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Structure and shape recovery characteristics of Ti-50.0%Ni thermomechanically treated industrial wire

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Abstract. The effect of post-deformation annealing (PDA) temperature in a range 300 to 700°C and induced stain (3 to 24%) on shape recovery temperature range (SRTR) was studied for an industrial Ti-Ni wire. To determine the characteristic temperatures of martensitic transformations, a differential scanning calorimetry was used. SRTR was determined by the method of bending deformation followed by heating for shape recovery. The wire structure was studied by X-ray diffraction and TEM methods. The original structure of wire is a B19'- martensite or a mixture of B19', R -phase and B2- austenite containing a well-developed dislocation substructure. To obtain structure uniformity along the wire length, the PDA temperatures of 500-600ºC are recommended. The SRTR at the wire of near-equiatomic Ti-Ni alloys produced by warm drawing can be controlled using PDA in the temperature range 400 to 700°C. SRTR in the 70-100°C range is achieved by means of PDA at 400 to 650°C (SRTR increases in this PDA range). With the increasing of induced strain from 5 to 24%, the high-temperature shape memory effect is appears and grows: a non-monotonic A_F growth from 90 to 150°C and SRTR broadening are observed. Shape recovery parameters of studied wire are high: the maximum completely recoverable strain of 4 - 5%, the maximum recoverable strain of 7 - 13%, and they can be controlled using PDA.

1. Introduction

Techniques for regulation of functional properties (FP) are different for Ti-Ni shape memory alloys (SMA) of different composition. For the non-aging equiatomic and near-equiatomic Ti-Ni SMA, the basic method of FP control is thermomechanical treatment (TMT), including severe plastic deformation (SPD), forming various structures : from well-developed recovered and polygonized dislocation substructure to nanocrystalline structure [1-4]. Shape memory sensors and thermoregulators for fire-prevention devices are successfully used in practice [5]. The wire of "high-temperature" near-equiatomic Ti-Ni alloys is an optimum material for production of heat-sensitive elements (fire sensors, starting arrangements). The aim of the present work is to study the effect of thermomechanical action parameters on the structure and FP of Ti-Ni industrial wire.

2. Experimental

Three Ti-Ni based alloys (1, 2 and 3) having near-equiatomic chemical composition were studied. The effect of post-deformation annealing (PDA) and induced strain (3 to 24%) on shape recovery temperature range (SRTR) was studied for an industrial Ti-Ni wire.

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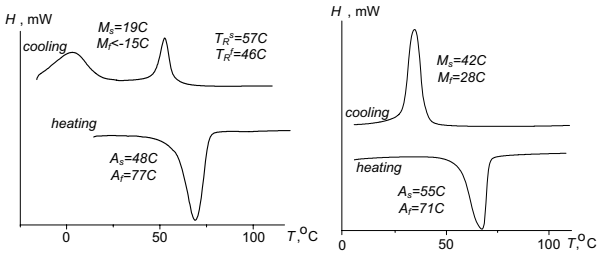
After warm drawing, the wire samples were annealed in the 300 to 700°C range for 1 hr and 30 min (at 700°C). To determine the temperature range of martensitic transformations (TRMT), a differential scanning calorimeter "Perkin Elmer" was used. SRTR and shape recovery characteristics were determined by the method of bending deformation followed by heating for shape recovery. The structure was studied by X-ray diffraction (DRON 3.0 diffractometer) and TEM (Tesla BS-540 microscope) methods.

