Effect of initial strengthening on recovery stress generation and isothermal relaxation processes in TiNi alloys

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ABSTRACT. Recovery stress (RS) generation and forced isothermal relaxation processes in Ti-50.5 at. % Ni and Ti-50.7 at. % Ni shape memory alloys were investigated using *in situ* X-ray structure analysis, X-ray texture analysis, transmission electron microscopy and mechanical tests. The alloys were subjected to thermomechanical treatments (TMT) including cold rolling followed by post-deformation heating in the range from 400 to 700°C for obtaining partially recovered, completely polygonized or recrystallized B2-austenite structures. Specimens containing partially recovered, polygonized or recrystallized structures obtained by thermomechanical treatment (TMT) were deformed in tension by 3 - 12 % strain inducing shape memory. When the austenite get low hardening by TMT or by deformation inducing SME, the RS generation is accompanied by elastic and plastic deformations. When the austenite get high hardening , the RS generation is accompanied by elastic deformation only. The isothermal RS relaxation is accompanied by elastic deformation of the austenite excluding the case of very high RS (> 1000 MPa) and recovery strain during relaxation (8 - 9%) when plastic deformation also takes place. During RS generation the reverse martensite transformation by the orientation variant «directly backwards» is restricted by external counteraction and other orientation variants can partially realize.

1. INTRODUCTION

Recovery stress (RS) is an important functional property of shape memory alloys. It generates in conditions of shape restoration at external counteraction. Mechanical behaviour of SMA at the recovery stress generation and relaxation is studied in details [1]. Structure and deformation mechanisms of these processes attracted less attention. It is known that on reaching a maximum recovery stress value a martensite relief in Cu-Al-Mn alloy disappears, i.e. RS generation process completes at A_f^σ temperature, and forced isothermal RS relaxation during following unloading at A_f^σ develops by austenite deformation [2]. At the same time there is no clear understanding of what structure and lattice orientation changes and deformation mechanisms accompany the RS generation and forced isothermal relaxation. It is shown that after the RS generation a two-way shape memory effect of «martensitic» type appears in Ti-Ni alloy [3]. It can be a consequence of the martensite plastic deformation during RS generation. However the residual martensite strain can be induced at a preliminary deformation stage. It is also important to know the peculiarities of an influence of the initial structure state formed by heat and thermomechanical treatment on the RS generation and isothermal relaxation processes. The present work is devoted to studies of the influence of preliminary alloy strengthening on recovery stress generation and forced isothermal relaxation in Ti-Ni shape memory alloys.

2. EXPERIMENTAL

Ti-50.5 % Ni ($M_s=28$, $M_f=0$, $A_s=40$, $A_f=50^0$C) and Ti-50.7 % Ni ($M_s=-8$, $M_f=-24$, $A_s=5$, $A_f=15^0$C) shape memory alloys were investigated using *in situ* X-ray structure analysis, texture analysis, transmission electron microscopy and mechanical testing.

Induction vacuum-melted alloy ingots after hot pressing and rotation forging were warm - or cold - drawn into wires of 0.9 or 1.2 mm in diameter. The wires were subjected to various thermomechanical treatments (TMT) including multi-pass cold rolling to bands of 0.2 - 0.25 mm in thickness followed by post-deformation heating at 400, 500 or 700 °C. These treatments create partially recovered, completely polygonized or recrystallized B2-austenite structures respectively.

Band specimens were deformed at room temperature in tension up to 3-3.5, 5 or 10 - 12% residual (after unloading) strain ε_i inducing a shape memory due to oriented stress-induced martensite formation and martensite reorientation. In the cases of $\varepsilon_i = 5$ or 10-12 % a shape change was accompanied by a conventional deformation-induced hardening. The conditions of TMT and following tensile deformation allow to create various combinations of TMT - induced hardening and tensile deformation-induced hardening levels.

