### Effect of Heat Treatment on Temperature Response and Recovery Stress of Ti-Ni-Cu Shape Memory Alloy after Pre-Deforming

Y. Takeda\*, T. Yamamoto\*\*, A. Goto\*\* and T. Sakuma\*\*, \*TAKE R&D, Shizuoka 426-0087 \*\*Faculty of Engineering, Oita University, Oita 870-1192, sakuma@cc.oita-u.ac.jp

This work describes the effect of heat treatment on temperature response and recovery stress of Ti-Ni-Cu shape memory alloy. The materials used in this study are Ti-41.7Ni-8.5Cu (at%) alloys which have different cold working ratio (10, 40%). Heat treatment temperatures of wire specimen are varied from 623 K to 773 K. Heating-cooling tests after pre-strain ranged from 1% to 3% at the isothermal temperature of 263K, are carried out to investigate the thermomechanical behavior of Ti-Ni-Cu alloy. The temperature response is decreased with increasing pre-strain and decreasing heat treatment temperature. However, the recovery stress becomes larger with increasing pre-strain and decreasing heat treatment temperature, cold working ratio and pre-strain on transformation

Key words: Ti-Ni-Cu alloy, shape memory alloy, actuator, pre-strain, heat treatment temperature

#### **1. INTRODUCTION**

temperature hysteresis.

The shape memory alloy (SMA) is better known as a functional material, since it has unique characters which are shape memory effect and superelastic behavior <sup>[1]</sup>. Ti-Ni SMAs have been already put to practical use in the fields of industry and medical because they have not only superior shape memory characteristic, but also ductility, toughness, fatigue resistance, corrosion resistance, abrasion resistance <sup>[2-4]</sup>. For example, Ti-Ni SMAs are used as a pipe coupling, an antenna of a mobile telephone, auto parts, an actuator and spring in the field of industry <sup>[5, 6]</sup>. They are also used as a stent in the chest surgery field and an orthodontic wire in the dentistry field <sup>[7, 8]</sup>. Furthermore, SMAs are being expected to be used as on actuator in the micromechanics field, since SMAs allow reduction in size and weight and can also generate recovery stress and strain <sup>[9, 10]</sup>.

The response of SMA actuators is dependent on its temperature, and the generative force of them is determined by the recovery stress of SMAs. Ti-Ni-Cu alloys are one of promising materials for a component of SMA actuators, because the recovery stress increases and the stress hysteresis and transformation temperature hysteresis decrease with increasing copper content in the alloys <sup>[11, 12]</sup>. However, it is reported that the recovery stress and transformation temperature of SMAs are influenced by manufacturing processes such as processing rate and heat treatment <sup>[13-16]</sup>.

This work carried out heating-cooling tests under constrained strain condition using the Ti-41.7Ni-8.5Cu (at%) alloy, for studying the effect of heat treatment on temperature response and recovery stress of Ti-Ni-Cu shape memory alloy after pre-deforming.

### 2. EXPERIMENTAL PROCEDURES

The chemical composition of the alloy used in this study is Ti-41.7Ni-8.5Cu (at%). The specimen shape is a

wire with 1 mm diameter and 70 mm gage length. The specimen was processed in the following manner; the Ti-Ni-Cu shape memory alloy ingot was made using a high frequency induction vacuum furnace, and then was hot forged and hot extruded followed by cold drawing and intermediate annealing. Cold working ratio (CW) of the specimen was varied from 10% and 40%. Furthermore, the specimen was heat treated at the temperature range from 623 K to 773 K for 3.6ks in air. Martensite start temperature  $M_s$ , martensite finish temperature  $M_f$ , austenite start temperature  $A_s$  and austenite finish temperature  $A_f$  of the specimen measured by differential scanning calorimetry (DSC) are listed in Table I.

To investigate the effect of heat treatment on the temperature response and recovery stress of the Ti-Ni-Cu shape memory alloy, heating-cooling tests under constrained strain condition were carried out using the Ti-41.7Ni-8.5Cu (at%) alloy wire.

Figure 1 (a) shows a schematic diagram of the experiment to obtain the stress-strain curve in the heating-cooling test under constrained strain condition. The specimen was loaded to given pre-strains ranged from 1% to 3% at the isothermal temperature of 263 K lower than martensite transformation finish temperature

Table I Transformation temperatures of Ti-41.7Ni-8.5Cu (at%) alloy measured by DSC

T <sub>HT</sub> (K)	Transformation temperatures (K)							
	M <sub>f</sub>		M <sub>s</sub>		A <sub>s</sub>		A <sub>f</sub>	
	CW10	CW40	CW10	CW40	CW10	CW40	CW10	CW40
623	313.7	274.7	336.1	323.5	333.9	292.7	351.5	336.2
673	311.5	291.6	332.4	318.5	332.4	309.7	346.8	335.4
723	315.4	306.2	336.4	320	333.9	324.9	350.3	339.7
773	318.1	320.4	330	333.7	336.1	338.2	347.5	350.6