3. Results and Discussion

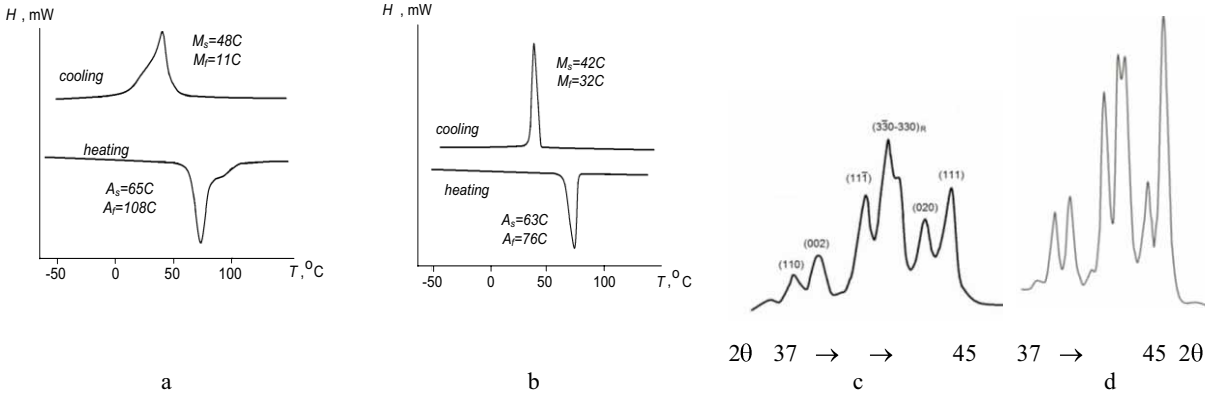
3.1 DSC, X- ray diffraction

The DSC results show the R-phase presence in initial state after warm drawing and after PDA in 400°C in alloys 1 and 2. The B2→R→B19′ (T_{Rs} = 57ºC и T_{Rf} = 46ºC) transformation sequence on cooling and B19′→B2 on heating (Fig. 1a). In this case, the characteristic temperatures of direct martensitic transformation are M_s =19ºC, M_f below –15ºC; characteristic temperature range of the reverse one-stage transformation are A_s = 48ºC, A_f = 77ºC.



a b

Fig. 1. DSC results for alloy1 after PDA at 400ºC(a) and 650°C(b).



a b c d

Fig. 2. DSC results and X-ray diffractograms for alloy 2 after PDA at 400ºC(a, c) and 650°C(b, d).

In alloy 2 the peaks of the transformation heat on cooling and heating are asymmetrical and broadened, and one can not say definitely about B2→R transformation occurence. The shape of curves testifies to heterogeneity of wire structure. The B2→R transformation presences proved to be true by X-ray diffraction study of alloy 2 in initial warm-drawing state and after PDA at 400-600°C: there is a distinct (330)–($\bar{3}30$) R-phase doublet in the diffractograms (Fig.1 c,d).

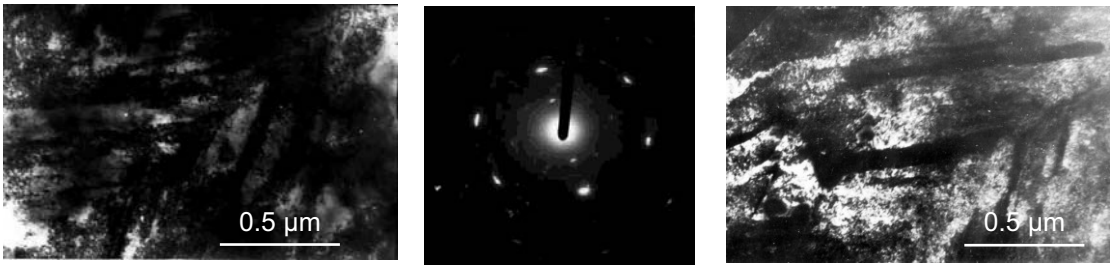
With the PDA temperature increasing to 600-700°C, the structural heterogeneity is eliminated and lattice defectness is reduced. As a result of recrystallization, the temperature range of direct martensitic transformation of alloy 1 is shifted to higher temperatures: M_s = 42°C, M_f = 28°C against 19 and -15°C after PDA at 400°C (compare Figs. 1a and 1b), while less difference between the reverse MT temperatures was determined: A_s = 55°C, A_f = 71°C against 48 and 77°C respectively (compare Figs. 1a and 1b).

