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DISLOCATION CLIMB AND DETERMINATION OF STACKING FAULT ENERGIES IN Al AND Al-1% Mg

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ABSTRACT

Quenching and annealing experiments on bulk specimens and hot stage investigations of climb of loops in thin foils have been made on Al-1%Mg, and the results compared to those for pure aluminum. One percent Mg in solid solution has no effect on the type of loop produced after quenching nor on the precipitation of vacancies after subsequent aging. As in pure aluminum, quenching stresses determine whether Frank or perfect loops are formed in the alloy. Magnesium has little effect on the climb rate of loops, the activation energy being in the range 1·2 - 1·28 eV, which are similar to the values obtained by identical experiments on pure aluminum. It is concluded that the binding energy vacancy-magnesium atom is extremely small.

The stacking fault energies were estimated by comparing the rates of climb of large Frank and perfect loops in the same area of the foil so as to minimize the errors that arise if only the climb of Frank loops is measured. The stacking fault energies were determined to be 190 ± 10 ergs/cm^2 for Al-1%Mg and 210 ± 10 ergs/cm^2 for pure Al.

I. INTRODUCTION

The annealing of quenched-in vacancies in FCC metals and alloys has received much attention during the past few years,[1-17] and many of the important results have been described in a recent book.[5] It is now known that the type of dislocation defect formed in quenched metals as a result of vacancy condensation is much influenced by the quenching conditions (see Ref. 4 for review). For example, in aluminum the production of imperfect Frank loops ($\vec{b} = \frac{a}{3} < 111 >$) or perfect loops ($\vec{b} = \frac{a}{2} < 110 >$) can be controlled by controlling the amount of plastic deformation occurring during the quenching operation.[10-12] This result indicates that stacking fault energy and/or impurity effects may not be as important in determining the type of loop as has been thought.[8]

Besides providing information on the nature and geometry of dislocation loops, electron microscopy techniques can be used to determine the kinetics of climb processes by annealing thin foils in situ (i.e., by hot-stage electron microscopy). In the case of pure aluminum, loops are always observed to shrink during heating above 150°C.[3] The rate of shrinkage at various temperatures enables an estimate to be made of the activation energy for loop climb.[3,16,18,19] In the case of aluminum alloys, provided both solute atoms and vacancies are in supersaturation, it has been observed that loops can grow during annealing of thin foils indicating that large vacancy supersaturations can remain after quench-aging.[16,18]

The kinetics of climb in such alloys are different from that in pure aluminum, since the solute-vacancy binding energy may perturb both the energy of formation of vacancy E_f and its migration energy E_m.[16]

In an Al-5% Mg alloy, the activation energy for loop growth was determined to be 0.95 eV[16] as compared to 1.2 - 1.28 eV for that of pure aluminum.[3] Eikum and Thomas suggested that a complex, large "binding energy" exists in supersaturated Al-Mg alloys,[16,18] a result confirmed by other workers.[14,15] It also appears that the climb kinetics vary with %Mg.[16,18] However, the recent equilibrium measurements of Beaman and Simmons[17] have shown that the true binding energy, vacancy-magnesium atom, in Al-1.1%Mg is very small (< 0.01 eV). It is important, therefore, to investigate why the results for different Al-Mg alloys are so widely different.

The aim of this investigation was two-fold. Firstly, it is necessary to determine whether or not the supersaturation of solute atoms is important in controlling the climb processes and whether supersaturated solute atoms affect the values of the apparent binding energy. In order to do this, an alloy of Al-1% Mg was prepared (the solubility limit of Mg in Al is ~ 2% at 20°C). In addition, the effect, if any of 1% Mg on the nature of the dislocation loops was investigated by quenching experiments similar to those described by Das and Washburn.[12]

Secondly, an estimate of stacking-fault energy can be made by comparing the rates of shrinkages of perfect and imperfect loops at the same temperature. This method is less subject to error than that using only one type of climbing loop.[19] Parallel experiments were done on pure aluminum in order to make a comparison with Al-1% Mg alloy under the same conditions.

II. EXPERIMENTAL PROCEDURE

Standard methods were followed in the preparation of the alloy and thin foils. The final analysis of the alloy is given in Table I. The aluminum used was of 99.999% purity.

Table I. Chemical analysis of the Al-1% Mg alloy used in this investigation

Alloy	Mg	Cu	Mn	Fe	Si
Al-1% Mg	1.01%	< 100 ppm	< 100 ppm	< 200 ppm	< 200 ppm

Thin foil annealing experiments were carried out using a Siemens heating stage in the Siemens Elmiskop Ib microscope. A 12 volt storage battery or a constant voltage power supply served as a power source. Current to the platinum furnace was regulated through a variable resistance and the temperature was read from a predetermined calibration curve. The temperature is known only to within ± 10°C, because of beam heating effects. The latter is minimized by operating at the lowest possible beam currents and by taking the usual precautions.[19] The climb of loops was recorded using a Paillard-Bolex H1b movie camera driven at 12 frames/sec. A Bell and Howell f 0.95 lens, fully open, was used. Tri-X movie films were found to be the most suitable for the dynamic experiments.

