

# The Aging Effect on Cu-Zn-Al Shape Memory Alloys with Low Contents of Aluminum

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The effects of various quenching and aging treatments including direct quenching (DQ), up-quenching (UQ), and step-quenching in a water bath at 100 °C (SQWB) and in an oil bath at 100 °C (SQOB) have been studied on two Cu-Zn-Al shape memory alloys (SMA's) with the aluminum content less than 3.8 wt pct. The structures of two heats at room temperature are parent (alloy A) and martensite phases (alloy B), respectively. It is found that the rapid rate of cooling after solution treatment is an essential requirement for a Cu-Zn-Al SMA with low aluminum content; otherwise, a less rapid rate of cooling during the quenching process causes the precipitation of  $\alpha$  phase, which has been observed in SQWB specimens. The temperature of the martensitic transformation ( $M_s$ ) is found to be increased with both the size of ordering domain and the degree of ordering. Nevertheless, a stable and sound shape memory property has been obtained on specimens of either UQ or SQOB. Furthermore, the periods of isothermal aging required to initiate precipitation of either  $\alpha$  or  $\alpha_1$  phase have been examined at various temperatures; then the time-temperature-transformation (T-T-T) diagrams are constructed. From the calculation of the activation energy for precipitation of  $\alpha_1$ , the shape memory life expectancy of alloy B with 3.14 wt pct Al content has been determined to be only 41 days when operating at a temperature as small as 100 °C. For this reason, it is suggested that a suitable operating temperature of Cu-28.2Zn-3.14Al SMA should be less than 80 °C.

## I. INTRODUCTION

THE application of martensitic alloys in shape memory devices requires a stable and reliable transformation temperature to trigger a mechanical action at the designed temperature. However, both the transformation temperature and the shape memory behavior of martensitic structure have been found to be sensitive to the aging treatment, especially for certain copper-base SMA's. Stice and Wayman have found this problem of martensitic stabilization in Cu-Sn SMA as the degradation of shape memory behavior by aging at low temperature.<sup>[1]</sup> Many researchers have recently reported the effects of low-temperature aging in martensite and parent phases of Cu-Zn-Al SMA's.<sup>[2-8]</sup> These degrading effects of thermal aging may limit the reliability of the material as a temperature-triggering-action component and may cause the malfunction of a balance spring in certain devices, such as actuators and thermostats, etc.

Scarsbrook *et al.*,<sup>[5]</sup> Segers *et al.*,<sup>[6]</sup> Van Humbeeck *et al.*,<sup>[7]</sup> and Qixuan *et al.*<sup>[8]</sup> have found that the stabilization of Cu-Zn-Al martensite is a thermally activated process and that the stabilization problem can be reduced by step-quenching (SQ) treatment. Van Humbeeck *et al.*<sup>[7]</sup> have also reported that if no precipitation is involved, once the stabilized martensite phase is heated to a temperature above the increased  $A_s$  and  $A_f$  (the temperatures at which the transformation of martensite to austenite starts and finishes during heating, respectively), the samples

are restored to their original state. Therefore, Schofield and Miodownik<sup>[9]</sup> have proposed to employ an up-quenching (UQ) treatment to solve the room-temperature aging problem of Cu-Zn-Al martensite as the previous SQ one does.

The foundation for the study of thermal aging on the shape memory effect (SME) in Cu-Zn-Al alloy has been laid in previous works.<sup>[2-9]</sup> Because no comprehensive tests of shape memory recovery between SQ and UQ treatments, as well as the difference between oil bath and water bath in the SQ process, and because the effect of a low content of aluminum in Cu-Zn-Al alloys during aging treatment have never been discussed, we attempt in this paper to fill this gap by presenting the effects of direct-quenching (DQ), UQ, and SQ in a water bath at 100 °C (SQWB) and in an oil bath at 100 °C (SQOB) on shape memory recovery, transformation temperature, and X-ray diffraction of two heats of Cu-Zn-Al having a low aluminum content (less than 3.8 wt pct). For the purposes of comparison, the structures of these two heats at room temperature are designed to be parent (alloy A) and martensite phases (alloy B), respectively.

## II. EXPERIMENTAL PROCEDURE

Two Cu-Zn-Al SMA's, with low contents of aluminum (3.72 wt pct Al content in alloy A, 3.14 wt pct Al in alloy B) and other chemical compositions, as listed in Table I, were prepared from raw materials of electrolytic grade with an induction melting furnace. The resulting ingots were first homogenized at 850 °C for 2 hours, then hot-rolled at 650 °C to sheet plate about 2-mm thick. All test specimens cut from rolled plate were divided into four groups for various heat treatments shown in Figure 1. The four groups, DQ, UQ, SQWB, and SQOB, are described as follows:

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**Table I. Chemical Compositions of Alloys (in Weight Percent)**

Alloy	Zn	Al	Cu
A	27.4	3.72	bal.
B	28.2	3.14	bal.

(1) Group DQ specimens were solution-treated at 850 °C for 5 minutes and directly quenched into a water bath at 20 °C ± 3 °C (room temperature).

(2) Specimens of group UQ were directly quenched into water at 20 °C ± 3 °C after solution treatment, then immediately up-quenched into an oil bath at 100 °C for 10 minutes and quenched again in water at 20 °C ± 3 °C.

(3) and (4) Specimens of groups SQWB and SQOB were immediately quenched into boiling water and oil baths at 100 °C for 10 minutes after solution treatment, respectively, then quenched again into water at 20 °C ± 3 °C.

In order to study the effects of various quenching processes on the SME of the two Cu-Zn-Al heats, specimens of the four groups were carefully examined after isothermal aging at room temperature, as well as the UQ specimens at selected temperatures in the range of 150 °C to 600 °C for various periods of aging.