Effect of Heat Treatment on Temperature Response and Recovery Stress of Ti-Ni-Cu Shape Memory Alloy after Pre-Deforming



Fig.1 Schematic drawing of experimental procedure.

and was subsequently unloaded (O $\rightarrow$ A $\rightarrow$ B). And then, the specimen was heated at 3 K/min (B $\rightarrow$ C) and cooled at 5 K/min (C $\rightarrow$ B') under constrained strain condition. From the stress-strain curve and the stress-temperature relation during heating-cooling progress (B $\rightarrow$ C $\rightarrow$ B') shown in Fig.1 (b), the recovery stress  $\sigma_R$ , martensite start temperature  $M_s$ ', reverse transformation start temperature  $A_s$ ' and reverse transformation finish temperature  $A_f$ ' after the pre-strain were obtained, respectively.

### 3. RESULTS AND DISCUSSION

### 3.1 Effect of heat treatment on recovery stress

Figure 2 shows the variation of the recovery stress with heat treatment temperature. The recovery stress decreases with increasing heat treatment temperature and with decreasing pre-strain. In addition, the recovery stress after pre-strains of 2% and 3% below the heat treatment temperature of 723 K increases with increasing CW. However, there is little effect of CW on the recovery stress after pre-strain of 1% and that after pre-strains of 2% and 3% at the heat treatment temperature of 773 K. It is reported that the critical stress for slip increases since the cold working increases the dislocation density within the material <sup>[17]</sup>. Therefore, the recovery stress increases with increasing CW since the extra damage introduced into the material due to pre-strain decreased with increasing CW. On the other hand, the recovery stress decreases with increasing heat







Fig.3 Variation of  $\Delta A_s$  (=A<sub>s</sub>'-A<sub>s</sub>) with heat treatment temperature.



Fig.4 Variation of  $\Delta A_f$  (= $A_f$ '- $A_f$ ) with heat treatment temperature.

treatment temperature, because an increase of heat treatment temperature causes the rearrangement and annihilation of dislocations <sup>[18]</sup> so that the dislocation density within the material decreases.

## 3.2 Effect of heat treatment on transformation temperature

Figure 3 shows the variation of  $\Delta A_s$  (=A<sub>s</sub>'-A<sub>s</sub>) with heat treatment temperature. The A<sub>s</sub>' and A<sub>s</sub> show the reverse transformation start temperature after pre-strain



Fig. 5 Variation of  $\Delta M_s$  (=M<sub>s</sub>'-M<sub>s</sub>) with heat treatment temperature.



Fig. 6 Variation of  $A_{\rm f}$ '- $A_{\rm s}$ ' with heat treatment temperature.

and that measured by DSC, respectively. In the case of CW=40%, the  $\Delta A_s$  decreases with increasing heat treatment temperature, because an increase of heat treatment temperature relax the elastic strain energy stored during cold working so that the As becomes a large as shown in Table I. In the case of CW=10%, the heat treatment temperature hardly affect the As since the release of elastic strain energy is saturated. Therefore, the  $\Delta A_s$  of CW=10% is maintained nearly-constant. Furthermore, the  $\Delta A_s$  decreases with increasing CW and with decreasing pre-strain. It is reported that the A<sub>s</sub> is raised by the deformation in M phase because the elastic strain energy stored during martensite transformation is relaxed by slip <sup>[19]</sup>. The dislocation density increases with increasing CW. And also, the further slip decreases with decreasing pre-strain. Consequently, it is believed that the As' becomes a small since the stored elastic strain energy is hardly relaxed with increasing CW and with decreasing pre-strain.

The variation of  $\Delta A_f$  (= $A_f$ '- $A_f$ ) with heat treatment temperature is shown in Fig.4. The  $A_f$ ' and  $A_f$  show the reverse transformation finish temperature after pre-strain and that measured by DSC, respectively. The  $\Delta A_f$ decreases with increasing heat treatment temperature. As shown in Fig.2, the recovery stress decreases with increasing heat treatment temperature. Thus, it is



Fig. 7 Variation of  $A_s$ '- $M_s$ ' with heat treatment temperature.



Fig. 8 Relationship between  $A_f'-A_s'$  and recovery stress  $\sigma_R$ .

presumed that the  $\Delta A_f$  becomes a small because a decrease of the recovery stress decreases a driving force needed to finish the reverse transformation. In contrast, the  $\Delta A_f$  increases with increasing CW and with increasing pre-strain due to the increase of a driving force needed to finish the reverse transformation, because the recovery stress increases with increasing CW and pre-strain as shown in Fig.2.

The variation of  $\Delta M_s$  (=M<sub>s</sub>'-M<sub>s</sub>) with heat treatment temperature is shown in Fig.5. The M<sub>s</sub>' and M<sub>s</sub> show the transformation start temperature after pre-strain and that measured by DSC, respectively. M<sub>s</sub> decreases with the decrease of A<sub>f</sub>. Therefore, the  $\Delta M_s$  decreases with increasing heat treatment temperature and with decreasing CW and pre-strain.