Then a part of the deformed specimens was cut and pinched in a special heating stage at X-ray diffractometer DRON 2.0, heated to A_f^σ temperature (100 - 120 °C) for a maximum RS generation and isothermally unloaded at A_f^σ for a complete RS relaxation. The control experiments were heating of thermomechanically treated specimens to A_f point or thermomechanically treated and deformed in tension specimens to A_f point in conditions of free shape restoration. The schematic representation of the X-ray experiment is shown in Fig. 1, where the points of interest are numbered. The martensite and austenite X-ray line profiles were obtained by step scanning at numbered points, the $Fe_{K\alpha}$ - radiation was used. The B2 - austenite X-ray line half-width, interplanar distances d_{hkl}, extrapolated lattice parameter a_{B2} as well as austenite and martensite X-ray line integral intensities were estimated.
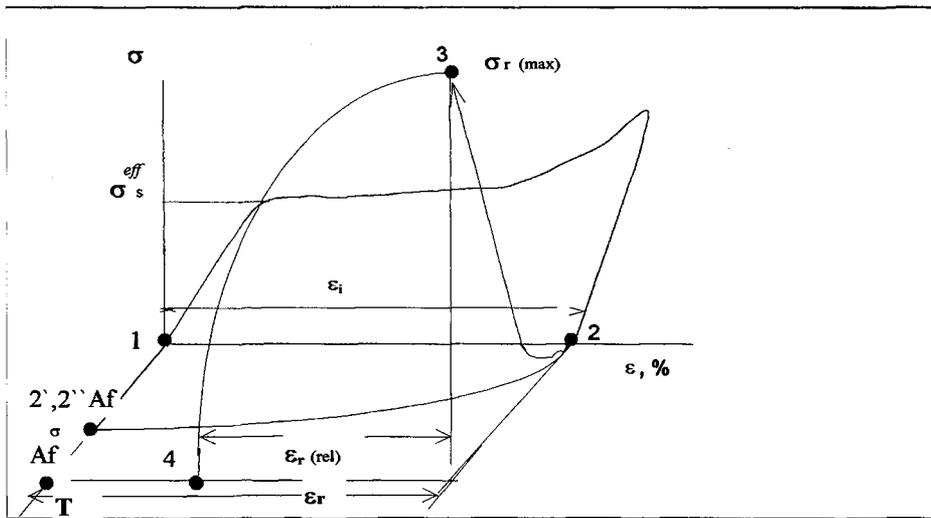

Figure1 – Schematic representation of the X-ray experiment.
1 - at room temperature after TMT; 2 - after tensile deformation and unloading at room temperature, ε_i – strain inducing SME, 2'- near A_f after reverse martensitic transformation; 2''- after free shape restoration near A_f, ε_r – recovery strain; 3 - after maximum recovery stress σ_r^{max} generation; 4 - after recovery stress isothermal relaxation, ε_r^{rel} – recovery strain during relaxation.

Another part of specimens after tensile deformation inducing SME and unloading was pinched in the testing machine, heated to the A_f point for a maximum RS generation and unloaded at this temperature for a forced isothermal RS relaxation. The following parameters were determined from mechanical tests: σ_r^{max} – a maximum RS, ε_i – an induced strain, ε_r^{rel} – recovery strain at maximum RS relaxation, σ_s^{eff} – an effective yield stress (see Fig. 1). A recovery strain ε_r was determined after free heating of deformed specimens to A_f point.

The elastic deformation was deduced from a comparison of B2-lattice parameter measured from rolling plane with the one measured in initial state. The plastic deformation was deduced from a comparison of the B2 X-ray line widths after recovery stress generation and relaxation with line width after free shape restoration.

Electron microscopic investigation was carried out at room temperature using 120 kV «Tesla BS-540» microscope. Thin foils were prepared from thermomechanically treated bands by window technics and initial structures were observed before the deformation inducing SME .

The B2-austenite crystallographic textures were investigated at room temperature after RS generation and relaxation and after free shape recovery. The direct pole figures of $\{110\}_{B2}$ and $\{211\}_{B2}$ reflexes were obtained.

3. RESULTS

Electron microscopic investigation shows that preliminary thermomechanical treatment including plastic deformation and post-deformation heating at different temperatures leads to formation of different types of B2-austenite structure. Post-deformation heating at 400 ^{0}C is accompanied by recovery processes in the austenite but a dislocation density remains rather high, a polygonization does not yet develop. So, thermomechanical hardening is high in this case. After heating at 500 ^{0}C very fine equiaxed subgrains 0.1 – 0.2 µm in diameter are observed through the whole specimen. It is a polygonized structure which determines partial softening of the austenite. So, the thermomechanical hardening is moderate in this case. Heating at 700 ^{0}C leads to a complete recrystallization and maximum softening of the austenite.