Since the Siemens hot-stage is non-tiltable, the same diffraction conditions were maintained while heating the foil by tilting the gun. This eliminates any change in size of the loop due to variation in diffraction condition, since the image of a dislocation line falls to one side or the other of the true position, depending upon whether $(\bar{g}.\bar{s})$ is positive or negative. A discussion of these and other factors related to high temperature electron microscopy is given in ref. 19.

III. EXPERIMENTAL RESULTS AND INTERPRETATION

1. Effect of Aging Treatments on Vacancy Precipitation

B lk annealing of 8 mil thick specimens quenched from 545°C into oil (at 20°C) was carried out at room temperature for 24 hrs and at 45°C, 175°C and 200°C for 5 min each (see Table II). All specimens were preaged at 20°C for 10 min after quenching and before bulk annealing. Thin foils were prepared from bulk annealed samples by electropolishing. Typical micrographs are shown in Fig. 1.

It can be seen that loops are formed at random showing no evidence for heterogeneous precipitation, i.e., no helices or climb sources are observed.[4,5] The vacancy concentration C_v' can be calculated from these micrographs as follows: If N is the number of loops per cm^3 and r the radius, then

$$C_v' = \pi r^2 b N \qquad (1)$$

where $\bar{b}$ = Burger's vector [$= \frac{a}{2} < 110 >$ for a perfect loop and $\frac{a}{3} < 111 >$ for a Frank loop].

In using this formula, the thickness of the specimen was assumed to be 3000A in all cases, based on determinations from slip traces, in some foils, after prolonged observation. According to the visibility criterion, loops will be either visible or invisible according to whether $\bar{g} \cdot \bar{b}$ is zero or non-zero. All the micrographs in Fig. 1 are in [001] orientation. Assuming equal probability of formation of all possible loops ($\bar{b} = \frac{1}{2} < 110 >$ or $\frac{1}{3} < 111 >$) those not visible due to contrast effects can be accounted for.

The vacancy concentrations C_v' calculated using Eq. (2), are listed in Table II. These values include corrections for those loops which are invisible. It should be noted that unlike Al-5% Mg alloys,[16] the values

Table II. Data on loop density and vacancy concentrations obtained after quench-aging treatments on Al-1% Mg.

Alloy Al-1% Mg treatment	Loops/cm^3	Average dia. Å	C'_v
A. Quenched in oil at 20° from 550°C			
(i) as-quenched	~4 X 10^{13}	1000Å	0.31 X 10^{-4}
Bulk-annealed at			
(ii) 45°C, 5 min.	2 X 10^{13}	1200Å	0.4 X 10^{-4}
(iii) 175°C, 5 min.	6.3 X 10^{12}	1500Å	0.28 X 10^{-4}
(iv) 200°C, 5 min.	4.3 X 10^{12}	1800Å	0.28 X 10^{-4}
B. Water quenched 550°C annealed at room temp. 24 hrs.	8.4 X 10^{13}	750Å	0.8 X 10^{-4}

obtained are insensitive to the aging treatment.

Using the equation $C = A \exp[(E_f)/kT]$ for Al-1% Mg alloy and taking E_f as 0.76 and A = 1 as in the case of pure aluminum, (which appears to be valid in view of Beaman and Simmons results[17]) we obtain C'_v ~0.2 X 10^{-4}. This value is of the same order as those listed in Table II. This clearly indicates that soluble Mg atoms appear to have little effect on the total vacancy concentration and, hence, on the energy of formation of a vacancy.

The results of Table II also indicate that no supersaturation of vacancies exists in this particular alloy after quenching. Comparison of the above results with those obtained for concentrated Al-Mg alloys by Eikum and Thomas[16,18] shows the following. In Al-5% Mg alloy, C'_v increased from 10^{-5} to 10^{-3} by bulk annealing a quenched specimen at 180°C for 5 min. In the present case no such increase was observed. It is suggested, therefore, that the ratio: $\frac{\text{\% supersaturated solute atoms}}{\text{\% vacancy}}$ determines whether or not vacancy supersaturations can exist in a specimen.

2. Effect of Quenching Conditions on Loops in Al-1% Mg

Investigations of specimens quenched at different rates (e.g. by quenching in different media or changing specimen thickness)[12] showed that for minimum plastic deformation during quenching, the predominant defects were imperfect (Frank) loops. This is illustrated in Fig. 2. It can be seen that the number of Frank loops is much greater after oil quenching than after water quenching. Frank loops are recognized readily from the existence of extinction fringe contrast and different kinds of large loops can be identified as described by Bell and Thomas.[20] Water quenching results in more plastic deformation due to the higher quenching stresses. Thus, our results confirm those of Das and Washburn[12] in that the formation of perfect loops by dislocation reactions, such as $\frac{a}{3}[111] + \frac{a}{6}[11\bar{2}] \rightarrow \frac{a}{2}[110]$, is stress aided. Furthermore, the presence of 1% Mg does not modify this result.

