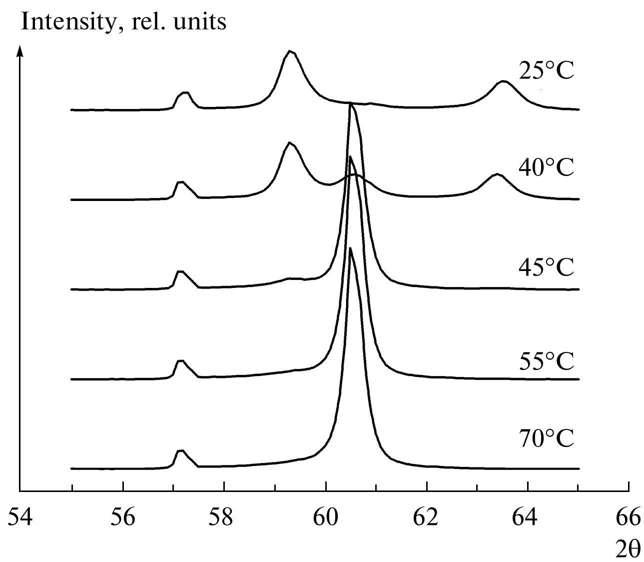

Fig. 3. Diffraction patterns of melt-spun Ti_2NiCu ribbon, obtained upon cooling from 70 to 25°C.

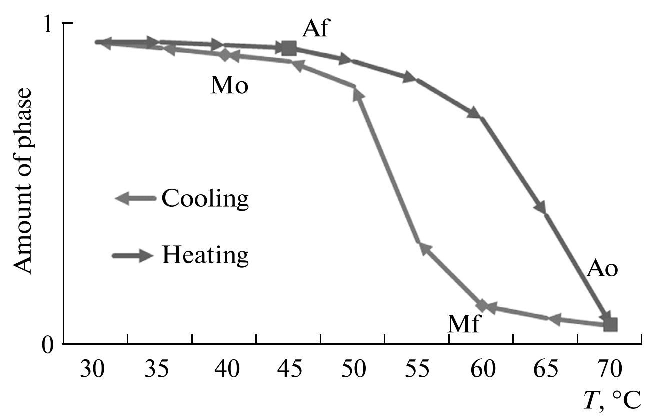

Fig. 4. Temperature dependence of the phase composition of melt-spun Ti_2NiCu ribbon upon heating and cooling.

Upon cooling from 70 to 25°C (Fig. 2), two peaks corresponding to the *B*2 phase were observed at $T =$ 70°C. The peak corresponding to the martensite phase started forming at a temperature of 45°C. At 25°C, the peak corresponding to the *B*2 phase nearly vanished, while the peak corresponding to the B19 phase grew in intensity.

Upon heating from 30 to 70°C (Fig. 3), two peaks corresponding to the B19 phase were observed at $T =$ 30°C. At a temperature of 50°C, the austenite phase started nucleating and a small peak arose near 60.5°. At 70°C, this peak attained its maximum and the other two peaks vanished.

The amounts of the martensite and austenite phases for each heating and cooling temperature were determined from the ratio of intensities of the main peaks. The change in the number of phases upon heating and cooling (Fig. 4) had the pronounced hysteresis typical of materials with martensitic transformation. Analysis of this dependence allows us to establish the characteristic temperatures of the phase transformation: $T_{Mo} = 60°C$, $T_{Mf} = 40°C$, $T_{Ao} = 45°C$, and $T_{Af} = 70°C$.

CONCLUSIONS

Using X-ray diffraction analysis, we studied the structure of melt-spun Ti_2NiCu ribbon. It was found to have different surface structures after melt spinning: the side adjacent to the drum was amorphous while the opposite side was crystalline. After annealing at a temperature of 520°C, both sides had the same crystal structure.

The phase composition of the annealed ribbon changed in the temperature range of 40 to 50°C; the temperatures of the onset and finish of martensitic transformation upon heating did not coincide with the corresponding temperatures upon cooling. The forward martensitic transition started at $T_{Mo} = 60°C$ and finished at $T_{Me} = 40°C$. The reverse transformation started at $T_{Ao} = 45°C$ and finishes at $T_{Af} = 70°C$.

ACKNOWLEDGMENTS

This study was supported by the RF Ministry of Education and Science. It was conducted at the Materials Science and Metallurgy Shared Facilities Center of the Moscow Institute of Steel and Alloys, National University of Science and Technology.

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Translated by E. Bondareva