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Pseudoelastic Behavior and Aging Effect in Thermoelastic In-Tl Martensite

By

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Abstract

In this paper, the authors report the interesting stress-strain results and an aging behavior found in single crystals of In-21, 22, 23, 24, 25 and 26 at.% Tl alloys. The stress-strain curves can be classified into three groups according to their shapes. In a high temperature region above martensitic temperature, M_s , while at a considerably low temperature region, superelastic loops are obtained. While at an intermediate temperature below M_s , the residual strain remains after removal of the applied stress, and this strain returns to zero when the specimen is heated up to above the reverse transformation temperature. That is the so-called shape memory effect. The twin boundaries migrated by the external stress were stabilized by strain aging under stress, and the activation energy for the aging process was found to be 0.51 ± 0.03 eV. The strain rate dependence of pseudo-elastic behavior is observed in the martensite phase and is explained by the dynamic aging between the moving twin boundaries and Tl atoms.

1. Introduction

In-Tl alloys with a composition of around 20–25 at.% Tl have a hytseresis of transformation temperature of about 2 degrees, and are well known as typical thermo-elastic martensite alloys, such as Au-Cd and Cu-Al alloys. The In-Tl alloy has a fcc structure as a parent phase, and undergoes a martensitic transformation to a fct structure on cooling, accompanied by a very small amount of transformation heat $(11.13 \times 10^{-3} \text{ J/atom})$ and of volume difference. It is also known that this transformation can be proceeded by a single interface transformation, and is related to an acoustic soft mode of the $\{110\} < 1\overline{10} >$ shear, as a premartensitic stage such as in the cases of Au-Cd (Nakanishi, Mori, Miura, Murakami and

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Kachi 1973)1) and Au-Cu-Zn alloys (Miura, Maeda and Nakanishi 1974)2).

Since Burkart and Read (1953)³⁾ have reported the super-elastic behavior associated with the stress-induced transformation of an In-Tl alloy, such super-elastic behavior has also been found in many other thremoelastic martensite alloys; for example, Au-Cd (Birnbaum and Read 1960⁴⁾, Miura, Mori, Nakanishi, Mura-kami and Kachi 1976⁵⁾), Cu-Al-Ni (Rachinger 1958)⁶⁾ Ag-Cd (Krishnan and Brown 1973, Miura, Mori and Nakanishi 1973)⁷⁾, Ti-Ni (Wasilewski 1971)⁸⁾ and Au-Cu-Zn (Miura, Maeda and Nakanishi 1974)²⁾. In these alloys, detailed stress-strain relationships including the shape memory effect over a wide range of temperatures have been reported. However, in the In-Tl alloys, studies have been made on only the internal friction (De Morton 1969)⁹⁾, elastic constants (Novotny and Smith 1965)¹⁰⁾, a crystallographical analysis of the transformation (Basinski and Christian 1954)¹¹⁾, and the effect of the Tl concentration on the transformation temperature. No detailed stress-strain relations have so far been investigated, save in the work of Burkart and Read, in which the super-elastic behavior of the In-20.7 at.% Tl alloy at 346K was shown.

The present authors have reported preliminary studies on the pseudo-elastic behavior of In-21 and 22 at.% Tl, in which was shown the strain rate dependence of the pseudo-elastic behavior in the martensite phase. (Miura, Ito and Nakanishi, 1976¹²⁾, Miura, Ito, Endo and Nakanishi, 1976¹³⁾).

In the present paper, the authors report on the pseudo-elastic behavior found in In-21, 22, 23, 23, 25 and 26 at.% Tl alloys: 1) The super-elastic behavior associated with the martensitic transformation, the shape memory effect, and the super-elasticity in the martensite phase. 2) The effect of static strain aging on the super-elasticity in the martensite phase. 3) The strain rate dependence of the pseudo-elastic bheavior in the martensite phase, and its explanation by the dynamic aging effect.