During mechanical tests the maximum values of the σ_r^{max} =1000 - 1200 MPa were obtained after high strain inducing SME, ε_i=10 - 12 %, on specimens with polygonized austenite substructure. At the same time very high recovery strains at RS relaxation were obtained, ε_r^{rel}=8 - 9 % (fig. 2a). Moderate values of σ_r^{max} =500 - 900 MPa were obtained after high strain inducing SME, ε_i=10 - 12 %, of specimens with recrystallized austenite, ε_i=5 % strain of specimens with partially recovered substructure, low values of σ_r^{max} =300 - 370 MPa were obtained after ε_i=3 - 3,5 % strain of specimens with polygonized and recrystallized structures (Fig. 2b). In the last two cases ε_r^{rel} was of 1 - 2 %.
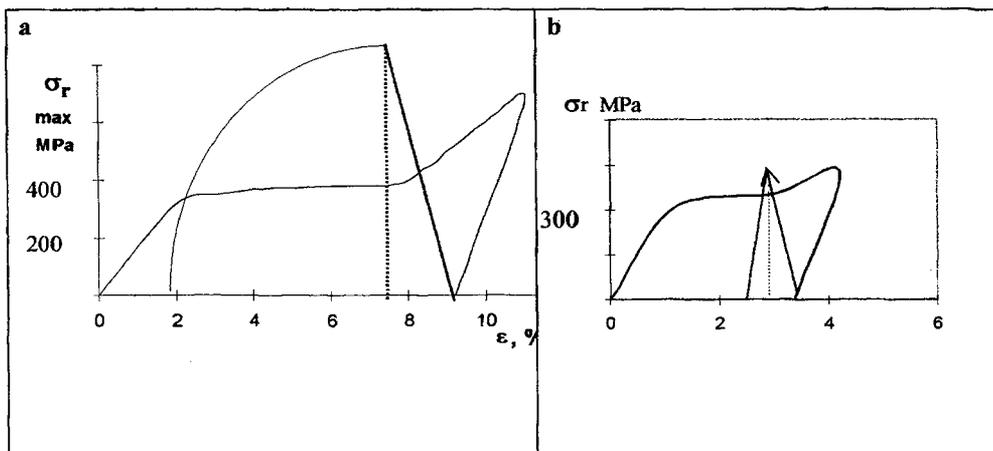
Figure 2 – Examples of strain, recovery stress generation and isothermal relaxation diagrams.   a – Ti-50,7 % Ni alloy, initially polygonized austenite, ε_i=10-12 %, σ_r^{max}=1200 MPa. b– Ti-50,5 % Ni alloy, initially recrystallized austenite, ε_i=3-3.5 %, σ_r^{max}=300 MPa.

There are two main cases for tensile deformation inducing SME: the first one (ε_i=5 and 10-12 %) results in «overdeformation» accompanied by conventional plastic flow, the second one (ε_i=3 %) brings no «overdeformation». The first case is characterized by incomplete strain recovery during heating to A_f point. In the second case strain recovery is complete.

According to X-ray study at room temperature the structure of the alloy 2 after thermomechanical treatment was mainly austenitic, and the structure of the alloy 1 was partially austenitic and martensitic. The martensite volume fraction increases with the increase in heating temperature from 400 to 700 ^{0}C. Therefore a comparison of X-ray line widths after these treatments and heating to A_f point is incorrect without taking into account a possible transformation-induced dislocation substructure. After inducing strains ε_i=5 and 10 - 12 % the structure of the alloy 1 becomes martensitic but after ε_i=3 - 3.5 % approximately 10% of the austenite remains untransformed.

The elastic deformation accompanies both recovery stress generation and relaxation process in all cases, as it seen from the lattice parameter changes after tensile deformation, recovery stress generation and isothermal relaxation in the example shown in (Fig. 3).
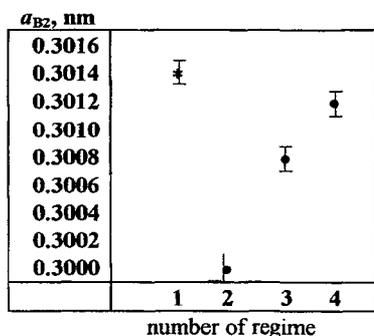
a_{B2}, nm

| 0.3016 |
| 0.3014 |
| 0.3012 |
| 0.3010 |
| 0.3008 |
| 0.3006 |
| 0.3004 |
| 0.3002 |
| 0.3000 |

1   2   3   4

number of regime

Fig. 3. Lattice parameter of B2- austenite of Ti-50,7 % Ni alloy.
Initially polygonized austenite,
ε_i =10 - 12 %.;
1 - initial state;
2 - after tensile SME deformation;
3 - after σ_r^{max} generation near A_f^σ point;
4 - after recovery stress isothermal.
relaxation near A_f^σ point.

The presence of plastic deformation depends on the hardening level of the austenite which can be evaluated approximately by σ_r^{max} level (Fig. 4).