The following conclusion can also be inferred from Fig. 2. For fast quenches (into water) the loop nucleation rate is higher than for slow quenches (compare b with c and d) and so after water-quenching the stress fields from close neighboring loops may also assist in the transformation of a Frank to a perfect loop.

Cotterill and Segall[8] have reported that in the case of 99.98% pure aluminum, Frank loops were seen only after repeated quenchings. In the case of high purity aluminum, very large Frank loops were commonly seen after a single quench. In the case of the present alloy, Frank loops were seen after a single quench even though there is 1% Mg present. A possible explanation for this behavior can be given as follows. If the impurities are insoluble, they could act as sites for heterogeneous nucleation of vacancy clusters. Since, however, 1% Mg dissolves completely

in aluminum, magnesium atoms may not produce any heterogeneous precipitation of vacancies. It follows from the present results that magnesium cannot lower the stacking fault energy of aluminum sufficiently to allow easy conversion of a Frank to a perfect loop. This conclusion is supported by our measurements of the stacking fault energy (section 3). Thus, the quench-aging behavior of Al-1% Mg and pure aluminum is very similar.

3. Activation Energy for Loop Climb and Stacking Fault Energy

Figures 3 and 4 are sequences from movie films showing the shrinkage of Frank loops A and perfect loops B in Al-1% Mg and Fig. 5 is a similar sequence for pure aluminum. Silcox and Whelan[3] were the first to study annealing of loops in pure aluminum. They concluded that loops shrink by emitting vacancies which then migrated to nearby sinks, e.g., free surfaces and grain boundaries.

Silcox and Whelan obtained the equation for the rate of shrinkage of perfect circular loops lying on (111) plane, from Friedel's theory[21] of jog motion, as follows:

$$\frac{dr}{dt} = -\frac{1}{2} Z \nu_a b \exp\left(-\frac{E}{kT}\right)\left[\exp\left(\frac{Fcb^2}{kT}\right) - 1\right] \qquad (2)$$

where Z = 11 (the atomic coordination number of the jog site); ν_a is the atomic frequency factor ($\sim 10^{13}$); $\bar{b}$ is the Burger's vector; E is the activation energy for self-diffusion; kT is the Boltzman temperature factor and F_c is the force due to the line tension of the loop. From this equation, the parabolic relationship between loop radius and time can be obtained:

$$r = r_o (1 - t/\tau) \qquad (3)$$

where $$\tau = \tau_o \exp\left(\frac{E}{kT}\right)$$

$$\tau_o = \frac{r_o^2}{Z\nu_a b^2 \alpha}$$

where α is a temperature-dependent parameter.[3] Following the method of Silcox and Whelan, activation energies for loop climb in Al-1% Mg were determined from radius-time plots at various temperatures, such as those shown in Figs. 5 and 6. The resultant values are given in Table III. It can be seen that the activation energies for Al-1% Mg lie in the range 1.20 - 1.28 eV, which is the same as that reported for pure aluminum by Silcox and Whelan using identical methods.[3] Furthermore, our results for pure aluminum (Fig. 7) also give the activation energy as 1.28 eV (at 210°C).

The estimation of stacking fault energy from hot stage experiments is best done by comparing, in the same area of the foil, the rates of shrinkage of perfect and imperfect loops at the same temperature.[19] For a circular loop, enveloping the stacking fault, the loop energy E_I is given by

$$E_I = \frac{Gb^2}{4\pi(1-\nu)} \ln\left(\frac{r_o}{b}\right)\left(2\pi r + \pi r^2 \gamma\right) \qquad (4)$$

where γ is the stacking fault energy and the other symbols are as defined earlier. $|b|$ is 2.36Å for a Frank loop in Al. It can be seen that Eq. (2) can be modified for the shrinkage of an imperfect loop as follows. The stacking fault energy γ will assist the climb in addition to the climb force F_c due to the line tension, thus

$$\left(\frac{dr}{dt}\right)_{imperfect} = -\frac{1}{2} Z \nu_a b_i \exp\left(-\frac{E}{kT}\right)\left[\exp\frac{(F_c+\gamma)b_i^2}{kT} - 1\right] \qquad (5)$$

Table III. Results obtained for the activation energies for loop climb and stacking fault energies

Temp. (±10°)°C		Activation energy E in eV	Stacking fault energy, γ in ergs/cm^2
Al-1%Mg	180	1.21	--
	210	1.28	190
	220	1.21	195
	260	1.20	--
Al	210	1.28	210

For loops of the sizes observed in this work, the energy due to the fault far exceeds that due to the dislocation line tension, and hence, we can neglect F_c in comparison with γ in Eq. (5). This assumption is valid for the initial stages of shrinkage (large loops). With this approximation and from Eqs. (2) and (5) we obtain

$$\frac{\left(\frac{dr}{dt}\right)_{imperfect}}{(dr/dt)_{perfect}} = \frac{b_i}{b_p} \; \frac{\exp\left(\frac{\gamma b_i^2}{kT}\right) - 1}{\exp\left(F_c b_p^2/kT\right) - 1} \qquad (6)$$

From this equation and using the data obtained from climb sequences similar to those of Figs. 5 - 7, a value of ~190± 10 ergs/cm^2 for the stacking fault energy in Al-1% Mg and 210± 10 ergs/cm^2 for that in pure aluminum (see Table III) were obtained.

