

Characteristics of Transformation and Low-temperature Deformation of Ti-51.1Ni Shape Memory Alloy



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Abstract: Effects of annealing temperature on the phase transformation and the low temperature deformation characteristics in the deformed Ti-51.1Ni (at%) shape memory alloy (SMA) were investigated by differential scanning calorimetry (DSC), optical microscope (OM) and tensile test. The results show that the transformation types of Ti-51.1Ni alloy are changing from $A \rightarrow R/M \rightarrow R \rightarrow A$ to $A \rightarrow R \rightarrow M/M \rightarrow R \rightarrow A$ to $A \rightarrow R \rightarrow M/M \rightarrow A$ (A-parent phase B2, R-R phase, M-martensite phase) upon cooling/heating along with increasing annealing temperature. The R transformation temperatures and martensite temperature hysteresis decrease, while the M transformation temperature increases and the R temperature hysteresis nearly keeps at about 6.5 °C. When deformation happen at 10 °C, the 400~550 °C annealed Ti-51.1Ni SMA exhibits as the shape memory effect (SME) + superelasticity (SE), the 600~700 °C annealed SMA shows SE, and the characteristics of alloy change from SME + SE to SE. In addition, the annealing recrystallization temperature of Ti-51.1Ni SMA is 590 °C, and the 590~650 °C annealed alloy could obtain excellent capability of plastic deformation and 50.83 % values of fracture strain, so the forming processing temperature could be in the range of 590~650 °C. When the Ti-51.1Ni alloy are used for energy consumption of damper and damping device, the suitable annealing temperature could be higher than 550 °C, and for making superelastic device, the suitable annealing temperature could be below 400 °C or above 600 °C.

Key words: Ti-51.1Ni alloy; stress induced martensite; shape memory effect; superelasticity; phase transformation; deformation

For Ti-Ni shape memory alloy (SMA) well-known for its unique shape memory effect (SME) and superelastic (SE) properties^[1,2], it has been widely used for production of thermo-sensitive control device^[3-5] and superelastic damping device^[6,7]. The SME and SE are closely related to forward/inverse thermoelastic martensite transformation upon cooling/heating occurring between parent phase B2 (CsCl-type structure), R phase (rhombohedral structure) and martensite phase M (monoclinic structure) of Ti-Ni SMA, and heavily depend on the relationship between start temperature (M'_s) and complete temperature (M'_f) of M reverse transformation and test temperature (T_d)^[8,9]. When T_d is less than M'_s , the Ti-Ni SMA is totally in M status occurrence M reorientation and show SME upon deformation, though it can be restored to its original shape by heating to phase B2; when T_d is greater than M'_f , the Ti-Ni SMA is totally in B2 status occurrence

stress-induced M transformation and shows SE properties upon deformation, and obtains large recovery strain after unloading. When T_d is between M'_s and M'_f , the Ti-Ni SMA shows both properties of SME and SE upon deformation.

The change of Ni content has a significant effect on the transformation temperature, SME and SE characteristics of two component Ti-Ni SMA^[10]. With the increasing content of Ni the transformation temperature is sharply decreased. But when the Ni content is too high (greater than 50%, atom fraction), the Ni of alloy matrix is reduced due to the precipitation $TiNi_2$, $TiNi_3$ and rich Ni compound, which leads to the increasing of transformation temperature and the embrittlement of alloy matrix due to the emergence of the rich Ni compound^[11,12]. For example: when the Ni is increased from 50.5% to 51.5%, about 1.0%, the martensitic transformation temperature of about 100 °C is decreased^[13].

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However, the matrix alloy will change embrittlement due to Ni rich precipitation phase transformation, and in the deformation process the matrix alloy prone to fracture and lose effectiveness. But with the reduction of Ni (i.e. with the increase of Ti), the transformation temperature of the alloy increases largely.

The published literature shows that the transformation characteristics, SME and SE of Ti-Ni alloys are affected by the following treatments: annealing following cold working^[14], aging treatment in Ni-rich Ti-Ni alloys^[15], stress-strain cycling^[16,17], and the addition of third elements^[18-20]. Among these treatments, the aging treatment for Ni-rich Ti-Ni alloys is an effective method to improve shape memory and mechanical properties due to the formation of Ti_3Ni_4 precipitates, which act as effective obstacles such as pinning points against the movement of dislocations, with the consequence that the critical stress for slip can be improved^[21,22]. For the near-equiatomic Ti-Ni alloys, the cold working, heat treatment or a combination of both is an effective method to improve shape memory and mechanical properties. Cold working increases the strength of Ti-Ni due to the introduction of random dislocations into the material. Annealing restores SME by rearranging the dislocations^[14].