Tensile tests of plate-type specimens were conducted at room temperature with a moderate strain rate of 0.02 min<sup>-1</sup> to reduce the effect of aging at room temperature during tests. All of the tested specimens were immediately unloaded after reaching 2 pct strain according to the strain gages and then were uniformly heated to a temperature above the reverse martensitic transformation temperature,  $A_f$ , to study the mechanical behavior and the shape memory capability. The four-probe technique was employed for resistance measurement of all specimens to find the starting temperatures of martensitic transformation,  $M_s$ .

### III. RESULTS AND DISCUSSION

#### A. Structure

The microstructure examinations of all specimens after various quenching processes reveal that their average grain sizes are close to each other, about 600 μm, as shown

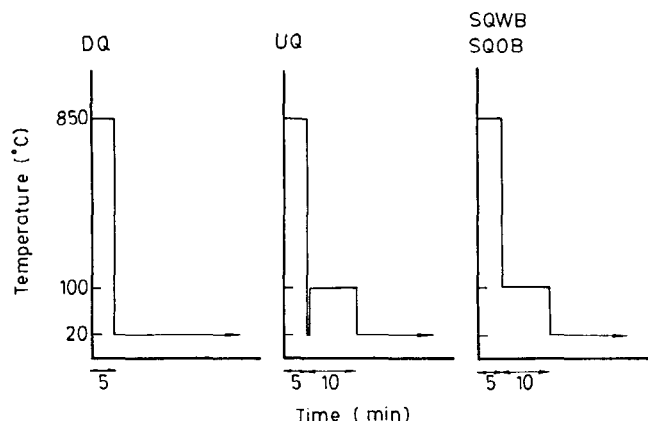


Fig. 1—Schematic illustration of various heat treatments.

in Figure 2. The DQ alloy A specimens demonstrate a parent phase structure indicated in Figure 2(a). All of the other alloy A specimens (*i.e.*, UQ, SQWB, SQOB) also have the parent phase appearance. The metallograph of the DQ alloy B specimen in Figure 2(b) shows many plate-type martensite phases. The same type of martensite phase is also observed in UQ, SQWB, and SWOB alloy B specimens. Besides, many irregular and coarse precipitates within the interior of grains and mostly along the grain boundary are found in SQWB alloy B specimens, as shown in Figure 2(c).

The phase identifications have been conducted with the selective-area diffraction pattern (SADP) of transmission electron microscopy (TEM). Figure 3(a) displays that the parent phase of DQ alloy A specimen has a  $D0_3$  ordering structure. The structures of plate-type martensites in DQ and UQ alloy B specimens were identified as 9R and 18R long-range stacking order, respectively,<sup>[10]</sup> as shown in Figures 3(b) and (c). The results of phase identifications of all specimens are listed in Table II. All of the alloy A specimens exhibited a  $D0_3$  ordering structure. The martensite phases in the UQ, SQWB, and SQOB alloy B specimens were determined to have the 18R structure. However, DQ alloy B specimen has the 9R martensite. The irregular blocky precipitates in SQWB alloy B sample were identified to be  $\alpha$  precipitates with face-centered cubic (fcc) structure, similar to the  $\alpha$  phase in quenched Cu-Zn alloys reported by Kajiwara and Kikuchi<sup>[11]</sup> and Thumann and Hornbogen.<sup>[12]</sup>

The transformation from body-centered cubic (bcc) to B2 parent phase is a second-order transition which occurs during the process of quenching in water,<sup>[13]</sup> but the ordering reaction from B2 to  $D0_3$  can be depressed by water quenching. The  $D0_3$  structure observed in DQ alloy A specimens might be due to the aging at room temperature to transform B2 to  $D0_3$  during the preparation of the thin foil for TEM examination.<sup>[14]</sup> The temperature for the transition from B2 to  $D0_3$ ,  $T_c(D0_3)$ ,<sup>[15]</sup> of alloy A is about 290 °C; therefore, the  $D0_3$  structure is easily found in UQ, SQWB, and SQOB alloy A samples after being treated at 100 °C for 10 minutes.

The 9R martensite structure in DQ alloy B specimens was directly transformed from B2 ordering of the parent phase and associated with depression of the  $D0_3$  ordering transition. When the 9R martensite immediately was up-quenched at 100 °C for 10 minutes during the UQ treatment, all of the martensite reversely transformed to parent phase and completed the  $D0_3$  ordering transformation. In the step-quenched process, the 100 °C temperature of either water bath (SQWB) or oil bath (SQOB) after solution treatment is greater than the  $M_s$  but lower than  $T_c(D0_3)$  of alloy B; therefore, the  $D0_3$  ordering transition has been accomplished in these two processes. Finally, all of the UQ, SQWB, and SQOB alloy B samples were quenched to room temperature and all of the  $D0_3$  structures transformed to the 18R martensite.

#### B. Shape Recovery Property

The study of the effect of aging at room temperature on shape recovery property of either parent phases (alloy A) and martensite phases (alloy B) was conducted