IV. DISCUSSION

The model adopted for vacancy diffusion in thin foils to obtain the climb kinetics from isothermal experiments, on one hand, and the accurate knowledge of the various parameters involved, such as temperature, loop size (effect of any variation of diffraction conditions), on the other,

are the limitations involved in the calculations of E and γ. We will discuss each factor as follows:

(a) The climb process

Seidman and Balluffi[22] have discussed two models for calculating the radius-time relationships. The first one due to Silcox and Whelan[3] (Eq. (2)) is for the case of a vacancy emission controlled process where the diffusional loss of emitted vacancies to the surface has been assumed to be extremely rapid. The second one, due to Seidman and Balluffi is based on a diffusion controlled process. In its final form, their equation can be given as

$$-\frac{dr}{dt} = \frac{4}{b\pi} D_s \quad \exp\left(\frac{\mu_v}{kT} - 1\right) \tag{7}$$

where D_s is the self-diffusion coefficient and μ_v the chemical potential of the lattice vacancies. Substituting the force due to line tension or stacking fault energy for the μ_v term, the rates of shrinkage of perfect or imperfect loops can be obtained respectively.

The climb kinetics for perfect loops given by either the Silcox-Whelan equation or the Seidman-Balluffi equation are equivalent, and either equation will give the same results for the activation energy. Thus the values we obtained for E are independent of the model adopted.

Integration of Eq. (5) yields a linear radius-time relationship for the size range where the force due to line tension can be neglected in comparison to the force due to stacking fault energy. This is precisely true for pure aluminum (see Fig. (7)). However, in the case of Al-1% Mg alloys, the slope of the (r-t) curve does not remain constant. Thus, the kinetics which hold precisely for pure aluminum do not hold exactly for climbing Frank loops in Al-1% Mg. The discrepancy may be a

result of the segregation of magnesium atoms to the tension side of the extra half-plane of the dislocation loop (which is pure edge for Frank loops).

The results of our bulk annealing experiments and the values of activation energy in Al-1% Mg alloy clearly suggests that the vacancy-magnesium atom binding energy in this alloy is negligibly small. This is in excellent agreement with the results of Beaman and Simmons,[17] who measured E_b as < 0.01 eV in Al-1.1% Mg alloys. On the other hand, climb experiments on supersaturated Al-Mg alloys by Eikum and Thomas[16,18] indicate a marked perturbation of the climb kinetics as a result of the presence of magnesium atoms. This has been attributed to a complex vacancy- magnesium cluster binding energy. They estimated this to be in the range .08 - 0.16 eV[16] depending on Mg content.[18] A summary of the data on Al-Mg alloys is shown in Fig. 8 which also included a suggested variation in activation energy with magnesium concentration.

In the case of supersaturated alloys, the formation of the complex β-phase ($Mg_3 Al_2$) upon prolonged aging probably depends on nucleation from complex magnesium-vacancy clusters which can subsequently grow. In view of our present experiments (and the conclusion by Beaman and Simmons that the true binding energy in Al-1% Mg is < 0.01 eV)[17] the vacancy-magnesium binding in Al-5% Mg alloy investigated by Eikum and Thomas[16] cannot be a simple single magnesium atom and single vacancy interaction.

(b) Parameters used in evaluating the data

In out electron microscopes there is no way of measuring the temperature of the foil directly during annealing (e.g., with a thermocouple inside the microscope). Any accurate external calibration of the hot stage does not take beam-heating into account. However, as far as

the measurement of activation energy by Eq. (3) is concerned, a deviation of 10°C in temperature will not affect the results by more than 2%. The measurement of stacking fault energy using Eq. (6) is also relatively insensitive to temperature changes.[19]

Edington and Smallman[23] have recently estimated the stacking fault energy in pure aluminum to be 285 ergs/cm^2 at ~160°C after isothermal hot-stage annealing of Frank loops. This value is much higher than that obtained in our work. However, Seidman and Balluffi[21] and Thomas[19] have pointed out various objections to the method used by these workers.[23] Furthermore, Saada[24] has shown that the data used by Edington and Smallman[23] do not obey the theoretical kinetics used to evaluate γ. Another very important factor which affects the value of γ determined from isothermal experiments on only Frank loops is the value chosen for the self-diffusion energy in Eqs. (4), (5). For example, by using E_D = 1.25 eV instead of E_D = 1.3 eV as used by Edington and Smallman,[23] their value of stacking fault energy is reduced from 285 ergs/cm^2 to 198 ergs/cm^2, which is much closer to the value obtained in our work. The advantage of using comparative climb measurements is that the results do not depend on any knowledge of E_D nor on the model assumed to control the climb process, i.e., the other factors given by either Eqs. (5) or (7), because the terms are cancelled when the ratio of the shrinkage rates is obtained. The main assumption in the comparative method is that the only difference between the climb rates of perfect and imperfect loops is that due to the stacking fault energy. This assumption is reasonable.