2. Experimental Method

Single crystals of In-Tl alloys containing 21, 22, 23, 24, 25 and 26 at. % Tl were prepared from elements of high purity (99.99%). Preweighed amounts of the elements were melted at about 773K in selaed quartz tubes under a partial pressure of argon. The alloys grown by the Tammann method were homogenized at 413K for 24 hrs. The quartz tubes around the specimens were removed with hydrofluoric acid. The orientations of f.c.c. single crystals were determined by the Laue back-reflection method. Tensile tests were performed with an Instron type testing machine. Specimens, 2.0 mm in diameter with 30 mm gauge length, were used for the tensile testing.

3. Experimental Results

3.1. Temperature dependence of stress-strain curves and the martensitic transformation temperature

Typical examples of the stress-strain curves of In-22 at.% Tl alloy from 328K to 77K are shown in Fig. 1. The stress-strain curves can be classified into three groups according to their shapes. In the high temperature region, i.e. above 317.5K, and the low temperature region, i.e. below 232K, super-elastic loops can

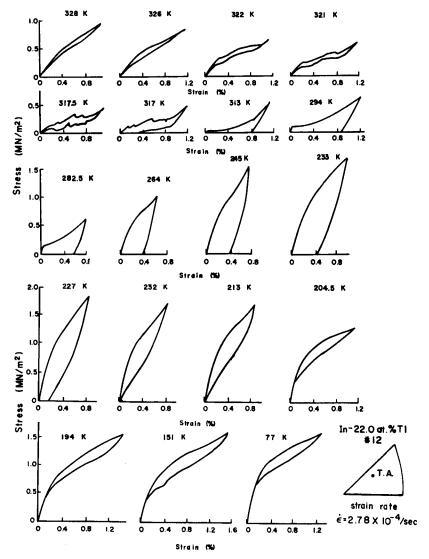


Fig. 1. Variation of stress-strain curves with temperatures in In-22.0 at. %Tl single crystal.

be seen. In the intermediate temperature region, i.e. from 227K to 317K, the removal of the stress left residual strain making ferro-elastic loops (Nakanishi, Mori, Miura, Murakami and Kachi 1973)¹⁾, and the residual strain went back to zero when the specimen was heated up to above the transition temperature. This behavior was called a "shape memory effect". It is considered that the super-elasticity observed in the high temperature region is associated with the stress-induced martensitic transformation. Furthermore, in the low temperature region it is thought to be due to the reversible movement of the internal twin boundaries in the martensite phase. The same type of stress-strain curves in the In-24.0 at.% TI single crystal is shown in Fig. 2.

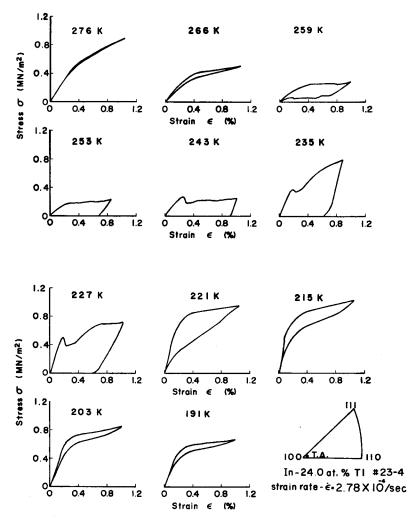


Fig. 2. Variation of stress-strain curves with temperatures in In-24.0 at. %Tl single crystal.

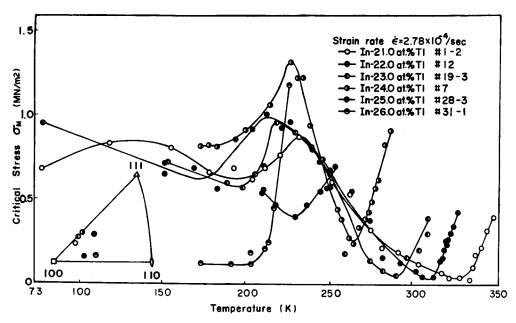


Fig. 3. Temperature variation of critical yield stress in In-21.0~26.0 at. %Tl single crystal.