The purpose of the present study is to address the transformation and low-temperature deformation behavior in the Ni-rich Ti-51.1Ni at% Ni alloy annealed at 350~700 °C for practical applications. Therefore, this initial work focuses on transformation characteristics and SE properties such as platform-stress, fracture strain, residual strains, energy consumption and strength as a function of heat treatment and microstructure.

1 Experiment

Sponge titanium (purity>99.7%) and electrolytic nickel (purity>99.9%) were used to prepare Ti-Ni alloy ingots for castings by the high-frequency induction-vacuum-melting process. The nominal composition of the ingot is Ti-51.1 at. Pct Ni. The ingot was hot forged and hot extruded followed by cold drawing, with a repeated intermediate annealing to produce wires of 1.0 mm in diameter, with a final cold drawing to 20% reduction in cross section. Specimens with 4, 10 and 120 mm in length were cut from the wires for DSC measurement, microscopy analysis and tensile tests, respectively. All the specimens were finally solution treated at 350~700 °C for 30 min in a DRZ-4 Box-type resistance furnace. The differences of the transformation behaviors (the transformation temperature and temperature hysteresis, etc) of the Ti-51.1Ni alloy were analyzed by DSC using a Shimadzu DSC-50. The range of scanning temperature was -150~100 °C with a heating/cooling rate of 10 °C/min. The tensile test was carried out at 10 °C with a WAW electrohydraulic servo universal testing machine and a strain meter, 50 mm in gauge

length, at a crosshead speed of 2.0 mm/min. After the specimens were stressed up to 8.0% strain, the stress was unloaded to 0 MPa at the same speed. The unloading strain of 8.0% was considerably larger than the elastic limitation, and consequently suitable to distinguish super-elasticity from shape memory effect or plastic deformation by evaluating residual strain and the transformation temperatures. Properties of apparent proof stress (0.2%), tensile strength, residual strain and elongation were obtained from the stress-strain diagrams. In order to prepare specimens for optical microscopy (OM) analysis, grinding, polishing and etching were used with a solution of HF, HNO₃ & H₂O of volume ratios as 1:4:5, respectively.

2 Results and Discussion

2.1 Effect of annealing temperature on microstructures of Ti-51.1Ni alloy

Fig.1 show OM microstructures for the samples after annealing at 400, 570, 590, 600, 630 and 650 °C.

The deformed microstructures of low and intermediate temperature annealed Ti-51.1Ni alloys are fibrous. With increasing the annealing temperature (θ_a), the homogenization of fibrous microstructures is increased, which leads to the rearrangement of the dislocation networks, so the density of dislocation and lacuna stress field is decreased. With increasing the θ_a further, the fibrous microstructure evolves gradually to equiaxed grain. The sample annealed at 590 °C shows some small grains, indicating the start of recrystallization. With the annealing temperature increasing the new grains occupy more space and become coarser as can be observed from the OM images for the samples annealed at 600, 630 and 650 °C, as shown in Fig.1d, 1e and 1f.

2.2 Effect of annealing temperature on transformation characteristic of Ti-51.1Ni alloy

The DSC curves, transformation temperatures and temperature hysteresis of the Ti-51.1Ni SMA specimen annealed at 400~700 °C for 30 min, are shown in Fig.2. As Fig.2a, it can be seen that the 400, 500 and 600 °C annealed Ti-51.1Ni alloys possess the various transformation type: A→R/M→R→A, A→R→M/M→R→A and A→R→M/M→A, respectively upon cooling/heating (A: parent phase B2, CsCl-type structure; R: intermediate phase, rhombohedral structure; M: martensite B19', monoclinic distortion of B19 structure).

In the cooling process, the shifting rate of the M and R peaks are towards higher and lower temperature, respectively, and both M and R peaks become broader and closer to each other with increasing θ_a , which indicates that the transformation temperature range becomes broader. Both peaks are nearly coalesced when annealed at 650 °C due to A→M transformation.

In the heating process, the shifting rate of the M' peak is

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