V. CONCLUSIONS

1. Annealing of loops in thin foils in situ in quenched Al-1% Mg alloy follows similar but not identical kinetics to those found previously for

pure aluminum, suggesting that some Mg (and vacancy) dislocation interaction occurs. The activation energy for loop climb in the present alloy was estimated to be 1.2 - 1.28 eV, which is in the same range as that found for pure aluminum, by others[3] and by ourselves (1.28 eV).

2. The absence of heterogeneous precipitation of vacancies, and the bulk and thin foil annealing results, all indicate that the binding energy between magnesium atoms and vacancies is very small in the Al-1% Mg alloy. This is in excellent agreement with the conclusions of Beaman and Simmons[17] based on equilibrium measurements of the vacancy concentration in Al-1% Mg.

3. It is suggested that the ratio: $\frac{\%\ \text{supersaturated solute atoms}}{\%\ \text{vacancy}}$ determines whether positive or negative climb can take place. It is concluded that solute atoms in supersaturation trap vacancies to form complex groups and that the observed climb rates depend on the Mg concentration in supersaturation.

4. Stacking fault energies γ can be quite accurately determined, independently of the model assumed for the climb process, by comparing the rates of climb of Frank loops and perfect loops in the same area of the specimen. The results were: γ (Al) ~210 (±10) ergs/cm^2 and γ (Al-1% Mg) ~ 190 (±10) ergs/cm^2, indicating that Mg has a very small effect on the stacking fault energy of aluminum.

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FIGURE CAPTIONS

Fig. 1 Typical micrographs of Al-1% Mg after quenching bulk specimens from 550°C into oil (20°C); specimen thickness 6.5-7.0 mil.

a) annealed at room temperature for 10 min; Frank, perfect and rhombus loops.

b) annealed at 45°C for 10 min; mostly rhombus loops, some Frank loops.

c) annealed at 175°C for 10 min; growth of rhombus loops.

d) annealed at 200°C for 10 min; large rhombus loops.

Fig. 2 Typical micrographs Al-1% Mg: (a) water quenched specimen aged at room temperature for 24 hr, (b) same as (a), but no aging, (c) and (d) oil quenched and aged for 24 hr. Thickness of quenched sample was ~13 mil. Notice the absence of colonies of loops and almost all Frank defects in (c) and (d).

Fig. 3 Shrinkage of both perfect and imperfect loops at ~207°C in Al-1% Mg alloy aged inside the microscope. The time (in seconds) is given for each micrograph. Notice that the Frank loop A shrinks at a faster rate than the perfect loop B.

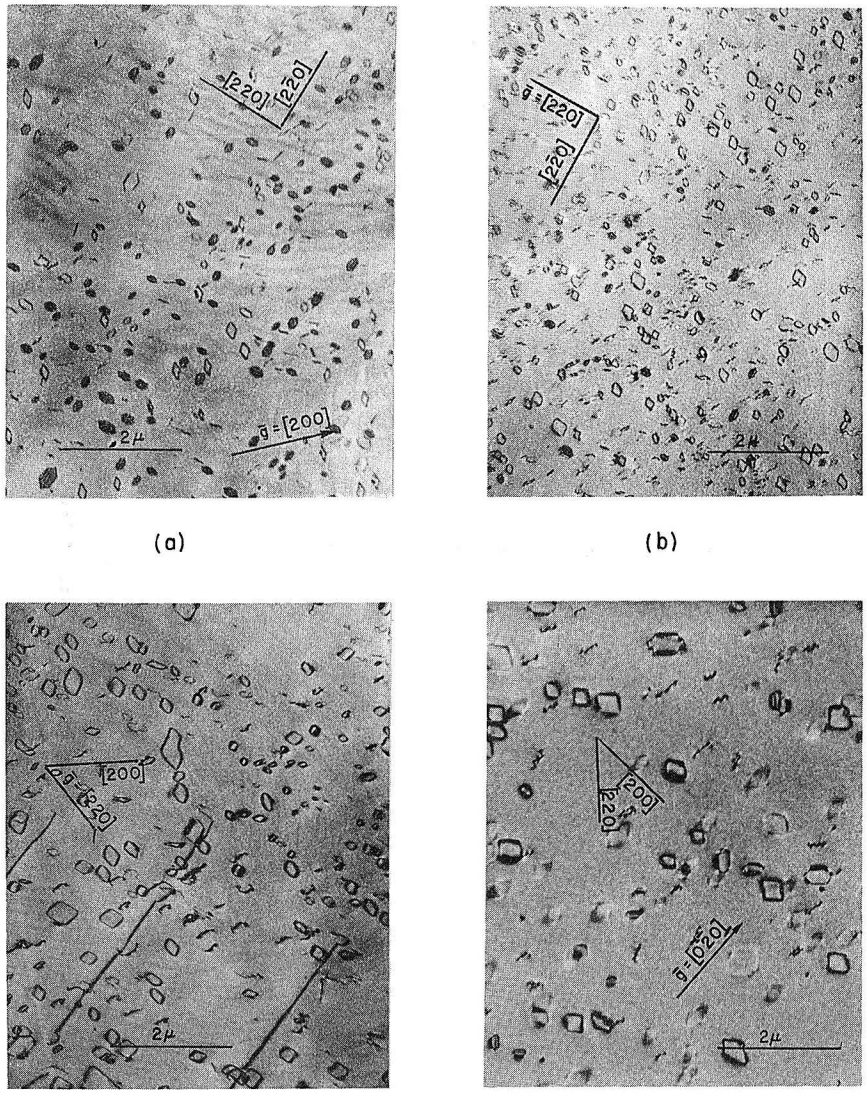
Fig. 4 Similar sequence to that in Fig. 3, showing shrinkage of both perfect and imperfect loops at 220°C in Al-1% Mg alloy (compare loops A and B). The time (in seconds) is given for each micrograph.