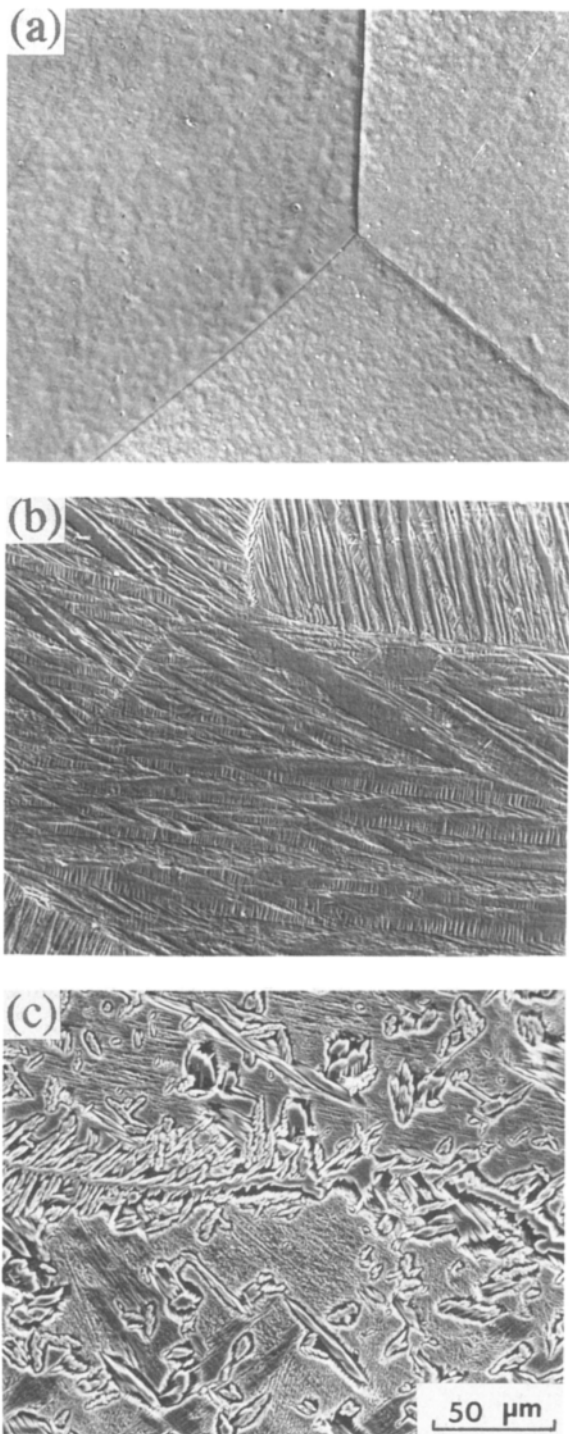


Fig. 2—Microstructures observed with SEM of (a) DQ alloy A specimen, (b) DQ alloy B specimen, and (c) SQWB alloy B specimen.

by tensile tests at room temperature. Typical stress-strain curves of various samples either as-quenched or aged at room temperature for 90 hours (alloy A specimens) or 8 hours (alloy B specimens) are demonstrated in Figures 4 and 5. The shape recovery capability ratio,  $\theta$ , the quantitative measurement of the shape recovery property of a material, is defined to be

$$\theta = 1 - \frac{\epsilon_p}{\epsilon_{\max}}$$

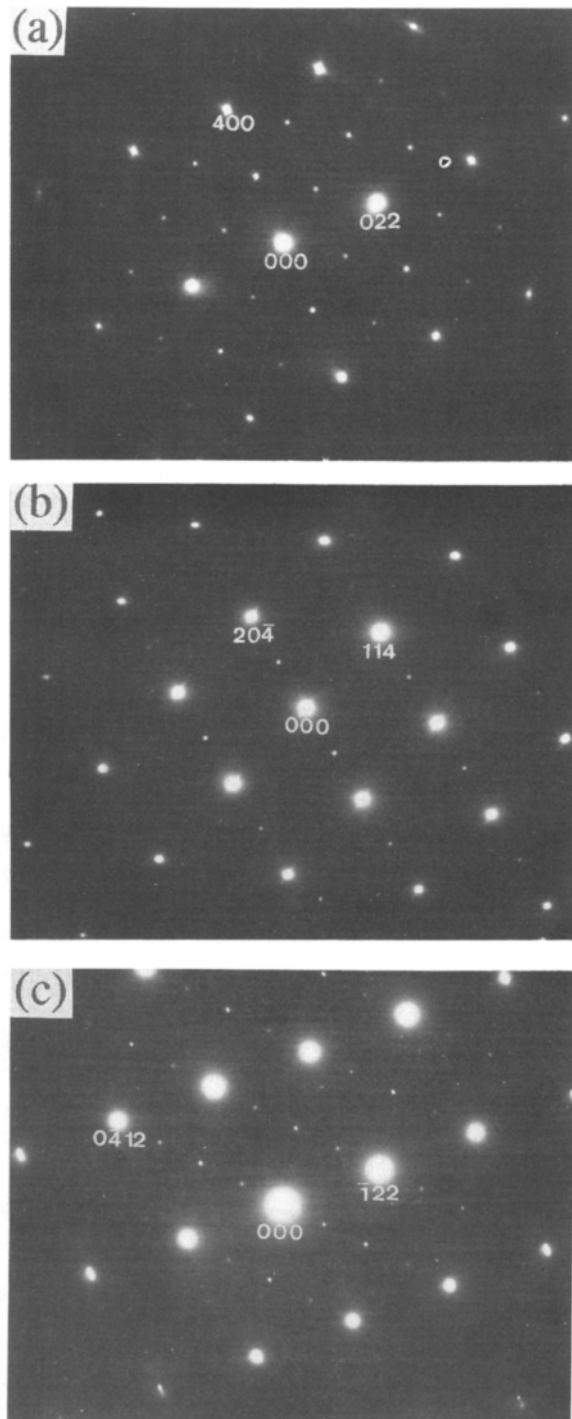


Fig. 3—The electron diffraction patterns of (a)  $D0_3$  ordering parent phase in DQ alloy A specimen, (b) 9R martensite phase in DQ alloy B specimen, and (c) 18R martensite phase in UQ alloy B specimen.

where  $\epsilon_p$  = residual plastic strain after tensile tested and  $\epsilon_{\max}$  = maximum tensile strain during testing, fixed as 2 pct in this study.

Comparing the yield stress ( $\sigma_y$ ) and  $\theta$  values of various alloy A samples with different room-temperature aging conditions in Figure 4, it is found that the specimen with the smaller  $\sigma_y$  value (SQWB < UQ < SQOB < DQ) always has a greater  $\theta$  (SQWB > UQ > SQOB > DQ), *i.e.*, with a smaller residual plastic strain. It is also

**Table II. The Observed Structure and  $M_s$  Temperature of Specimens after Various Heat Treatments**

Specimens	Structure	$M_s$	$M_s$	
		(As-Quenched)	(Aged for 18 h)	
Alloy A	DQ	$\beta$ phase ( $D0_3$ )	-32 °C	-12 °C
	UQ	$\beta$ phase ( $D0_3$ )	-16 °C	-7 °C
	SQWB	$\beta$ phase ( $D0_3$ )	-9 °C	-1 °C
	SQOB	$\beta$ phase ( $D0_3$ )	-19 °C	-9 °C
Alloy B	DQ	9R martensite	—	—
	UQ	18R martensite	49 °C	52 °C
	SQWB	$\alpha$ + 18R martensite	54 °C	45 °C
	SQOB	18R martensite	43 °C	50 °C

discovered that specimens aged longer at room temperature exhibit smaller values of  $\sigma_y$  and greater of  $\theta$ .