Figure 3 shows the temperature dependence of critical yield stress, σ_M . It is considered that the temperature obtained by extrapolating the value of critical yield stress to zero corresponds to the starting temperature of the martensitic transformation, M_s , in Au-47.5 at.% Cd (Nakanishi, Mori, Miura, Murakami and Kachi 1973)¹⁾ and Au-Cu-Zn (Miura, Maeda and Nakanishi 1974)²⁾. These values were taken for the In-21, 22 and 23 at.% Tl and the temperatures at which critical stress was minimum were taken as M_s for the In-24, 25 and 26 at.% Tl. The results are shown in Fig. 4 and agree with the results obtained by Guttman (1950)¹⁴⁾, Meyerhoff and Smith (1963)¹⁵⁾, and Patel and Ahmed (1978)¹⁶⁾.

3.2. Strain rate dependence of pseudo-elastic behavior

The strain rate dependence of the pseudo-elastic behavior in the martensite phase of the In-22 at.% Tl alloy at 221K is shown in Fig. 5. With a low strain rate, the residual strain remained, but an increase in the strain rate resulted in super-elastic or rubber like loops. Therefore, the higher the strain rate, the higher the recoverable ratio attained. This effect may correspond to a phenomenon where the maximum peak position in Fig. 3 moves to a higher temperature. This is shown in Fig. 6.

3.3. Strain aging in parent and martensite phase

Figure 7 shows the change in stress-strain curves in an In-21.0 at. % Tl alloy,

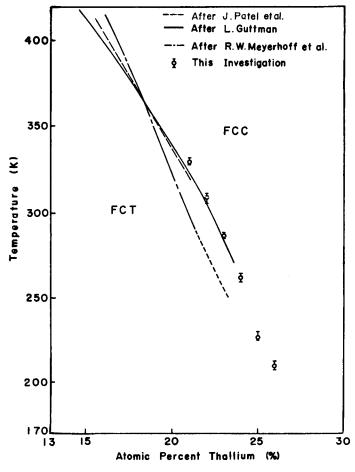


Fig. 4. Phase diagram of In-Tl alloys.

due to strain aging at 217K in the martensite phase and at 342K in the parent phase. The deformation temperature 217K is the upper critical temperature at which a super-elasticity in the martensite phase with a strain rate of 2.78×10^{-4} /sec can be obtained. At 217K in the martensite phase, the stress-strain curve changes to a super-elastic loop with a large stress hysteresis after aging for 3 min. at A. Upon reloading the residual strain is left after aging for 5 min. at B, but this residual strain returns to zero when heating the sample above the A_f temperature representing the shape memory effect. At 342K in the parent phase, the stress-strain curve shows a super-elastic loop without any change in the stress hysteresis after aging for 3 and 5 minutes. However, when the aging time is increased 10 or 30 min. residual strain occurs, but this residual strain cannot be recovered by heating the sample above the A_f temperature. This strain is considered to be a

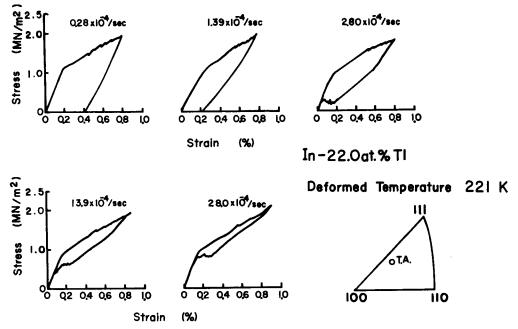


Fig. 5. Effect of strain rate on stress-strain curve in In-22 at. %Tl single crystal at 221K.