After PDA at 600-700°C, the B2↔B19' transformation sequence realizes (Fig.1 b, 2 b). A significant shortening of direct and reverse MT ranges in alloy 2 after PDA at 650°C is observed (Fig.2 b). The transformation sequence (on cooling and on heating) B2↔B19′ realizes like in alloy 1.

3.2 TEM

The TEM images analysis of Ti – Ni alloys with close to equiatomic composition is complicated because B19' - martensite due to features of the mechanism of transformation contains a rather high disposition density (Fig.3). The well-developed dislocation substructure of martensite is superposed on the dislocation substructure created in austenite by thermomechanical treatment. In this case, to differentiate between these two substructures (inherited from austenite and inherent in martensite) is not always possible. The TEM analysis also reveals some quantity of B2- austenite (and/or R – phase) at room temperature.

As can be seen at Fig. 3, in initial state after warm drawing, the alloy 1 has mixed structure including martensite, R – phase and austenite with very high dislocation density. The visual analysis of residual austenite areas shows that warm drawing forms a well-developed dislocation substructure of cellular type in austenite, termines a strong deformation hardening of the alloy. The corresponding both azimuthally and radial broadening of reflexes in the electron diffraction pattern observed (Fig.3 b). This is a consequence the imperfectness of crystal lattice orientation caused by the distortions brought by deformation- and transformation induced hardening.

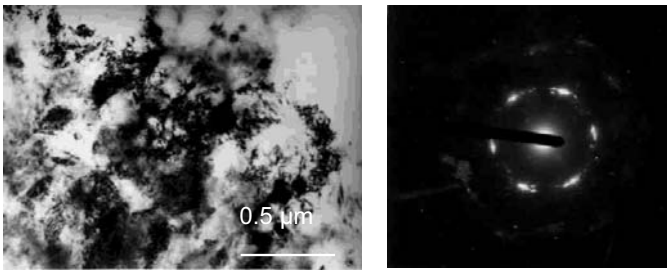


a) b) c)

Fig. 3. Structure of alloy 1 after warm drawing: zone axis close to <311>B2; a–bright-field image, b– electron diffraction pattern from "a", c– dark-field image.

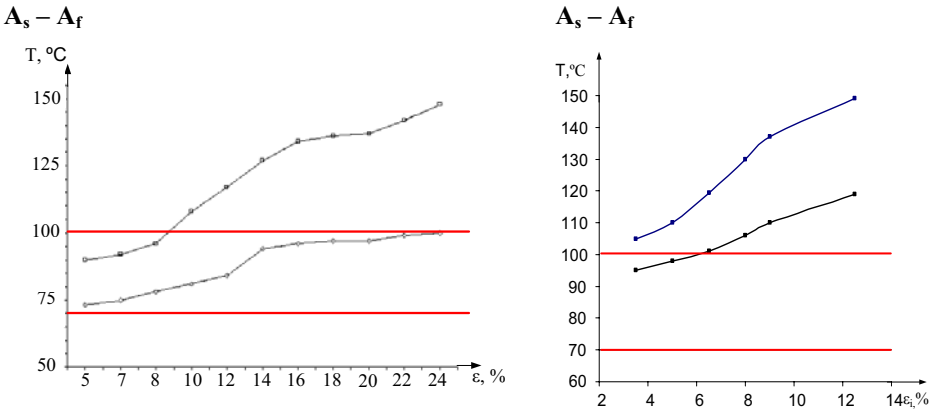
The post-deformation annealing of alloy 1 at 400°C leads to obvious changes in the dislocation substructure. The 50-200 nm subgrains inherited by martensite from austenite are observed (Fig. 4). In this case, the average orientation (positions of the diffraction reflex centers) and misorientation across the selected area (the length of arc reflexes) do not change, a fragmentation of arc reflexes becomes evident (Fig.4). That testifies to polygonization process development and polygonized dislocation substructure formation under 400°C- annealing [3,4].