Comparison of the tensile test results of alloy B samples shows a more interesting variation behavior after holding at room temperature, as shown in Figures 6(a) and (b). The shape memory property of an as-quenched DQ specimen is deteriorated seriously upon increase of both  $\sigma_y$  and  $\epsilon_p$  after aging at room temperature. A value of  $\theta$  as great as 90 pct for the as-quenched DQ alloy B specimens decreased rapidly to 70 pct after holding at room temperature for only 100 minutes, which was the typical result of stabilization of quenched martensite in Cu-Zn-Al SMA.<sup>[5,16]</sup> However, both Figures 5 and 6 indicate a poor SME of SQWB alloy B specimens, such

as the large fluctuation amplitudes and the smallest values of  $\theta$  for various aging conditions at room temperature, correlate well with the observed irregular blocky  $\alpha$  precipitates in Figure 2(c). Besides, the more the  $\alpha$  precipitate, the smaller the value of  $\theta$  obtained. It is concluded that both DQ and SQWB processes are unsuitable for alloy B with low aluminum content to preserve the stable, sound SME property. On the other hand, an excellent and stable SME behavior upon conditions of low-temperature aging is easily obtained on either UQ or SQOB alloy B specimens with values of  $\theta$  greater than 95 pct.

### C. Martensitic Transformation

For all of the as-quenched specimens of various groups, we quickly measured the change of resistance with respect to the sample temperature to obtain the  $M_s$  temperature in as-quenched condition. The  $M_s$  temperatures of various specimens after aging at room temperature for

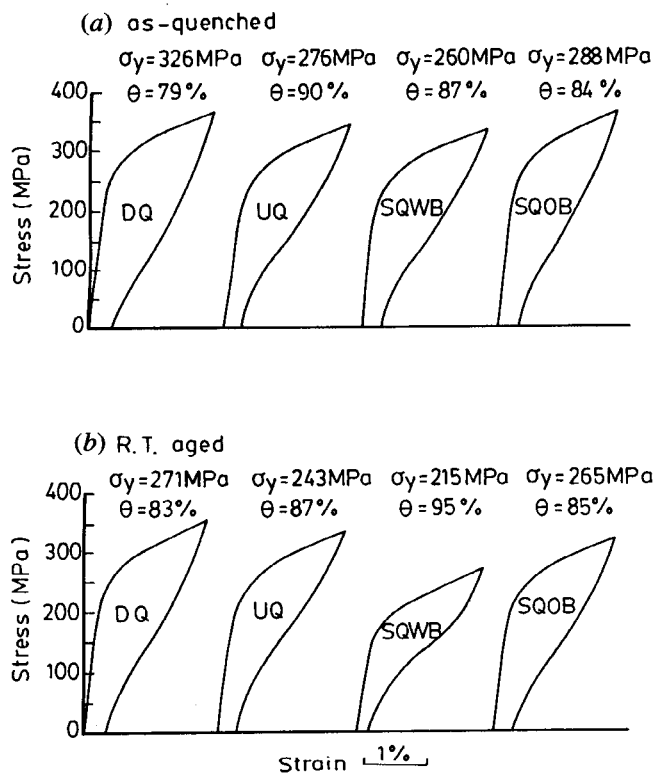


Fig. 4—Typical stress-strain curves of various alloy A specimens after aging at room temperature for either (a) less than 10 min (as-quenched) or (b) 5400 min. The measured yield stress and shape recovery ratios are also indicated.

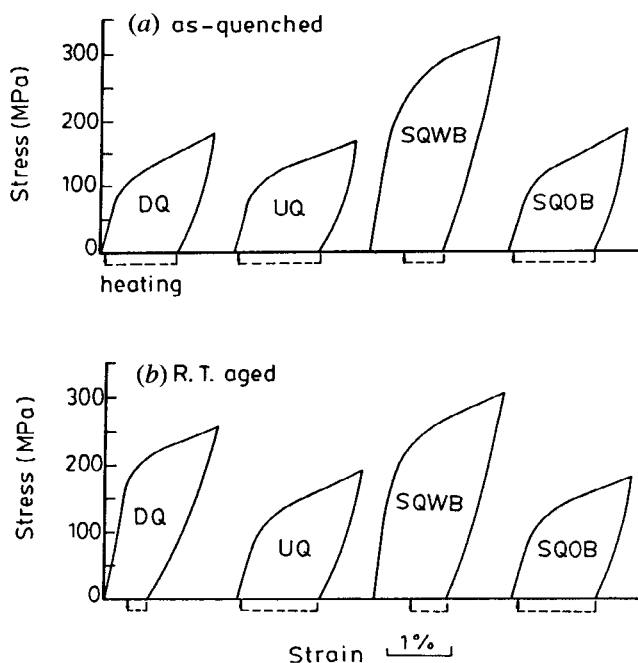


Fig. 5—Typical stress-strain curves of various alloy B specimens after aging at room temperature for either (a) less than 10 min or (b) 480 min