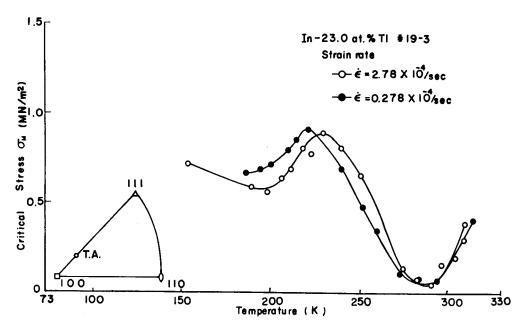


Fig. 6. Effect of strain rate on the temperature variation of critical yield stress in In-23.0 at.%Tl single crystal.

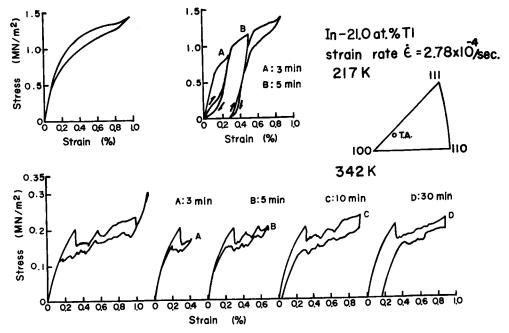


Fig. 7. Effect of strain aging on stress-strain curves in In-21.0 at.% Tl single crystal at 217 ± 0.5 K and 342 ± 0.3 K in martensite phase and parent phase respectively.

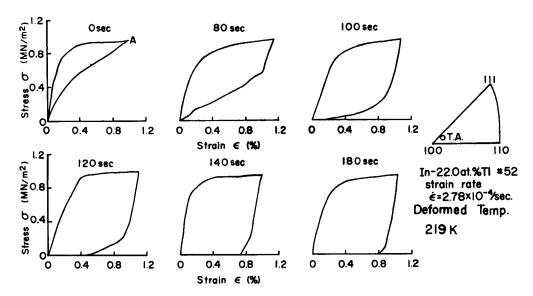


Fig. 8. Effect of strain-aging on stress-strain curves in In-20 22.0 at % Tl single crystal deformed at $219\pm0.5K$ in the martensite phase.

permanent strain introduced by creep deformation during aging under stress. The change in the stress-strain curve due to aging with time was investigated on In-22.0 at.% Tl at 213 and 219K. The result obtained at 219K is shown in Fig. 8. The specimen was strained under tension at 219K by 1% strain (point A), and was then kept at that stress for a time. After the aging, the stress was released to zero. The specimen shows a super-elastic loop with the aging within 100 sec. However, when, the aging exceeds 100 sec., residual strain was left upon unloading. However, this residual strain returns to zero upon heating above the A_f temperature, showing a shape memory effect.

In order to determine the activation energy for the aging process, the stress values, σ_0 , $\sigma_{t=0}$ and σ_t are established from the stress-strain curves as shown in Fig. 9, where σ_0 corresponds to the 1% strain, $\sigma_{t=0}$ the reverse yield stress when the aging time is zero, and σ_t that after being kept for t seconds. When the function $f=(\sigma_t/\sigma_0)/(\sigma_{t=0}/\sigma_0)$ was plotted against aging time, two curves were obtained. These were aged at 213 and 219K respectively, and are shown in Fig. 9.

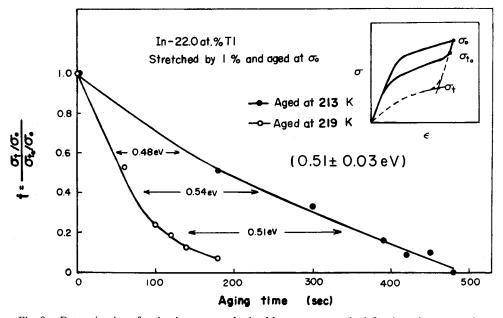


Fig. 9. Determination of activation energy obtained by cross-cut method for the aging process in In-22.0 at.%Tl single crystal.

