

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→R/M→R→A to A→R→M/M→R→A to A→R→M/M→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 TiNi₂, TiNi₃ 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 filed 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

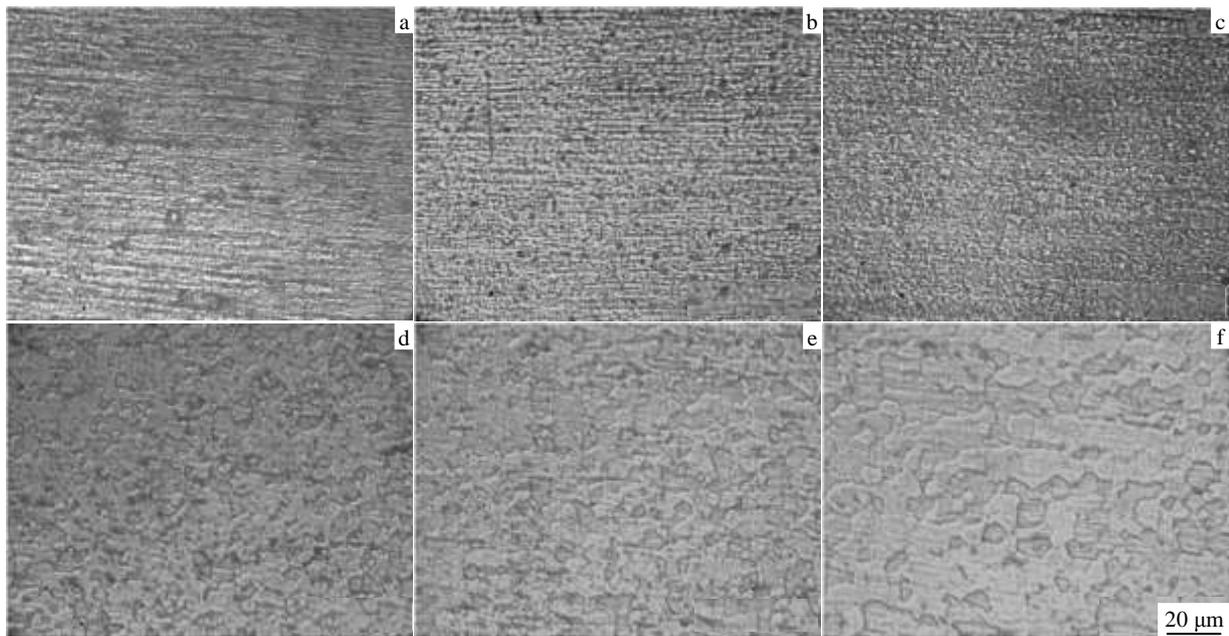


Fig.1 OM microstructures of Ti-51.1Ni alloys annealed at different temperatures: (a) 400 °C, (b) 570 °C, (c) 590 °C, (d) 600 °C, (e) 630 °C, and (f) 650 °C

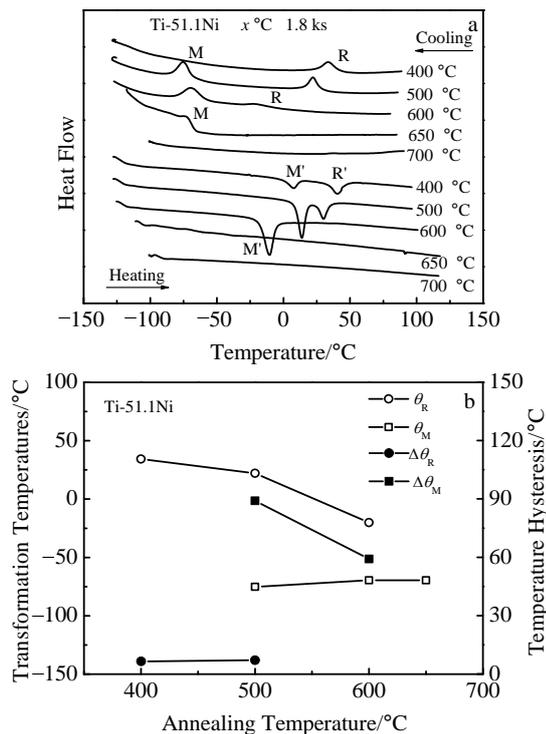


Fig.2 Effect of annealing temperature on DSC curves (a) and transformation temperatures (θ_R , θ_M), temperature hysteresis ($\Delta\theta_M$, $\Delta\theta_R$) (b) of Ti-51.1Ni alloy

