Effect of deformation on martensitic transformation behavior in $Ni_{50.1}Ti_{46.9}Nb_3$ shape memory alloy

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Recently, considerable interest has been generated in the Ni-Ti-Nb ternary alloy because of its wide transformation hysteresis [1, 2]. Parts made of this alloy can be stored and transported at ambient temperature, which is convenient for commercial applications. However, as the melting point of niobium is much higher than that of titanium and nickel, compositional deviation often occurs unavoidably even when considerable care is taken during melting. As a result, lowering the niobium content in Ni-Ti-Nb alloys is an effective way to reduce segregation. Besides, Ni-Ti-Nb alloy with low niobium content is more economic. We found that $Ni_{50.1}Ti_{46.9}Nb_3$ (at%) alloy also has an excellent shape memory effect, and a sufficiently wide transformation temperature hysteresis can be attained after deformation at (M_s +30 °C) [3]. The purpose of this work was to investigate deformation dependence of the reverse martensitic transformation behavior in $Ni_{50.1}Ti_{46.9}Nb_3$ alloy.

The nominal composition of the experimental alloy was 50.1 at% Ni, 46.9 at% Ti and 3 at% Nb. The alloy was prepared by vacuum induction melting in a calcium oxide crucible. The ingots with a weight of ten kilograms were hot swaged and rolled to rods with a diameter of 8.5 mm, and tensile samples 4 mm in diameter were machined from the rods. The samples were solution treated at 860 °C for 2.4 ks, followed by water quenching. X-ray diffraction experiments were carried out on a Rigaku D/max–2500pc diffractometer using Cu Kα radiation at 50 kV and 200 mA to identify the phases presented in the sample. Differential scanning calorimetry (DSC) tests were performed on a PerkinElmer Pyris Diamond DSC. The martensitic start temperature M_s, the martensitic finish temperature M_f, the austenitic start temperature A_s and the austenitic finish temperature A_f were −62, −117, −48 and 18 °C, respectively. Tensile tests were carried out on a Schimadzu Autograph DCS-10T type machine at a strain rate of 2.8×10^{-4} s^{-1} and a temperature of (M_s +30 °C). The DSC samples after deformation were carefully cut using a low speed diamond saw cooled by water to avoid any undesired phase transformation prior to the measurement.

Fig. 1 shows the DSC curves during heating of the samples after deformation to different strain levels. Prior to the measurement the DSC sample was cooled to −150 °C so that the residual austenite would transform to martensite thermally, then heated from −80 °C to 100 °C at a heating rate of 10 °C/min. As the total strain was less than 6%, the curves only show the reverse transformation of thermal-induced martensite. When the strain is in the range of 6 to 16%, the DSC curves exhibit two transformation peaks: the peak at lower temperature is due to the reverse transformation of thermal-induced martensite and the one at higher temperature corresponds to the reverse transformation of stress-induced martensite. With increasing deformation, the transformation peaks of stress-induced martensite become clear, and the transformation proceeds over a wider temperature range while those of thermal-induced martensite become diffuse. When the total strain is above 16%, the DSC curves only show the reverse transformation peak of stress-induced martensite. It means that the process of stress-induced martensitic transformation had been completed as the sample was deformed in tension to above 16% strain.

Fig. 2 shows the plots of $A_s{}'$ and $A_f{}'$ as a function of deformation, where $A_s{}'$and $A_f{}'$ denote the starting and finishing temperature for the reverse martensitic transformation of stress-induced martensite, respectively. We can see that with an increase of strain there is no obvious change of the temperature $A_s{}'$, and $A_s{}'$ has risen to about 37 ° after deformation. Accordingly, the transformation temperature hysteresis $|A_s{}'-M_s|$ follows the same trend as $A_s{}'$ and could reach about 100 °C after deformation, which implies that parts made of this alloy can be stored, transported and installed at ambient temperature. However, $A_f{}'$ increased progressively with increasing strain. This is in contradiction to the results in previous reports on $Ni_{47}Ti_{44}Nb_9$ alloy [4, 5]. In these papers [4, 5] these characteristic temperatures increase almost linearly with increasing strain. The algebraic differences between $A_f{}'$and $A_s{}'$are also plotted in Fig. 2. The transformation temperature interval is only 3.7 °C when the total strain is 6%, but it increases dramatically to 68.8 °C when the deformation increases to 24%.