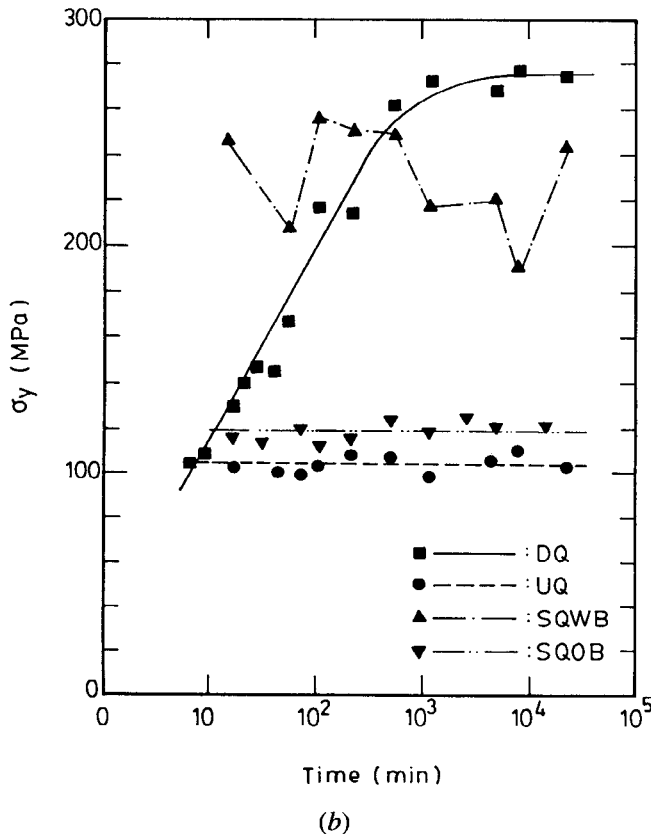
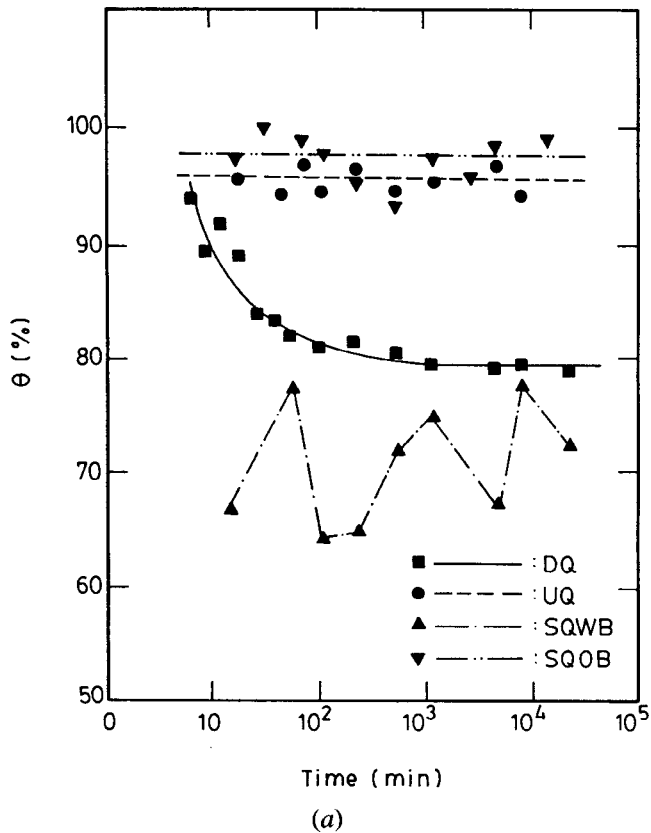


Fig. 6—Effect of the duration of room-temperature aging on (a) the shape recovery capability ratio,  $\theta$ , and (b) the yield stress,  $\sigma_y$ , of various alloy B specimens.

18 hours were also determined. All results are listed in Table II.

It is found that the  $M_s$  temperatures of various alloy A samples continuously increase with the duration of holding at room temperature. Besides, the alloy A specimen with a small value of  $M_s$  invariably corresponds to a large value of  $\sigma_y$  and a small value of  $\theta$ , as shown in Figure 4 and Table II. This result indicates that high tensile stress is always required at room temperature for a specimen having low  $M_s$  to induce the martensitic transformation; therefore, the movement of dislocations, *i.e.*, residue of plastic strain, is more easily found for this specimen. The relationship between  $M_s$  temperature and  $\sigma_y$  possibly corresponds to the Clausius-Clapeyron equation, as Warlimont *et al.* reported.<sup>[17]</sup> However, this relationship has not been quantitatively proved due to the difficulty of testing as-quenched specimens.

The possible mechanisms to cause the increase of the  $M_s$  temperature of alloy A specimen are the following: (1) external stress assisting martensitic transformation;<sup>[17]</sup> (2) thermal cycling effect;<sup>[18]</sup> (3) change of chemical composition due to the formation of solute-rich precipitates;<sup>[19]</sup> (4) increase of the  $\beta$  grain size;<sup>[20]</sup> (5) increase of the degree of ordering of the parent phase;<sup>[21]</sup> and (6) increase of the size of ordering domain.<sup>[22]</sup> In this study, no external stress has been applied to any specimen during  $M_s$  measuring procedure and no thermal cycling effect exists, because only the first martensitic transformation cycle has been conducted on each specimen to obtain the  $M_s$  temperature. Moreover, it was believed that the treatment at 100 °C for 10 minutes in either the UQ, SQWB, or SQOB process should cause neither precipitation of a second phase nor the growth of  $\beta$  grains. It has been confirmed with TEM that no precipitation is developed in specimens after room-temperature aging for 18 hours. Henceforth, the remaining possible mechanisms to cause the increase of  $M_s$  temperature of alloy A specimens are the degree of ordering and the size of ordering domain.