towards higher firstly and then lower temperature, while the R' peak towards lower temperature and both M' and R' peaks become steeper and closer to each other with increasing θ_a , which indicates that the reverse transformation temperature

range becomes narrower. These two reverse transformation peaks overlap when the specimen is heat-treated at 600 °C due to M→A transformation.

The R and M transformation peak temperature were denoted by θ_R and θ_M , respectively and the transformation temperature hysteresis were denoted $\Delta\theta_R$ and $\Delta\theta_M$, respectively, (the temperature hysteresis was equal to temperature difference value of transformation peak between forward transformation and inverse transformation, i.e. $\Delta\theta_M = \theta_M - \theta_M$).

The specimens annealed at 400~700 °C for 30 min are plotted in Fig.2. As shown in Fig.2b, with increasing θ_a , the θ_R and $\Delta\theta_M$, decrease rapidly, the θ_M increases slowly, and the $\Delta\theta_R$ is nearly not changed as ~6.5 °C. With increasing θ_a , the θ_R decreases gradually, when the θ_a increasing from 400 to 600 °C the θ_R is dropped from 34.37 to -20.19 °C and reduces 54.56 °C. With increasing θ_a , the θ_M increases gradually, when the θ_a increasing from 400 to 600 °C, the θ_M is increased by 5.6 °C. While the $\Delta\theta_M$ is decrease rapidly, when the θ_a increases from 400 to 600 °C, the $\Delta\theta_M$ decreased from 89 to 59 °C, decreased by 30 °C, a drop of 33.7%.

Compared with Ref.[18,19], it is found that the change of Ni content has a significant effect on the transformation type and transformation temperature of two component Ti-Ni SMA. When the Ni content is increased by 0.3%, low temperature (below 500 °C) annealed Ti-Ni SMA by second order transformation types turns into a first-order transformation type, the transformation peak of high temperature (greater than 650 °C) annealed Ti-Ni SMA completely retired, and the θ_M is decreased 29 °C. Increasing Ni can affect the transformation type and reduce the transformation temperature of Ti-Ni SMA, which is favorable for developing low

temperature superelasticity alloy.

2.3 Effect of annealing temperature on deformation characteristic of Ti-51.1Ni alloy

2.3.1 Effect of annealing temperature on stress-strain curves of Ti-51.1Ni alloy

Fig.3 shows a definition schematic of the characteristic parameters in typical stress-strain curve. In the chart, the stress-strain curve and the X axis to form area size mark a ability of alloy for energy consumption upon deformation, and directly reflects energy consumption properties of alloy.

Fig.4 shows the tensile stress-strain curve (Fig.4a), platform stress and fracture strain (Fig.4b) of Ti-51.1Ni alloys after annealing treatment for 30 min at 350, 400, 450, 500, 550, 600, 650 and 700 °C. As shown in Fig.4a, in the drawing process, the alloy with different annealing states have experienced elastic deformation→M re-orientation or stress-induced M (depending on the magnitude relationship between the M'_s , M'_f and T_d)→M elastic and plastic deformation→M strain hardening→fracture stages and so on. Figure 4b shows that the θ_a has a significant influence on the σ_m of alloy. The σ_m decreases and then increases with θ_a increasing. The minimum σ_m 210 MPa and the maximum σ_m 646 MPa are obtained after annealing at 450 and 700 °C, 436 MPa apart. Annealing treatment of the alloy improved the uniformity of the organization, made dislocation rearrangement and reduced the dislocation density, and weakened the strengthening effect of dislocations plug on the platform stress^[23], σ_m decreased, and when gradually increased primarily due to fine grain strengthening effect of recrystallization annealing.