Fig. 5 Shrinkage of both perfect and imperfect loops at 210°C in high purity aluminum (compare loops A and B). The time (in seconds) is given for each micrograph.

Fig. 6 Time-radius plot at ~270°C for Al-1% alloy obtained from shrinkage sequences such as in Figs. 3 and 4.

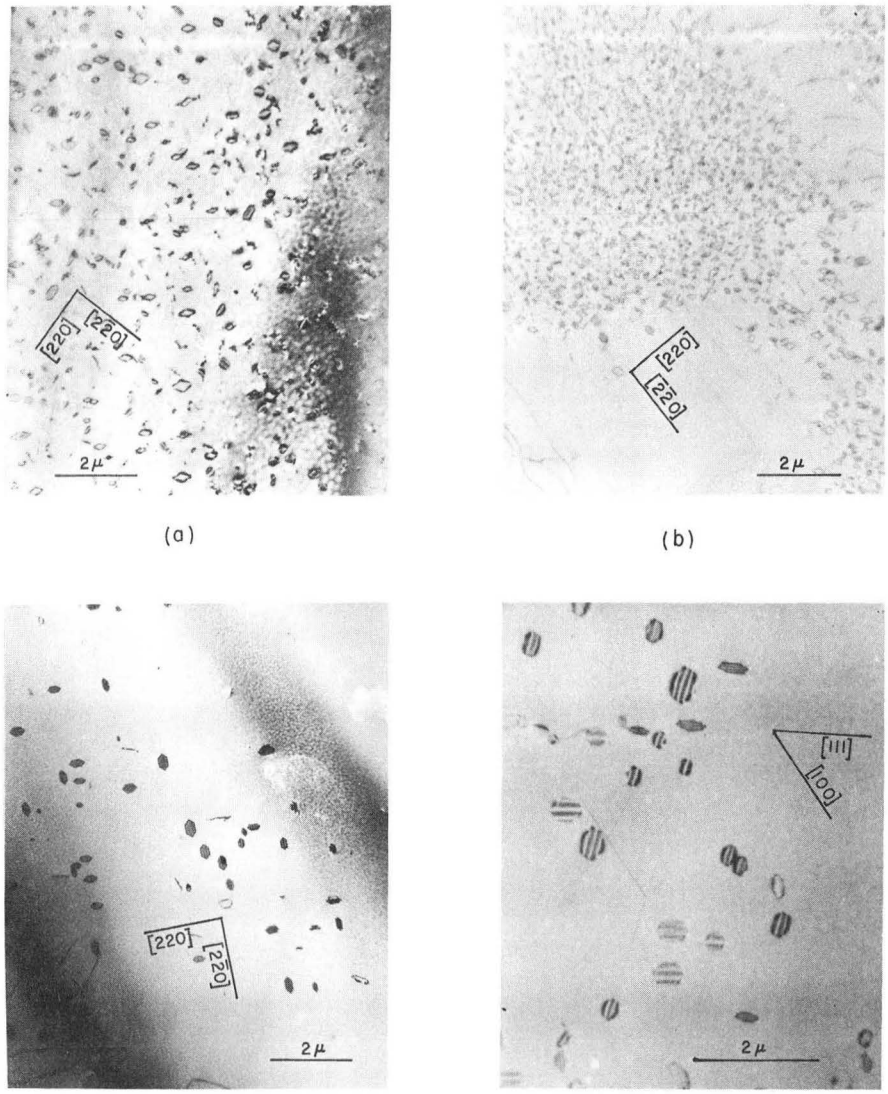
Fig. 7 Time-radius plot for pure aluminum (temp. ~210°C).

Fig. 8 A possible variation of activation energy for loop climb in aluminum as a function of magnesium content. Data for alloys > 1% Mg taken from refs. 16 and 18. The variation is expected to depend on the % Mg in supersaturation and so the data should be affected both by temperature as well as alloy content.