The TEM dark-field images with the  $(200)_\beta$  reflection spot of DQ, UQ, and SQWB alloy A specimens displayed the nearest-neighbor antiphase domains,<sup>[23]</sup> as shown in Figure 7. The sequence of size of ordering domain, SQWB > UQ > DQ, correlates well with the sequence of  $M_s$  temperatures in Table II. Rapacioli *et al.*<sup>[22]</sup> suggested that the martensitic transformation would elongate the antiphase boundaries and produce sharp boundary curvature at intersections of ordering domains. Therefore, the small domain size causes large densities of elongated boundaries and sharp curvature ones during a martensitic transformation process; thus, the low  $M_s$  temperature is observed. Table II indicates that the  $M_s$  temperatures of alloy A specimens are increased after room-temperature aging. However, no change of domain size by room-temperature aging is detected with TEM. It is suggested that the increase of  $M_s$  temperature of alloy A specimens may be due to the increase of ordering degree with decrease of the temperature of material; *i.e.*, the degree of ordering at 100 °C is smaller than that at room temperature.<sup>[14,21]</sup> The most significant decrease of  $M_s$  temperature on DQ alloy A specimens is possibly caused by the largest change of temperature

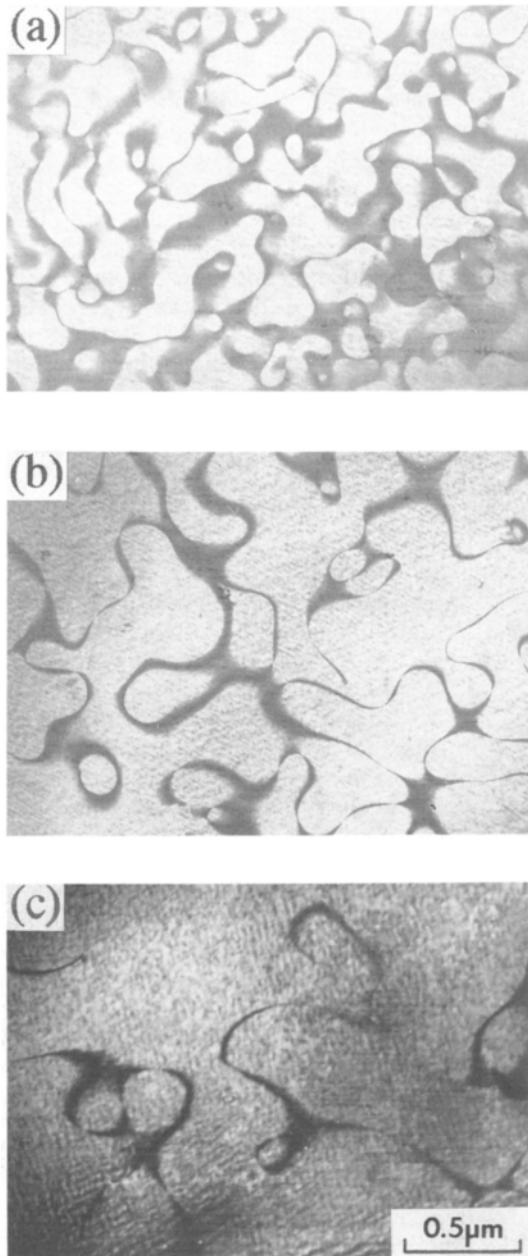


Fig. 7—The TEM dark-field images associated with the (200) diffraction spot indicate the size of nearest-neighbor antiphase domains of various alloy A specimens: (a) DQ, (b) UQ, and (c) SQWB.

(850 °C to room temperature) as well as the largest improvement of ordering degree.

Figure 7 indicates that the treatment at 100 °C for 10 minutes in the UQ process causes the growth of ordering domain by comparison with the domain size of DQ specimen. However, it cannot explain that the domain size of UQ specimen is smaller than that of SQWB samples similarly held at 100 °C for 10 minutes. Figure 8 displays the measured continuous cooling curves of those specimens through various quenching processes and media, such as water at room temperature (DQ and UQ), water at 100 °C (SQWB), and oil at 100 °C (SQOB). The slowest cooling process in a SQWB specimen is believed to be due to the rapidly formed vapor layer on the

specimen surface insulating it from the surrounding quenching medium. Therefore, it is concluded that the slow cooling rate develops the largest domain size and the greatest  $M_s$  temperature in SQWB specimens.

Table II also lists the measured  $M_s$  temperatures of alloy B specimens. The  $M_s$  temperature of the DQ specimen could not be measured due to the promotion of martensitic stabilization during the heating process for the resistance measurement, thus depressing the reverse martensitic transformation. The variations of  $M_s$  in those alloy B specimens with a sound SME property (*i.e.*, UQ and SQOB specimens) are quite similar to those of alloy A specimens. It is suggested that both domain size and degree of ordering also cause these variations. Meanwhile, it is found that the total amount of  $\alpha$  phase in SQWB specimens varies much from one to another, and no significant trend of  $M_s$  variation in SQWB specimen has been observed. It is believed that the quenching rate with a boiling water bath after solution treatment is too small to suppress the transformation of  $\alpha$  phase in Cu-Zn-Al SMA with a low content of aluminum (alloy B).

#### D. Isothermal Aging Treatment

The UQ specimens of both alloys A and B were selected for the isothermal aging treatment to eliminate the possible effects of ordering transformation, martensitic stabilization, and precipitation during the quenching process. The microstructures of all specimens after various aging treatments have been investigated; Figure 9 displays the scanning electron microscopy (SEM) metallographs of several alloy B specimens. It is found that irregular blocky precipitates and straight rod precipitates are developed in specimens after aging treated at temperatures above 450 °C and below 400 °C, respectively.

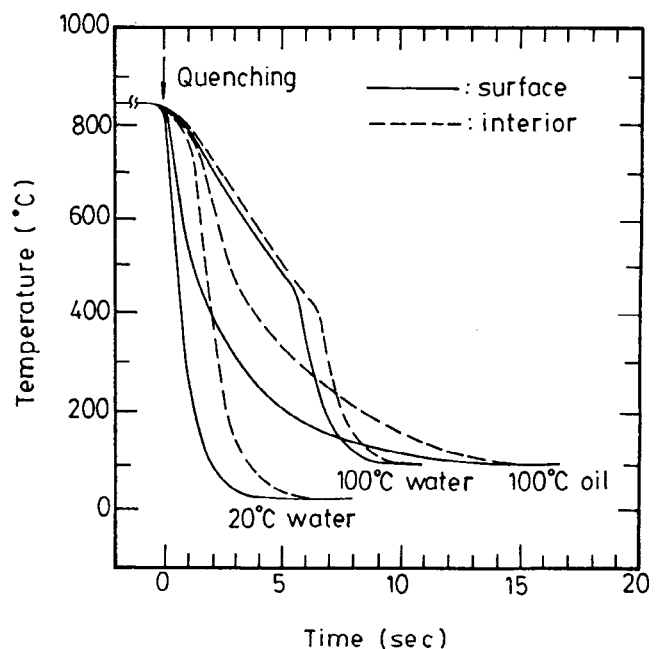


Fig. 8—Measured continuous cooling curves of specimens through various quenching processes and media.