Assuming that the aging phenomenon is caused by a single activation process, the fraction can be represented by

$$f = t \cdot \exp\left(-E/kT\right) \tag{1}$$

Here, E is the activation energy for the migration process, k the Boltzmann's constant, t the aging time and T the deformation temperature. From equation (1),

$$\ln(t_1/t_2) = (E/k)(1/T_1 - 1/T_2) \tag{2}$$

can be obtained. Since each aging time t_1 and t_2 at each temperature T_1 and T_2 in the case of f=0.6, 0.4 and 0.2 can be obtained from Fig. 9, the E values obtained from equation (2) were found to be 0.48, 0.54 and 0.51eV at f=0.6, 0.4 and 0.2, respectively. The average value, $E=0.51\pm0.03$ eV, was obtained for the aging process. This value agrees satisfactorily with E=0.67 eV, obtained from the measurement of the internal friction (De Morton, 1969)⁹⁾, and is considered to be adequate for the migration of the Tl atom as will be discussed later.

4. Discussion

4.1. Temperature dependences of stress-strain behavior and critical stress

The super-elasticity observed in a high temperature region above M_s temperature is associated with the stress-induced martensitic transformation, and that at a low temperature region is thought to be due to the reversible movement of the internal twin boundaries in the martensite phase. The super-elasticity observed in the martensite phase has accordingly been called "rubber like behavior".

In the Au-47.5 at.% Cd alloy, the super-elasticity was found to be associated with the reversible movement of the internal twin boundaries in the martensite, but this was observed only after aging the specimen at room temperature for about one day, and was not observed immediately after the transformation. In the case of Au-49 at. % Cd alloy, it was found that the super-elasticity in the martensite phase was due to the stress-induced transformation of thermal martensite to the other crystal structures of martensite. In the present investigation, it is considered that in the In-Tl alloy, the original twin boundaries in the martensite are stabilized without aging due to their perfect thermo-elastic nature or high diffusibility of Tl Below about 230K, the twin boundaries return to their original positions upon unloading, thus showing super-elasticity. With an increase of deformation temperatures from M_s , an increase of yield stress and the disappearance of the clear yielding and also a jerky flow in the plateau region following the yield point were observed. In the intermediate temperature range, from M_s to about 230K, the shape memory effect in which the residual strain recovers by heating appeared. The yield stress in this range increased with a decrease in the temperature, and was maixmum at the transition region between the ferro-elastic and super-elastic

region of the martensite phase, as shown in Fig. 3. In the In-Tl alloy, the superelastic strain at the plateau region associated with the stress-induced martensitic transformation was found to be very small compared with other thermo-elastic alloys. This was due to the smallness of both the transformation strain and the axial ratio in the tetragonal crystal of martensite. In the case of Au-Cd alloys below M_s , the negative coefficient, $d\sigma/dT < 0$, was not observed, but the critical stress was constant, save in the case when the negative coefficient was obtained only with the formation of new SIM from the thermal martensite. On the other hand, in the case of In-Tl, the critical stress increased with a decreasing temperature below M_s , experienced a maximum and then decreased. One must also carefully note the fact that the temperature range around 230K where the maximum, σ_c , was obtained, appears to be almost independent of the Tl content, while the M_s point was largely dependent upon the Tl content.

The reason for the peculiar behavior of critical stress below M_s may be considered to be as follows: Novotny and Smith (1965)¹⁰, Pace and Saunders (1972)¹⁷ have found extremely small values of the shear elastic constant, C', in cubic phase and C'_M in the tetragonal phase near the transition temperature. The values of C' and C'_M correspond to the resistances against the transformation shear to the martensite phase and the twinning shear in the martensite, respectively. Therefore, it can be understood that the critical yield stresses in both fcc and martensite phases increase when the deformation temperature departs from the transition temperature. The value of the maximum stress σ_c increased with in increase of the Tl content. This, plus the peak temperature moving to a higher temperature on increasing the strain rate (Fig. 6) suggest that interaction between moving the twin boundary and the diffusion of the Tl atom is strong in the vicinity of the maximum, peak temperature at around 230K, where the resistance against the movement of the twin boundary, σ_c is quite large.