θ_a can also affect the ε_b significantly. As shown in Fig.4b, the ε_b increased and then decreased with the θ_a up. The ε_b was 50.83% and 47.23%, respectively, when the θ_a was 600 and 650 °C. Thus, the alloy can obtain higher ε_b value after annealing treatment at 600~650 °C, that is to say the plastic deformation ability is better, suitable for molding processing.

Compared with Ref.[19], it is found that the change of Ni content has a significant effect on the deformation behavior of

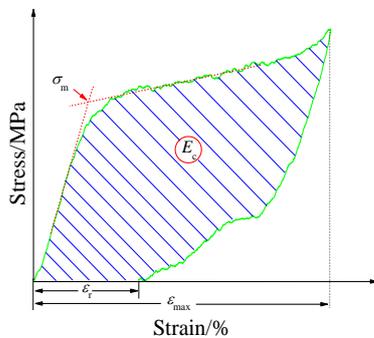


Fig.3 Definition of characteristic parameters σ_m , ε_r , ε_{max} and E_c in typical stress-strain curve (σ_m : platform-stress, ε_r : residual strain, ε_{max} : setting-strain value, E_c : energy consumption, i.e., the integral value of envelope area with stress-strain curve and the x axis)

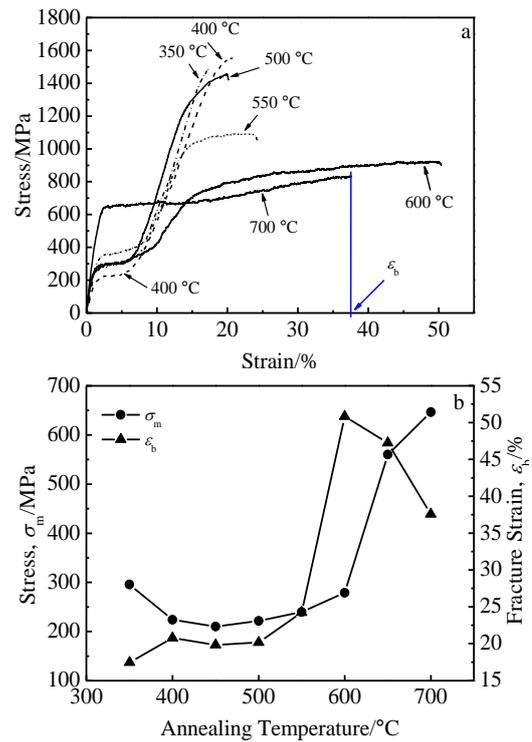


Fig.4 Effect of annealing temperature on stress-strain (a), platform stress σ_m and fracture strain ε_b (b) of Ti-51.1Ni alloy (test temperature 10 °C)

Ti-Ni SMA. The ε_b of the 550 °C annealed Ti-50.8Ni alloy was 80%, while the maximum value of the ε_b of the 600 °C annealed Ti-51.1Ni alloy was 50%, and their difference is 30%. It can be seen that the Ni content increased by 0.3% significantly decreased the ductility of the alloy, so that the temperature range of the alloy forming process is narrow. This is mainly related to the precipitation of Ni rich precipitates.

2.3.2 Effect of annealing temperature on shape memory effect, superelasticity of Ti-51.1Ni alloy

The following Fig.5 gives the SME, SE, E_c and ε_r of Ti-51.1Ni alloy after annealing treatment for 30 min at 350, 400, 450, 500, 550, 600, 650 and 700 °C. As shown in Fig.5a, stress platform existed on loading/unloading stress-strain curve of different annealed alloys, and the physical meaning of the platform depends on the magnitude relationship between the M'_s , M'_f and T_d . When the T_d is lower than the M'_s , the physical meaning of the stress platform is M reorientation; when the T_d is higher than the M'_s and lower than the M'_f , the physical meaning of the stress platform is M reorientation + stress induced M; when the T_d is higher than the M'_f , the physical meaning of the stress platform is stress induced M.