[220]
[2$\bar{2}$0]
$\bar{g}$ = [200]
2μ
(a)
$\bar{g}$ = [220]
[2$\bar{2}$0]
2μ
(b)
$\bar{g}$ = [$\bar{2}$20]
[200]
2μ
(c)
[200]
[220]
$\bar{g}$ = [0$\bar{2}$0]
2μ
(d)

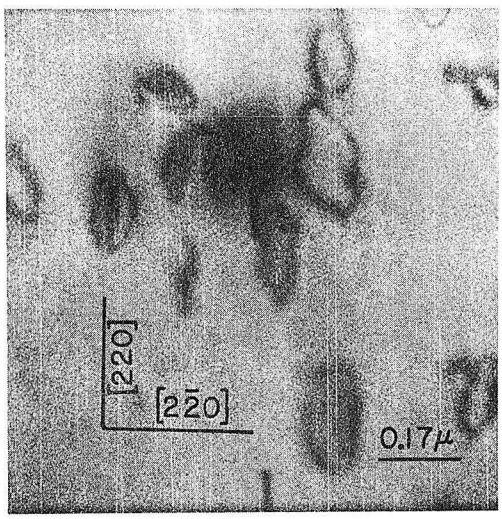
ZN-4823

Fig. 1

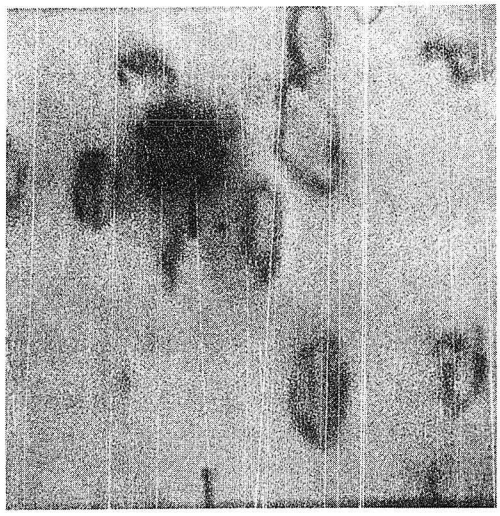


ZN-4822

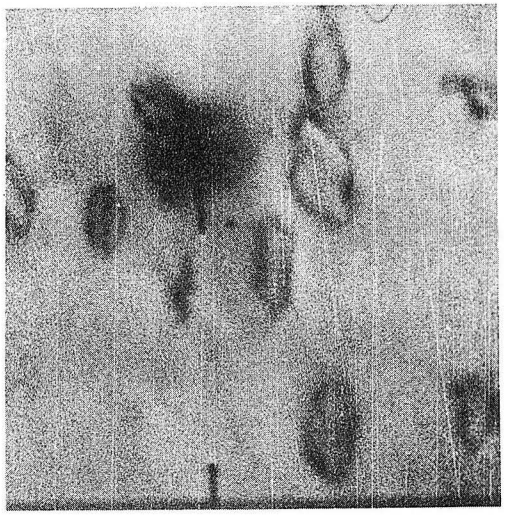