The structure of alloy 3 in the initial as-drawn state is similar to the structure of alloy 1. PDA at high temperature (650ºC) followed by water-cooling i.e., ordinary quenching, leads to formation of the martensite structure with high dislocation density which is typical for quenched near-equiatomic Ti – Ni alloys.



a) b)

Fig.4. Structure and electron diffraction pattern of alloy 1 after drawing and annealing at 400°C: a – bright field image; b–electron diffraction from "a".



a) b)

Fig.5. SRTR of alloy 3 after PDA at 700°C(a) and alloy 1 after PDA at 650°C(b).

As an example, typical dependences of SRTR on the induced strain after different regimes of PDA are given in Fig. 6 (a-d) for alloy 2.

The range of ε_i values which provides the required temperature range of shape recovery, also tends to broadening and shifts aside to lower values with annealing temperature raising: for PDA at 400ºC and 500ºC the ε_i range extends contains values from 8 % to 14 %, for PDA at 550ºC– from 8 to 13.5 %, for PDA at 640ºC from 4.5 to 12 %(Fig. 6 a-d) and for PDA at 700ºC from 5 – 10.5 %. For alloy 2, it is possible to recommend PDA at 400,500 and 550ºC as optimum regimes because a combination of high shape recovery characteristics with the required temperature range of their realization is observed.