The temperature dependence of the critical stress above M_s can be explained by the Clausius-Clapeyron formula:

$$\frac{d\sigma_{M}}{dT} = \frac{\rho}{\Delta \varepsilon} \cdot \frac{\Delta H}{T_{0}} \tag{3}$$

Here, σ_M is the critical stress for inducing the martensite, $\Delta \varepsilon$ is the amount of strain due to transformation, ΔH is the heat of transformation and ρ is the density of the crystal. From the result of Fig. 3, the values of $d\sigma_M/dT$ and T_0 were obtained. Also, the values $\Delta \varepsilon$ were taken from the stress-strain curves, and then ΔH was calculated from equation (3). The results are shown is Table 1.

	In-21% [T1 #1-2	In-22% T1 #12	In-23% T1 #193	In-24% T1 #7	In-25% T1 #28-3	In-26% T1 #31-1
Δε (%)	0,95	0.95	0.55	0.44	0.61	0.63
$(d\sigma_{\mathbf{M}}/dT) \times 10^{-3}$ $(MN/m^2 \cdot K)$	25.0	23.0	19.0	34.0	12.6	78.8
T_0 (K)	333	311	290	265	230	211
<i>∆H</i> (<i>J</i> /mol)	12.03	11.07	24.35	16.47	13.10	37.89

Table 1. Amount of transformation heat in 1n-Tl single crystals.

These values were compared with the ΔH measurements of Patel and Ahmed (1987), and are shown in Fig. 10. It is interesting to note that for both sides of T1=25 at.% of non-stoicheometllic compositions, the slope was found to be different in our case.

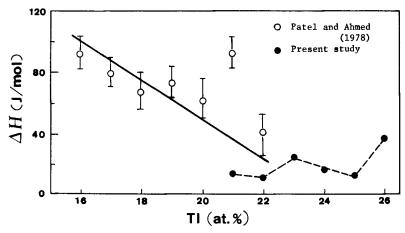


Fig. 10. H vs. alloy composition.

4.2. Strain rate dependence of pseudo-elastic behavior and aging effect

In Fig. 8, it is shown that in the intermediate temperaturees, a rubber-like behavior changes gradually, and is found to be time dependent. A specimen returns to its original position if the stress is released rapidly, but remains in the deformed position if kept at the stress interrupted for a time. The activation energy was found to be 0.51 ± 0.03 eV, and it is clear that some diffusion-controlled process occurs in this intermediate temperature range which stabilizes the displaced twin configuration. De Morton (1969)⁹⁾ has pointed out that the so-called Zener relaxation peak, due to the reorientation of Tl atoms in the In-matrix, was observed at about 273K. Also, another internal friction peak observed immediately

below M_s was considered to be due to the relaxation associated with the growth or coalescence of internal twins. Furthermore, some process, which is not simply the diffusion of the Tl in the In-matrix (the activation energy being 0.67 eV), will probably be associated with the interaction between the coalescencing twin boundaries and the Tl atoms, since it requires a higher activation energy of $1.08 \sim 1.26$ eV. In the case of the present experiment, the activation energy 0.51 eV obtained is rather small compared with 0.67 eV, but is considered to be the activation energy for the diffusion of the Tl atom under stress. Quite below 230K, the twin boundaries can move independently of Tl atoms, because the diffusion of Tl atoms is more difficult, and the observed rubber like recovery may be due to the restoring force from the Tl atom distributed orderly in the martensite.