The rise of the reverse transformation temperature in the deformed specimens in Fig. 2 is an indication of a stabilization effect on the stress-induced martensite. It was previously found that the microstructure of $Ni_{47}Ti_{44}Nb_9$ alloy consists of the NiTi matrix and β-Nb phase [6]. The deformation of the NiTi matrix and β-Nb phase would both relax the elastic stress field of

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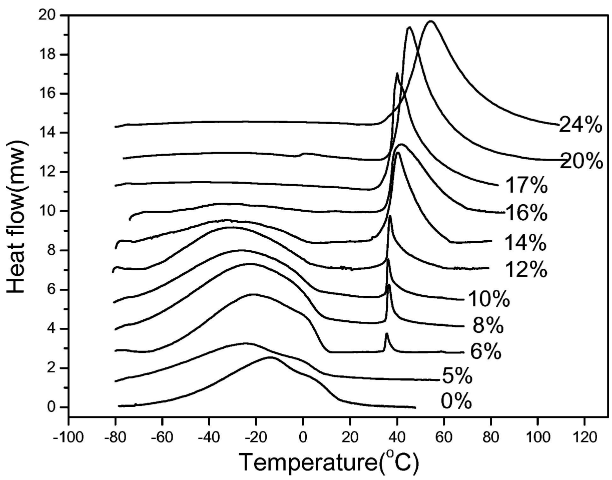

Figure 1 The effects of the deformations on the reverse transformation behavior of $Ni_{50.1}Ti_{46.9}Nb_3$ alloy during the heating.

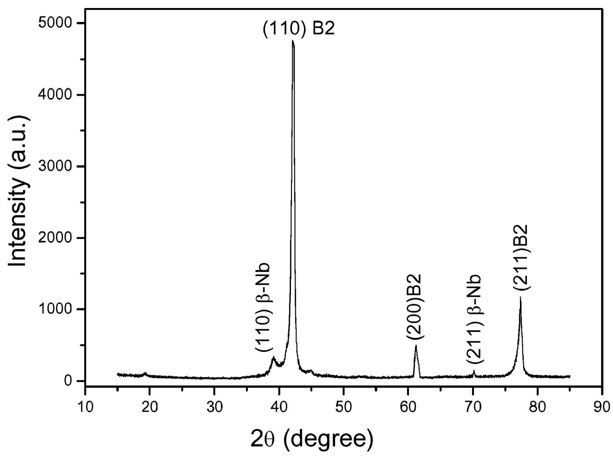

Figure 3 XRD pattern and its identification of $Ni_{50.1}Ti_{46.9}Nb_3$ alloys at room temperature.

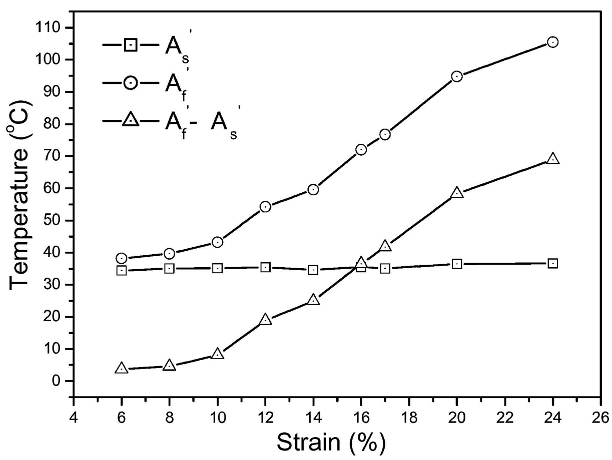

Figure 2 Effect of deformation on reverse transformation temperatures of stress-induced martensite.