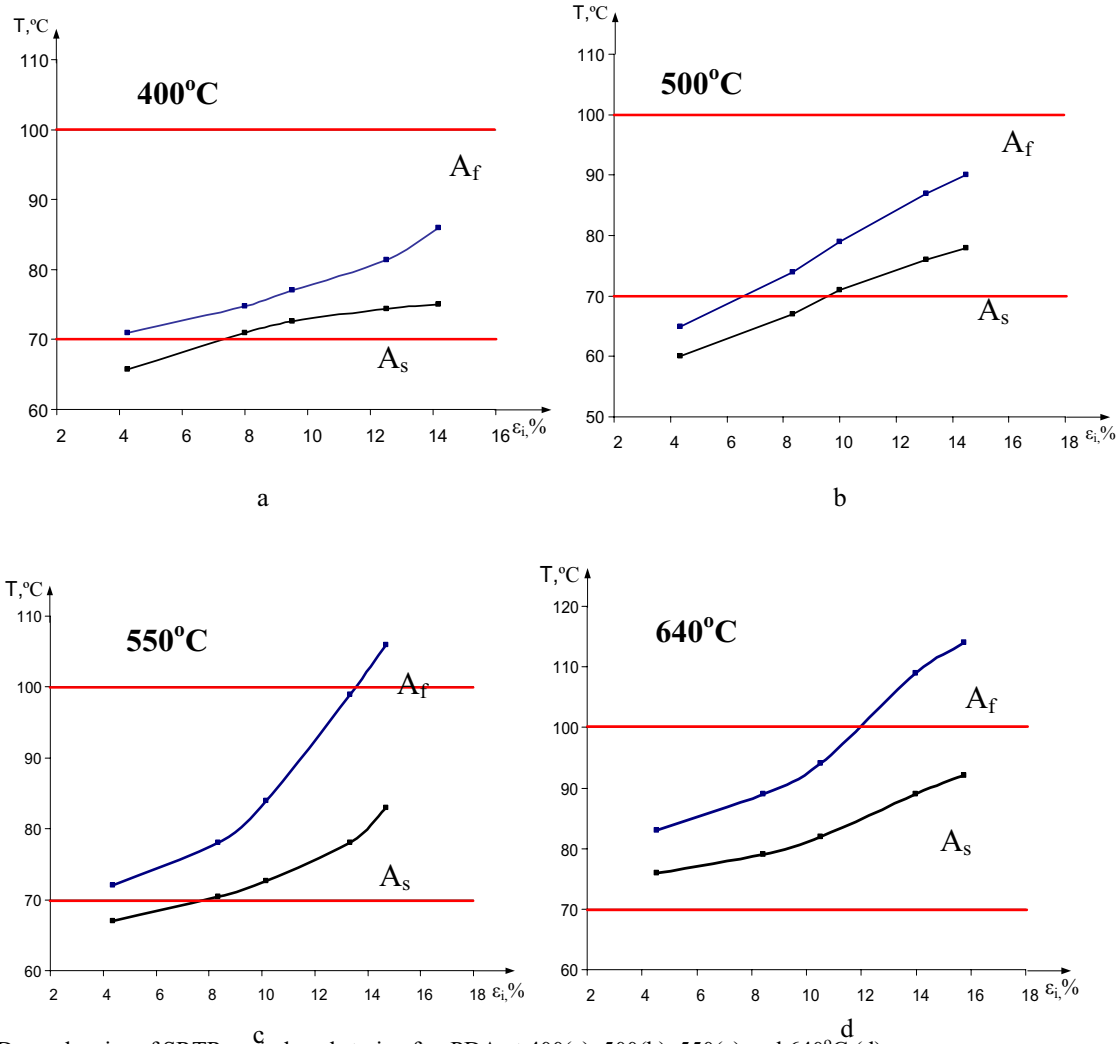


Fig. 6. Dependencies of SRTR on induced strain after PDA at 400(a), 500(b), 550(c) and 640°C (d)

3.3 Temperature range of shape recovery

As it has been shown above, the wire in initial state after warm drawing accumulates a high level of strain hardening. PDA leads to the alloy softening due to recovery, polygonization and recrystallization processes development, thus TRMT and SRTR are shifted to higher temperatures. The initial induced strain (which is completely or partialy recovered on heating) also shifts the transformation range to higher temperatures as compared to the temperatures determined without loading by DSC method. Thus, to obtain the required shape recovery (working) range from 70 to 100°C, it is necessary to find out the PDA regimes providing this temperature range for a wide range of induced strains.

A typical dependence of SRTR on the induced strain is presented in Fig. 5a for 1.0 - mm diameter wire in alloy 3 after PDA at 700°C (recrystallized structure of austenite). One can see non-monotonous change and broadening of SRTR with more intensive raising of A_f temperature (from 90°C to 148°C) in the induced strains range from ε_i=8 to 18 %. That illustrates the high-temperature SME realization, caused by the martensite plastic

deformation [6]. For the samples of alloy 1 annealed at 650ºC, the A_s – A_f temperatures do not lay into the required temperature range of 70-100°C (Fig.5b).

3.4 Shape recovery characteristics

Shape recovery parameters of wire: maximum recoverable strain (ε_r^{max}), residual irreversible strain (ε_f) and shape recovery rate ($\varepsilon_r/\varepsilon_i$), were determined as a function of induced strain, and PDA regimes (Fig.7).
It is necessary to note that all three alloys have very high shape recovery parameters for this category of "high-temperature" alloys: the maximum completely (100 %) recoverable strain $\varepsilon_{r,1}^{max}$ = 4÷5 %; the maximum recovery strain ε_r^{max} =7 – 13 %, and shape recovery rate $\varepsilon_r/\varepsilon_i$ not less than 80 %.