The blocky precipitates and rod ones are identified with X-ray diffraction to be  $\alpha$  and  $\alpha_1$  phases, respectively. The T-T-T transformation diagrams of both  $\alpha$  and  $\alpha_1$  precipitations in alloys A and B have been constructed as shown in Figure 10. The required durations to form  $\alpha$  precipitates in either alloy A or alloy B are much less than those for  $\alpha_1$  ones. The rapid transformation behavior of  $\alpha$  phase in the present study is possibly similar to the massive transformation from the  $\beta$  phase to the  $\alpha$  one in a Cu-Zn alloy.<sup>[11]</sup> Furthermore, the required durations for  $\alpha$  and  $\alpha_1$  precipitations in alloy A are greater than those in alloy B. Nakata *et al.*<sup>[24]</sup> and Wu and Wayman<sup>[25]</sup> have reported that the solute contents (Zn and Al) in the  $\alpha$  and  $\alpha_1$  phases are less than those in the matrix. Because both the melting point and atomic size of aluminum exceed those of zinc, the diffusion process of aluminum becomes a dominant factor in the rate of precipitation. As alloy A has a greater content of aluminum than alloy B, alloy A specimens require a greater aging duration for the aluminum atoms to diffuse away and to form  $\alpha$  and  $\alpha_1$  precipitates. The same reason applies to the formation of  $\alpha$  phase in the SQWB alloy B specimen, but there is no  $\alpha$  phase in SQWB alloy A specimen. Besides, there are no  $\alpha$  precipitates in some previous reports<sup>[13,23]</sup> on Cu-Zn-Al SMA's after quench-

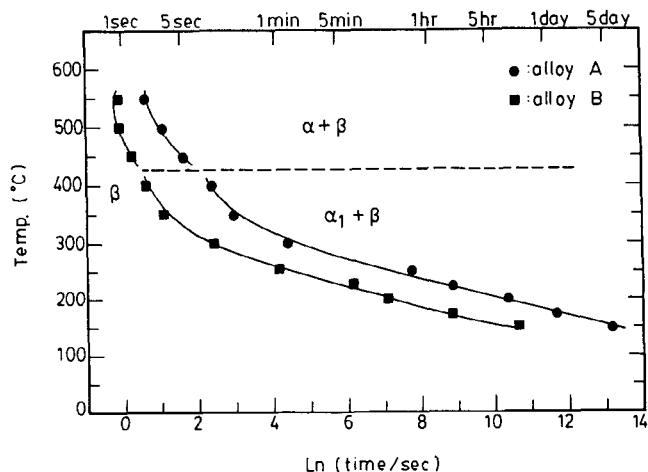


Fig. 10—The T-T-T transformation diagrams of  $\alpha$  and  $\alpha_1$  precipitations in alloys A and B.

ing in boiling water or air cooling due to the greater content of aluminum (6 to 8 wt pct) in those alloys.

It has been reported that the precipitation of either  $\alpha$  or  $\alpha_1$  phase would cause a serious deterioration in SME property. In this study, it was found that the specimens