We can infer from Fig.2a that the alloy is M+A states and shows SME+SE when the $T_d=10$ °C is higher than the M'_s and lower than the M'_f after annealing treatment at 400~500 °C. The M'_f of the alloy annealed at 600 °C is much lower than the

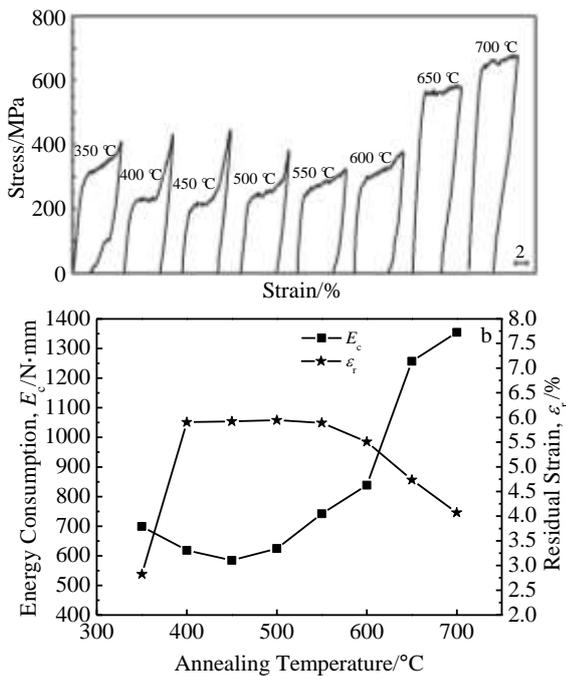


Fig.5 Effect of annealing temperature on shape memory effect, super-elasticity (a), energy consumption properties E_c and residual strain ϵ_r (b) of Ti-51.1Ni alloy (test temperature 10 °C)

$T_d = 10$ °C, as a result, the physical meaning of the stress platform is stress induced M. Overall, the alloy properties changes from SME+SE to SE with the θ_a increasing.

Alloy SE is mainly caused by M transformation induced by stress and the internal friction of its inverse transformation, and a lot of easily moving twin boundary and phase boundary can be found in the thermal elastic M. The anelasticity migration of these interfaces can absorb a lot of energy during the phase transformation, which result in a nonlinear relationship between the stress and strain of SMA and the hysteresis loop effect. As the ability of extra energy dissipation of alloy can be marked by the size of the hysteresis loop area, the damping value of SE alloy is much higher than that of other ordinary materials^[24]. As can be seen in Fig.5b, alloy E_c is increased with the increase of θ_a . So an annealing treatment temperature higher than 550 °C will be reasonable when the alloy is used to produce energy consumption of damper and related buffer shock absorption devices. The ϵ_r increases first and then decreases with the enhanced θ_a and the ϵ_r of the alloy annealed state at 350 and 700 °C shows a relatively smaller values, namely a bigger SE share and a better super elastic properties. An appropriate annealing treatment temperature can be chosen between 400 and 600 °C during the process of manufacturing super elastic devices.

2.4 Discussion

The transformation of Ti-Ni alloy in the state of cold processing + annealing is affected by the processing state, alloy elements and heat treatment process etc.

The alloy of low transformation temperature is beneficial to making super-elastic element in low temperature environment such as Arctic and north area, large thermal hysteresis alloy can be used as connecting element and small thermal hysteresis alloy is for temperature sensitive driving element. The size of transformation peak, morphology change and alloy tissue defect are affected by defect stress field, transformation stress field and phase transition process energy release of the alloy. A to R transformation of Ti-Ni alloy belongs to parent phase to martensite phase transformation, and the essence is the transformation of higher symmetry line ordered structure of CsCl to lower symmetry rhombohedral structure. R to M transformation belongs to M to M, and the essence is the transformation of low symmetry rhombohedral structure to lower symmetry monoclinic structure. Therefore, the stress field induced by R to M phase transition is higher than that of A and R transformation. When the annealing temperature is low, there are larger organization structure defect stress field, more effective nucleation site and smaller A to R transformation resistance of cold processing alloy, so the A to R transformation is easy to occur. The R phase transition peak is small and sharp and the transformation temperature is high because less energy is released during the cooling process. Various structural defects stress field of the alloy phase transition have larger resistance on R to M transformation, make M phase transition hardly occur, the corresponding released energy increases during the process of cooling; thus M phase transition peak is large and flat and the transformation peak temperature is low, and the R and M peaks are far away from each other. As the annealing temperature θ_a increases, the residual defect density of cold processed alloy declines, the interaction stress field and R to M transformation weaken, the released energy decreases, and the M phase transition peak temperature rises rapidly. The decrease of R phase transition peak temperature is related to the effective nucleation sites decrease. The R and M peaks and then toward each other and eventually merge.