Low hardening of the austenite by thermomechanical treatment or by deformation inducing shape memory effect or combined before the recovery stress generation leads to elastic and plastic deformations during the recovery stress generation.

High combined hardening of the austenite by thermomechanical treatment or by deformation inducing shape memory effect or combined before the recovery stress generation leads to the absence of plastic deformation during the recovery stress generation.

Forced isothermal recovery stress relaxation develops by elastic deformation of the austenite mechanism excluding the case of very high recovery stress (>1000 MPa) and recovery strain during relaxation (~6 - 9 %) when plastic deformation also takes place.

The austenite texture observed after high inducing strain and free shape restoration is rather week and complex due to complex thermomechanical prehistory (fig. 5). The crystallographic texture of the deformed austenite forming during recovery stress generation after high strains inducing SME ε_i=10 - 12 %, somewhat differs from the austenite texture after free shape restoration: the texture peaks are more weak and spread along β angle resembling a ring texture. So, in the case or recovery stress generation a recovery of the reverse martensitic transformation by the orientation variant «directly backwards» is restricted by external counteraction and other orientation variants partially set in. The said effect is not significant when ε_i=5 %.

$B_{B2\,(211)}$, 2θ	Alloy structure, ε_i	Strengthening by deformation, inducing SME	Strengthening by TMT	σ_r^{max}, MPa
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.5 % Ni austenite $\varepsilon_i = 3 - 3.5\,\%$	low	low	300
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.5 Ni polygonized austenite $\varepsilon_i = 3 - 3.5\,\%$	low	moderate	370
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50..5 Ni recrystallized austenite $\varepsilon_i = 10 - 12\,\%$	high	low	600
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.5 Ni partially recovered austenite $\varepsilon_i = 3 - 3.5\,\%$	low	high	500
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.7 Ni recrystallize austenite $\varepsilon_i = 10 - 12\,\%$	high	low	900
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.7 Ni recrystallize austenite $\varepsilon_i = 5\,\%$	moderate	low	600
0.3 / 0.2 / 0.1 / 0 / -0.1	Ti-50.7 Ni polygonized austenite $\varepsilon_i = 10 - 12\,\%$	high	moderate	1200 high ε_r^{rel}
$2''$　3　4				

Figure 4 – Comparison of X-ray line width of B2-austenite after free shape restoration, recovery stress generation and relaxation for different strengthening levels of Ti-50,5 and Ti-50,7 % alloys.
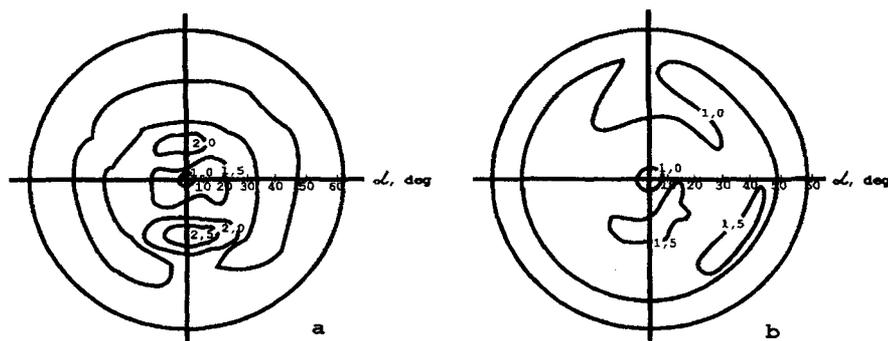
Figure 5 – Direct pole figures {211}$_{B2}$. Ti-50,7 % Ni alloy. Preliminary recrystallized austenite, induced strain ε_i=10 %. a - after free shape restoration; b - after recovery stress isothermal relaxation.

4. CONCLUSIONS

1. Low hardening of the austenite by thermomechanical treatment or by deformation inducing shape memory effect or combined before the recovery stress generation leads to elastic and plastic deformations during the recovery stress generation.

2. High hardening of the austenite by thermomechanical treatment or by deformation inducing shape memory effect or combined before the recovery stress generation leads to elastic deformation only during the recovery stress generation.

3. Forced isothermal recovery stress relaxation at A_f point develops by elastic deformation of the austenite mechanism excluding the case ofvery high recovery stress (>1000 MPa) and recovery strain during relaxation (~8 - 9 %) when plastic deformation also takes place.

4. When the recovery stress is high, the reverse martensite transformation by orientation variant «directly backwards» is resricted by strong external counteraction and other orientation variants partially realize.

ACKNOWLEDGMENTS

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