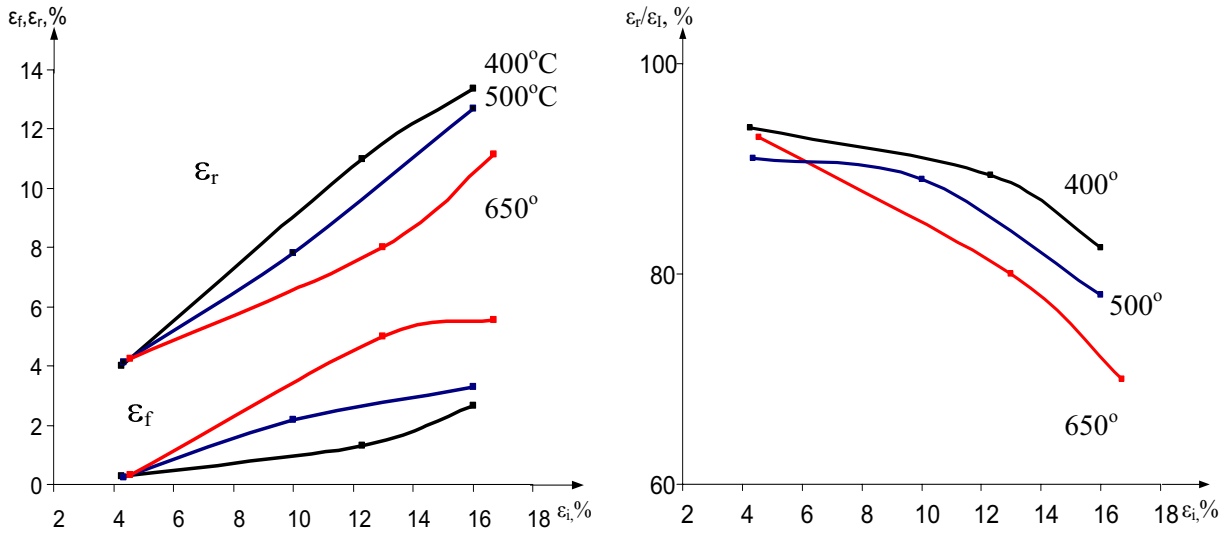


Fig. 7. Dependencies of shape recovery characteristics on induced strain and PDA temperature (alloy 2).

Besides the DSC analysis shows that the alloys have different phase composition at room temperature. Consequently, the strain inducing SME is accumulated by different structural mechanisms in alloys 1, 2 and 3 after different PDA regimes. Thus, in alloys 1 and 3 after PDA at 650°C, the structure of wire before deformation is mainly martensite, and the induced strain is accumulated by reorientation of B19′- martensite. In alloys 1 and 2 after PDA at 400°C, the induced strain is accumulated not only by the reorientation of martensite but also by the formation of stress-induced martensite from B2- and R-phases. Obviously, in this case deformation is accompanied by smaller irreversible distortions, that promotes more complete shape recovery.

4. Conclusions

1. The existing technology of Ti – Ni wire warm drawing not always provides homogeneous structure in diam. 1.0 – 2.0 mm sections. . The structure of the as-drawn wire represents itself B19'- martensite or a mixture of B19', R -phase and B2- austenite containing a well-developed dislocation substructure. To obtain structure uniformity, the PDA temperatures of 500-600ºC are recommended. It should be taken into consideration when forming a working shape of SME elements.
2. The SRTR of Ti-Ni near-equiatomic wire produced by warm drawing can be controlled using PDA in the temperature range 400 to 700°C. SRTR of 70–100°C is achieved by means of PDA at 400 to 650°C (SRTR increases in this PDA range).

The choice of optimum PDA temperature depends on melting composition of alloy. For example, the PDA temperature recommended for alloy 1 wire of is 400°C, for alloy 2 is 400 - 550°C, and for alloy 3 – 550 - 650°C, with 1 hr exposure.
3. With the increasing of induced strain from 5 to 24%, the high-temperature shape memory effect appears and grows: a non-monotonic A_f growth from 90 to 150°C and SRTR broadening are observed.
4. Shape recovery parameters of studied Ti-Ni wires are high: the maximum completely recoverable strain of 4 - 5%, the maximum recovery strain of 7 - 13%, and they can be controlled using PDA.

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