4.3. Kinetics of pseudo-elasticity associating with a dynamic interaction between the moving twin boundary and the diffusibility of Tl atoms

Miura, Takamura and Yamashita (1968, 1973)^{18,19)} have determined from the analysis of the critical condition for the serration due to the Portevin-LeChatelier effect that the effect is caused by an interaction of oxygen atoms or nitrogen atoms with moving dislocations. They also determined the critical condition of this effect from the deformation temperatures and strain rates. In the present study, we have attempted to determine the critical condition for the transition from superelasticity to shape memory phenomenon using the dynamic strain-aging technique.

If we take the number of twins as N, $\Delta \varepsilon$ the strain due to the twinning shear, the propagation speed of the twinning interface V_T can be represented as

$$V_c = N \cdot V_T \cdot \Delta \varepsilon \tag{4}$$

where V_c is cross-head speed;

and if 1 is the specimen length, equation (4) can be written

$$V_c/1 = (N/1)V_T \cdot \Delta \varepsilon \tag{5}$$

and

$$\dot{\varepsilon} = (N/1) V_T \cdot \Delta \varepsilon \tag{6}$$

 $\Delta \varepsilon$, the strain due to twinning deformation, is

$$1 + \Delta \varepsilon = (1 + S^2 \cos \phi_0 + 2S \cos \phi_0 \cos \lambda_0)^{1/2} \tag{7}$$

where S is the shear strain due to twinning, ϕ_0 is the angle between the tensile axis and the normal of the twinning interface, and λ_0 is the angle between the tensile axis and the direction of the twinning shear on the twinning interface.

Burkart and Read (1953)³⁾ reported that the twinning plane is parallel to {110} and the direction of the shearing deformation is $\langle 1\overline{1}0 \rangle$. For example, specimen #12 of our investigation has the values of $\phi_0=40.5^\circ$, $\lambda=50.5^\circ$ and $\cos\phi_0=0.766$, $\cos\lambda_0=0.636$. S can be given by Basinski and Christian (1954)¹¹⁾ as

$$S = (c/a) - (a/c) = (c^2 - a^2)/ac \simeq 2(c/a - 1)$$
(8)

where a and c are lattice constants of the In-Tl single crystals. If we take the values obtained by Gutterman (1950), c=4.855Å, a=4.688Å and c/a = 1.0356 in In-20.75 at.% Tl alloy at room temperature, we obtain S=0.0712 in our case. From Eq. (7) we obtain the value d = 3.5%. However, it was reported that the ratio of the A and B twin bariants is 2:1 (Burkart and Read, 1953). From our result in Fig. 11, if we consider the whole region of specimen, the A and B twin lamellae can be considered to be about 50% for each. Therefore, the twinning strain due to the shear of the internal twin can be estimated as $3.5 \times \frac{1}{2} = 1.75$ %, which closely agrees with the maximum strain of deformation of the martensite phase, max 1.6%, of In $21.0 \sim 26.0$ at.% Tl martensites, as shown 151K in Fig. 1. On the other hand, the migration velocity V_i of the solute (Tl) atom is given by the Einstein relation:

$$V_i = FD/kT = (F/kT)D_0 \cdot \exp(-E/kT) \tag{9}$$

Here, F is the interaction force of solute atoms with twin boundaries, D_0 the diffusion constant and B the activation energy for the diffusion of solute atoms.

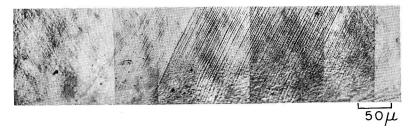


Fig. 11. The internal structure at 293K in the thermal martensite phase in In-22.0 at.%Tl single crystal.

Since whether the residual strain remains or not is dependent on the dynamic interaction of the moving twin boundaries with solute atoms, the critical condition, that the migration velocity of twin boundaries, V_T , becomes nearly equal to that of the Tl atoms, may be realized when the diffusion velocity of the Tl atoms increases sufficiently.