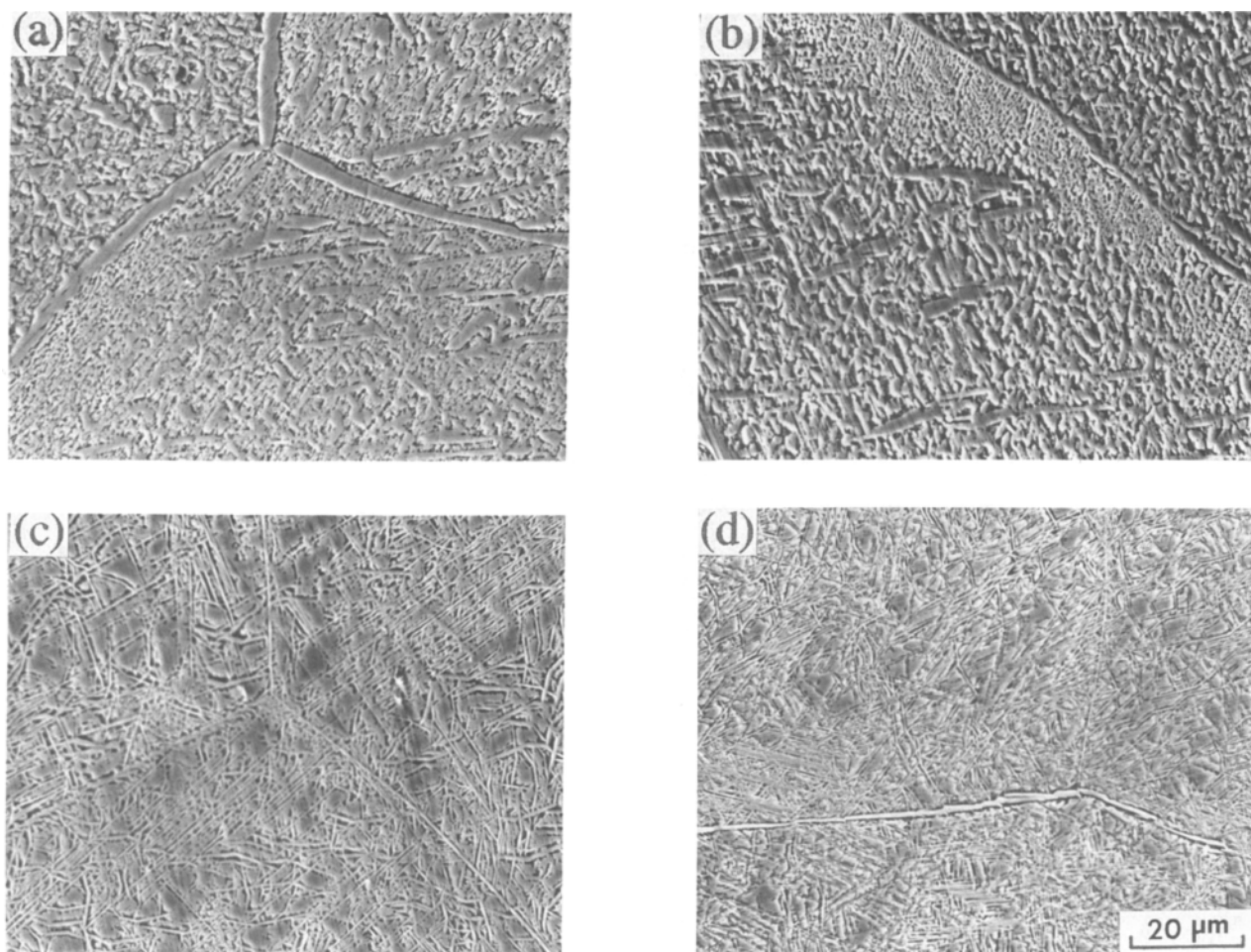


Fig. 9—SEM metallographs of alloy B specimens after isothermal aging treated at (a) 600 °C for 10 s, (b) 500 °C for 5 s, (c) 400 °C for 5 s, and (d) 300 °C for 40 s.

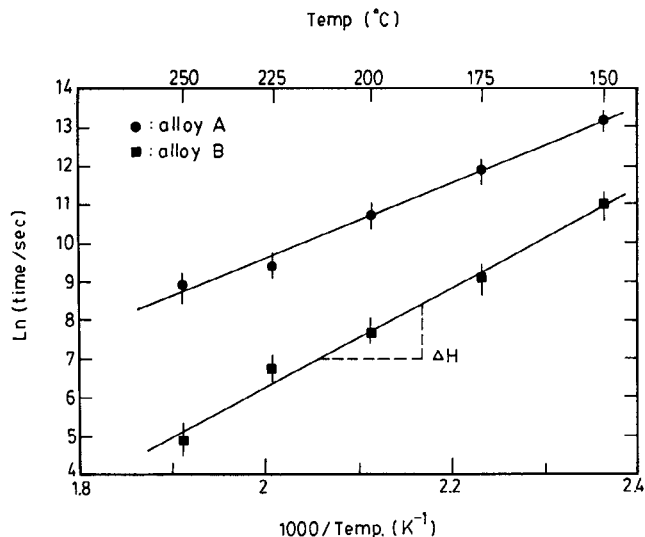


Fig. 11 — Arrhenius plots of the length of aging period vs temperature to develop a microstructure with 10 pct volume fraction of  $\alpha_1$  precipitates.

with more than 10 vol pct  $\alpha_1$  phase lost most of their SME property. Thus, the volume fraction measurement of  $\alpha_1$  precipitates with an image analyzer has been conducted, and the Arrhenius plots of the length of aging period vs temperature to develop a microstructure with 10 vol pct  $\alpha_1$  precipitates in total have been conducted, shown in Figure 11. The activation energies of  $\alpha_1$  precipitation in alloy A and alloy B are thus determined to be  $82 \pm 6 \text{ kJ} \cdot \text{mol}^{-1}$  and  $104 \pm 9 \text{ kJ} \cdot \text{mol}^{-1}$ , respectively. From the extrapolation of Arrhenius relationships to low temperature, the life expectancy of a Cu-Zn-Al shape memory device can be predicted, as shown in Table III. It is predicted that those devices made from alloy A and alloy B after total exposure at 100 °C for 131 days and 41 days, respectively, will lose their SME properties. It is concluded that the Cu-Zn-Al SMA's with a low content of aluminum are unsuitable for operation at relatively high temperature and long period working conditions.

#### IV. CONCLUSIONS

The effects of low content of aluminum and various quenching and aging treatments on two Cu-Zn-Al SMA's have been investigated. The results are summarized as follows:

1. There is a slight difference in pseudoelastic property among alloy A specimens according to the sequence SQWB > UQ > SQOB > DQ. However, the SME

Table III. Estimated Life of Cu-Zn-Al Shape Memory Alloys (in Days)

Temperature	100 °C	80 °C	60 °C
Alloy A	131	577	3030
Alloy B	41	292	2660

properties of alloy B specimens vary much. Both DQ and SQWB alloy B specimens have a poor shape recovery capability due to the martensitic stabilization effect and  $\alpha$  precipitation, respectively. Excellent and stable SME behavior is obtained on either UQ or SQOB alloy B specimens, with the value of  $\theta$  exceeding 95 pct.

2. The slowest cooling rate in the SQWB process causes the largest ordering domain size and the greatest  $M_s$  temperature of the specimens. Treatment at 100 °C for 10 minutes in the UQ process develops a moderately large sized domain and a moderately great  $M_s$  temperature of specimens according to a comparison with those of the DQ specimens.
3. The formation of irregular blocky  $\alpha$  precipitates in Cu-Zn-Al SMA with low content of aluminum at a temperature above 450 °C is rapid. A rapid quenching process after solution treatment to avoid the formation of  $\alpha$  phase is essential to assure a good SME property. Therefore, the UQ process should be better than the SQ one, especially in the SQWB.
4. A decrease of aluminum content in Cu-Zn-Al SMA accelerates the formation of both  $\alpha$  and  $\alpha_1$  phases. It is suggested that Cu-Zn-Al SMA's with a low content of aluminum are unsuitable for operation at relatively high temperature and for long periods of working conditions.

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