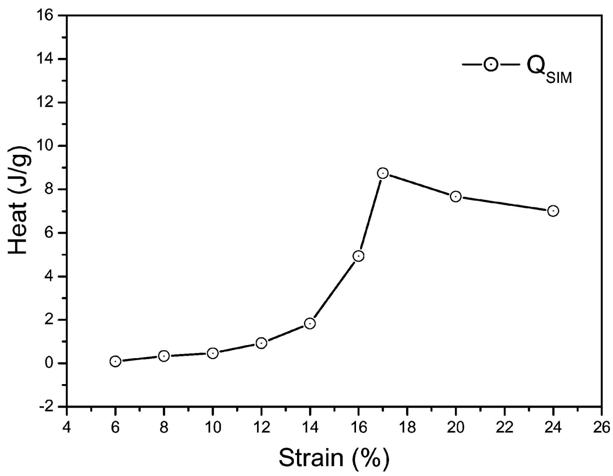

Figure 4 Effect of deformation on the heat effect of the reverse transformation of stress-induced martensite.

the front-end of the stress-induced martensite. The loss of the stored elastic strain energy, which is the driving force for the reverse transformation results in the martensite stabilisation. For $Ni_{50.1}Ti_{46.9}Nb_3$ alloy, the dominant phase is the NiTi matrix phase, and β-Nb particles are difficult to find in scanning electron microscope observations. However, the X-ray diffraction experiment shows there is a β-Nb reflection peak in this alloy, as shown in Fig. 3, which implies that the amount of β-Nb is small. A little amount of the β-Nb particle only appears at the front-end of the stress-induced martensite during deformation, and its plastic deformation would relax the elastic stress field of front-end of the stress-induced martensite. The small quantity of β-Nb particles may have all experienced plastic deformation as the specimen deforms above 6%, so A_s'reached 37 °C. With further increasing of strain, more plastic deformation will be mainly occurred in NiTi matrix phase. It has been found that the plastic deformation of the NiTi matrix phase has a little effect on the widening transformation temperature hysteresis [3]. As a result, A_s'does not obviously increase with increasing strain in this low niobium content alloy. However, the deformed NiTi matrix phase adjacent to the stress-induced martensite will greatly increase the resistance of the thermal elastic martensite freely shrinkage upon heating, so the reverse transformation temperature interval of $Ni_{50.1}Ti_{46.9}Nb_3$ alloy was widened.

The transformation heat of reverse martensitic transformation of stress-induced martensite Q_{SIM} *vs.* deformation is shown in Fig. 4. It can be seen that Q_{SIM} increases slightly with increasing strain at low strain level and increases dramatically when the deformation increases to 12%, then it starts to decrease slightly when the deformation exceeds 17%. With increasing strain, the heat flow intensity of reverse transformation of the stress-induced martensite increased progressively at the expense of that of thermal-induced martensite. However, a drop in transformation heat was observed as the deformation exceeded 17%, as shown in Fig. 4. This agrees with the results in literature [8], and the reason why a large deformation can reduce the heat is explained in previous reports [7, 8].

In conclusion, the DSC measurements suggest that the process of stress-induced martensitic transformation of $Ni_{50.1}Ti_{46.9}Nb_3$ alloy had been completed as the specimen was deformed in tension to above 16% strain. A_s' and transformation temperature hysteresis show no obvious change with increasing strain. The transformation heat effect associated with reversion of stress-induced martensite was found to increase with increasing strain until it reached a maximum, and then decreased slightly with further deformation.

References

1. K. N. MELTON, J. SIMPSON and T. W. DUERIG, in Proceedings of the Conference on Martensitic Transformations (The Japan Institute of Metals, Sendai Japan, August, 1986) p. 1053.
2. K. N. MELTON, J. L. PROFT and T. W. DUERIG, in Proceedings of the MRS International Meeting on Advanced Materials (MRS, Tokyo, 1989) p. 165.
3. X. M. HE, L. J. RONG, D. S. YAN and Y. Y. LI, *Mater. Eng. A* **371** (2004) 193.
4. C.S. ZHANG, W. CAI and L. C. ZHAO, *Acta Metallurgica Sinica (English edition)* **4A** (1991) 436.
5. M. PIAO, S. MIYAZAKI and K. OTSUKA, *Mater. Trans., JIM* **33** (1992) 346.
6. C. S. ZHANG, Y. Q. WANG, W. CAI and L. C. ZHAO, *Mater. Chem. Phys.* **28** (1991) 43.
7. H. C. LIN, S. K. WU, T. S. CHOU and H. P. KAO, *Acta Metall Mater* **39** (1991) 2069.
8. H. C. LIN, S. K. WU and J. C. LIN, in Proceedings of the Conference on Martensitic Transformations. Monterey (Monterey Institute for Advanced Studies, Monterey, 1992) p. 953.

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