This condition may be given as

$$V_T = \eta \cdot V_i \tag{10}$$

where η is a constant. From the above equations (6), (9) and (10),

$$\dot{\epsilon}T = (\eta \cdot F \cdot N \cdot \Delta \epsilon \cdot D_0/kl) \cdot \exp(-E/kT) \tag{11}$$

is obtained. Since η , F, N, $\Delta\varepsilon$, D_0 , k and 1 are the constants, if $\ln(T\dot{\varepsilon})$ vs. 1/T relation represents a straight line, the slope gives the activation energy for the diffusion of the Tl atoms. As shown in Fig. 12, when the border line between a super-elastic region (solid circles) and a shape memory region (open circles) can be drawn by using the activation energy 0.51 ± 0.03 eV obtained by the static strainaging (Fig. 9), the lines satisfactorily indicate the actual critical condition shown by the equation (11). The region indicated by the solid circles above M_s exhibits the super-elastic region associated with the stress-induced martensitic transformation.

Using the activation energy for the diffusion of the Tl atoms, the number of

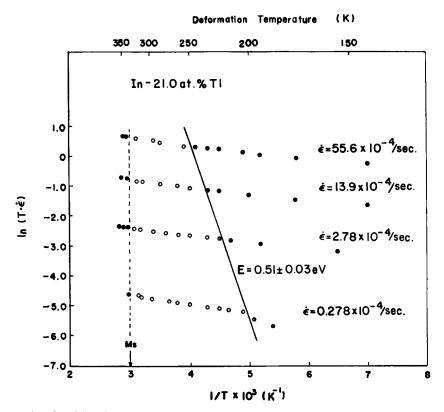


Fig. 12. Critical condition of the deformation temperature and strain rate required for the shape memory effect o in In-21.0 at.%Tl single crystal. (●: Super elasticity, ○: Shape memory region)

mean jumps of the Tl atoms to reach the twin boundaries is represented by

$$j = t \cdot \nu \cdot z \cdot \exp\left(-E/kT\right). \tag{12}$$

Here, t is the aging time, ν the Debye frequency ($\approx 10^{13}/\text{sec}$) and z the numbers of the nearest-neighbour atoms (=12). The mean migration distance, l, of Tl atoms can be represented as

$$l = \sqrt{j} \cdot a \,, \tag{13}$$

where a is a lattice constant.

As a result, the mean migration distance calculated appears to be rather small, $l=0.1~\mu\text{m}$, compared wit the twin-width ($\approx 1.5~\mu\text{m}$ in Fig. 11). This suggests that the dynamic contribution of the interaction force is confined to a short distance from the twin boundaries.

5. Conclusion

We reported some interesting stress-strain behaviors found in the In-Tl binary single crystals having 21~26 at.% Tl. The kinetics of super-elastic stress-strain loops which appear in the lower temperature martensitic phase and an aging effect of Tl atoms on the shape memory phenomena are investigated. Experimental results obtained were as follows: i) In the temperature range above A_t , super-elastic loops, which are associated with the formation of stress-induced martensite, were obtained. ii) Between M_s and about 220 K (-50°C), residual strains were left on the removl of applied stress, and these strains disappeared when the specimen was heated up to above A_f (shape memory effect). iii) Below about 220K, the stress-strain curve behaved super-elastically (or rubber-like) again. iv) A maximum of the critical stress was found around 220K, and the peak moved to a higher temperature with increasing strain rate. v) The activation energy for the diffusion of the Tl atoms, 0.51 ± 0.03 eV, was obtained by the static strainaging technique, and this value agreed well with that obtained in the dynamic strain-aging relation, $\ln(T\dot{\epsilon})$ vs. 1/T, where $\dot{\epsilon}$ is the strain rate and T the tempperature. A straight line obtained from the above relation in fact represented the boundary line between the super-elastic region and the shape memory region in the martensite phase. vi) The super-elastic behavior appearing in the In-Tl alloys was compared with a rubber-like behavior, so far found in the stabilized martensite in Au-47.5 at. % Cd and Au-Cu-Zn alloys.

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