Figure 6 shows the variation of the reverse transformation temperature difference  $(A_f'-A_s')$  with heat treatment temperature. The  $(A_f'-A_s')$  decreases with increasing heat treatment temperature and with decreasing CW and pre-strain. The temperature response increases with the decrease of the  $(A_f'-A_s')$  since a SMA can recover its shape by the small temperature change if the reverse transformation temperature difference is small. Therefore, this result means that the temperature response increases with increasing heat treatment temperature and with decreasing CW and pre-strain.

The variation of transformation temperature hysteresis  $(A_f'-M_s')$  with heat treatment temperature is shown in Fig.7. There is little effect of the heat treatment temperature on the  $(A_f'-M_s')$ . Additionally, the variation of  $(A_f'-M_s')$  with CW is within 5 K.

# 3.3 Relationship between reverse transformation temperature difference and recovery stress

Figure 8 shows the relationship between the transformation temperature difference  $(A_f'-A_s')$  and the recovery stress. The recovery stress decreases with increasing heat treatment temperature as shown in Fig.2. The  $(A_f'-A_s')$  also decreases with increasing heat treatment temperature as shown in Fig.6. As a result, the  $(A_f'-A_s')$  increases almost linearly with increasing recovery stress. The decrease of the  $(A_f'-A_s')$  increases the temperature response. Thus, this result indicates that the temperature response increases with decreasing recovery stress.

### 4. CONCLUSIONS

Heating-cooling tests under constrained strain condition were carried out using the Ti-41.7Ni-8.5Cu (at%) alloy, for studying effect of heat treatment on temperature response and recovery stress of Ti-Ni-Cu shape memory alloy after pre-deforming. The obtained results are summarized as follows;

(1) The recovery stress has decreased with increasing heat treatment temperature and with decreasing CW and pre-strain.

(2) The  $\Delta A_s$  has decreased with increasing heat treatment temperature. The  $\Delta A_f$  has also decreased with increasing heat treatment temperature.

(3) The reverse transformation temperature difference  $(A_f'-A_s')$  decreases with increasing heat treatment temperature and with decreasing CW and pre-strain. Therefore, the temperature response increases with increasing heat treatment temperature and with decreasing CW and pre-strain.

(4) There has been little effect of the heat treatment temperature on the transformation temperature hysteresis  $(A_f'-M_s')$ .

(5) The reverse transformation temperature difference  $(A_{f} - A_{s})$  has increased with increasing recovery stress.

This result shows that the temperature response increases with decreasing recovery stress.

### ACKNOWLEDGEMENT

Part of this study was supported by Grant-in-Aid for Scientific Research (C) (19560095).

### REFERENCES

[1] T. Honma, J. Jpn. Soc. Mech Eng., 87, 517-522 (1984).

[2] S. Miyazaki, T. Sakuma and T. Shibuya, *properties* and *Application Development of Shape Memory Alloy*, 233-260(CMC, Japan 2001).

[3] K. Yamauchi, Jpn. Inst. Met., 7, 495-499 (1993).

[4] M. Miyazaki, Jpn. Inst. Met., 24, 69-74 (1985).

[5] T. Honma, J. Jpn. Soc. Mech. Eng., 87, 786 (1984).

[6] K. Yamauchi, J. Jpn. Soc. Mech. Eng., 32, 495 (1993).

[7] I. Okata, J. Jpn. Soc. Mech. Eng., 107, 26-29 (2004).

[8] Y. Okamoto, *The stomatological Society.*, *Japan*, **55**, 5-14 (1988).

[9] D. Homma, *Journal of the Robotics Society of Japan*, **8-4**, 107-109 (1990).

[10] D. Homma, J. Jpn. Inst. Met., 77-84 (1989).

[11] S. Miyazaki, K. Mizukoshi, T. Ueki, T. Sakuma and Y. Liu, *Mater. Sci. and Eng.*, A, **273-275**, 658-63 (1999).

[12] T. Sakuma, U. Iwata, H.Takaku, N. Kriya, Y. Ochi,

and T. Matsumura, *Trans. Japan Soc. Mech. Eng.*, A, **66-664** 748-54 (2000).

[13] T. Saburi, *Metals and Technology*, **59**, 11-18 (1989).

[14] S. Miyazaki and H. Sakamoto Jpn. Inst. Met., 24, 33-44 (1985).

[15] T. Todoroki and H. Tamaura, J. Jpn. Inst. Met., 50, 538-545 (1986).

[16] T. Sakuma, Y. Mihara, H. Toyama, Y. Ochi and K. Yamauchi, *Jpn. Inst. Met.*, **47**, 787-791 (2006).

[17] S. Miyazaki, *Journal of the Society of Materials Science, Japan*, 27, 59-67 (1990).

[18] M. Hosogi, N. Okabe, T. Sakuma and S. Miyazaki, J. Soc. Mat. Sci., Jpn., **51**, 48-53 (2002).

[19] M. Piao, K. Otsuka, S. Miyazaki and H. Horikawa, *Mater Trans.*, *JIM*, **34**, 919-929 (1992).

(Recieved December 27, 2007; Accepted May 5, 2008)