Annealing temperature θ_a may have a great influence on the deformation properties of Ti-51.1Ni alloy at low temperature. The plasticity of Ti-51.1Ni alloy annealed at 600~650 °C is better than that at 350~550 °C, which is closely related to the microstructure, microscopic stress and dislocation density of the alloy due to annealing state at different temperatures. The organization of the alloy annealed below 590 °C is fibrous with large stress field of structure defects, high dislocation density, high strength and low plasticity. The organization of the alloy annealed at 590~650 °C shows a recrystallization annealing state such as axle, enhanced uniformity, weakened defect stress and dislocation strengthening effect, small grain size, grain refining strengthening effect, increased plasticity and reduced intensity.

3 Conclusions

1) The recrystallization temperature of Ti-51.1Ni SMA is about 590 °C.

2) With the increase of the θ_a , the transformation types of Ti-51.1Ni alloy upon heating/cooling are changed from A → R/M → R → A to A → R → M/M → R → A and then to A → R → M/M → A, θ_R and $\Delta\theta_M$ decrease, θ_M increased, $\Delta\theta_R$ does nearly not change at about 6.5 °C.

3) The θ_a affects low temperature deformation behavior of Ti-51.1Ni alloy significantly. When deformation is at 10 °C, the alloys show SME+SE characteristics annealed at 400~550 °C and show SE characteristics annealed at 600~700 °C. The alloy characteristics are transformed from SME+SE to SE with the θ_a increased.

4) The 590~650 °C annealed alloy could obtain excellent capability of plastic deformation and 50.83% of ε_b , so the forming processing temperature could be in the range of 590~650 °C. When the alloys are used for energy consumption of damper and damping device, the suitable θ_a could be higher than 550 °C, and for making superelastic device, the suitable θ_a could be below 400 °C less or above 600 °C.

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Ti-51.1Ni 形状记忆合金相变和低温形变特性

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摘要: 用示差扫描量热仪(DSC), 光学显微镜和拉伸试验研究了退火温度对形变处理态 Ti-51.1Ni (原子分数, %) 形状记忆合金相变和低温形变特性的影响。结果表明, 随退火温度升高, 合金冷却/加热时相变类型由 A→R/M→R→A 型向 A→R→M/M→R→A 型再向 A→R→M/M→A (A-母相 B2, R-R 相, M-马氏体相) 型转变, R 相变温度和 M 相变热滞降低, M 相变温度升高, R 相变热滞变化不大, 保持在 6.5 °C 左右。在 10 °C 变形时, 400~550 °C 退火态合金呈现形状记忆效应(SME)+超弹性(SE)特性, 600~700 °C 退火态合金呈现 SE 特性, 随退火温度升高, 合金由 SME+SE 向 SE 特性转变。合金的退火再结晶温度为 590 °C, 在 590~650 °C 退火后合金可获得 50.83% 的断裂应变值, 塑变性能优良, 成型加工温度可选定在 590~650 °C 之间。在使用该合金制作能耗阻尼器及相关缓冲减震装置时退火处理温度可选择大于 550 °C; 制作超弹性器件时, 退火处理温度可选择低于 400 °C 或高于 600 °C。

关键词: Ti-Ni 形状记忆合金; 应力诱发马氏体; 形状记忆效应; 超弹性; 相变; 形变

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