Fig. 2



(a) 0



(b) 17

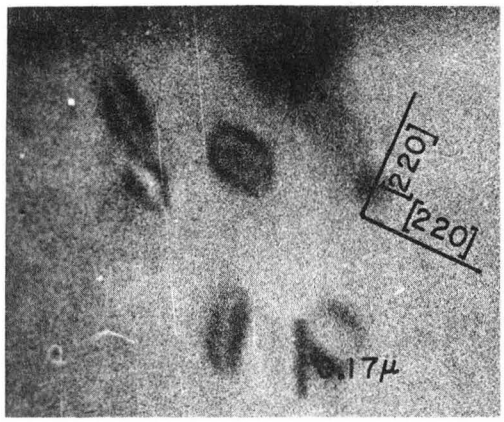


(c) 33

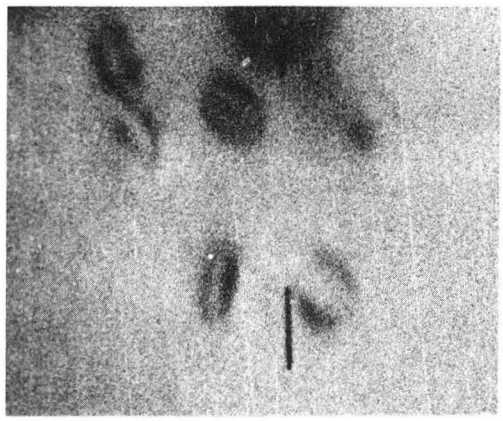
(d) 50

ZN-4819

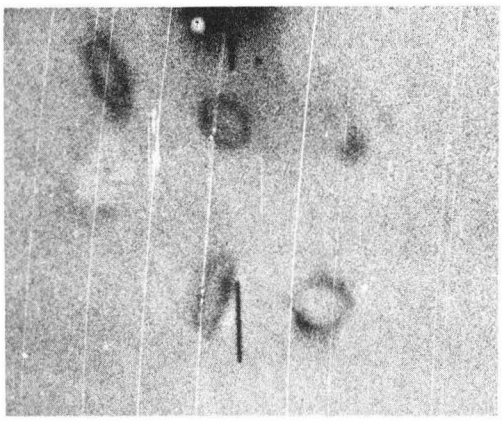
Fig. 3



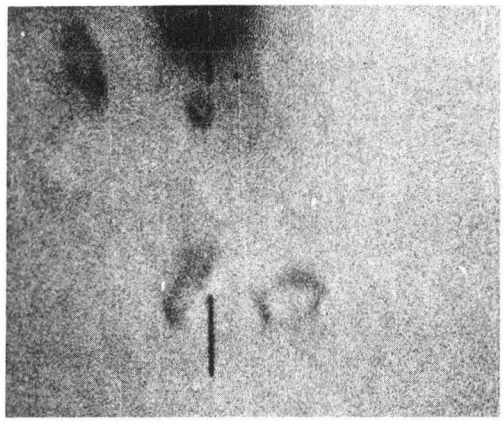
(a) 0



(b) 23



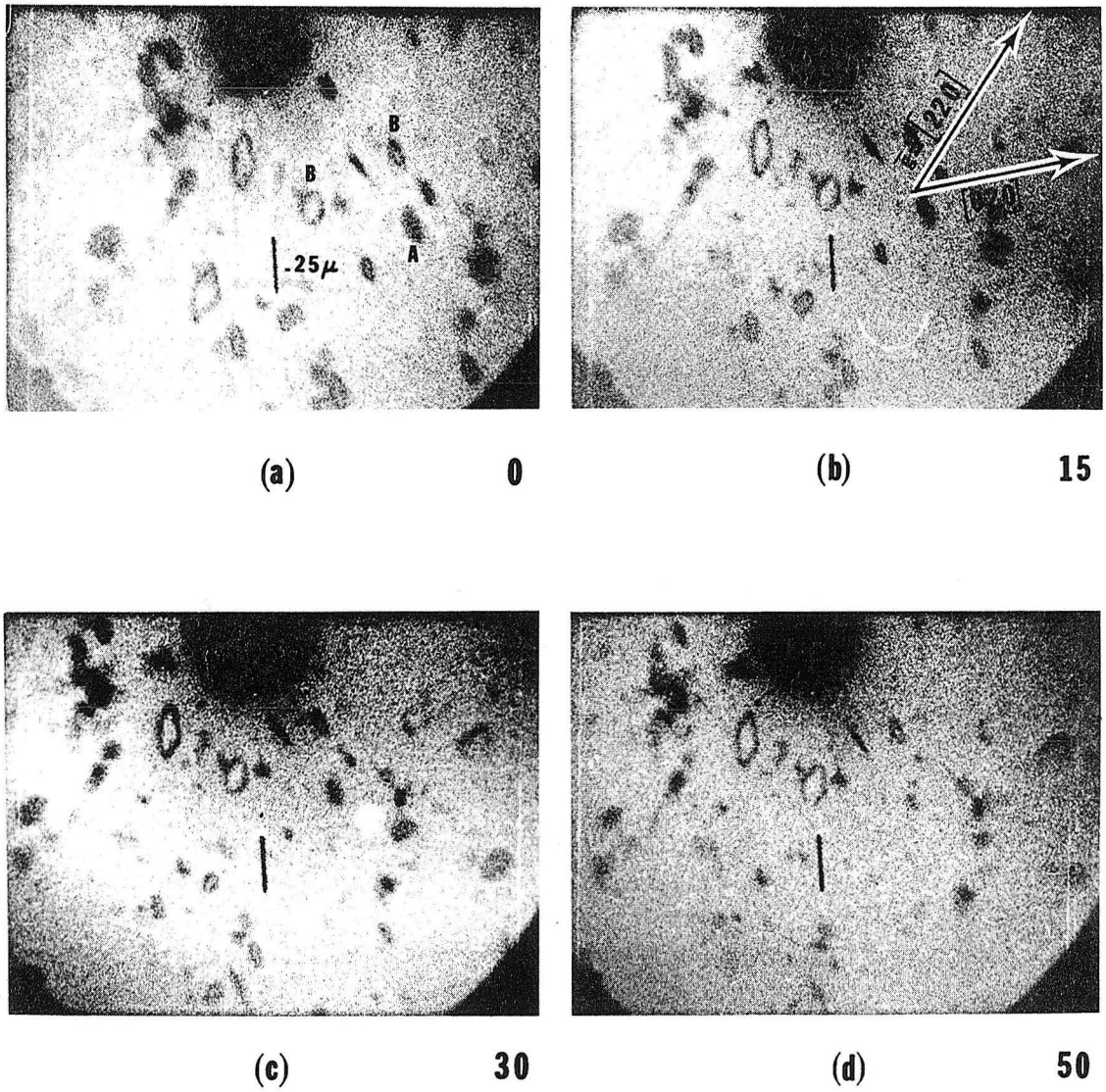
(c) 43



(d) 50

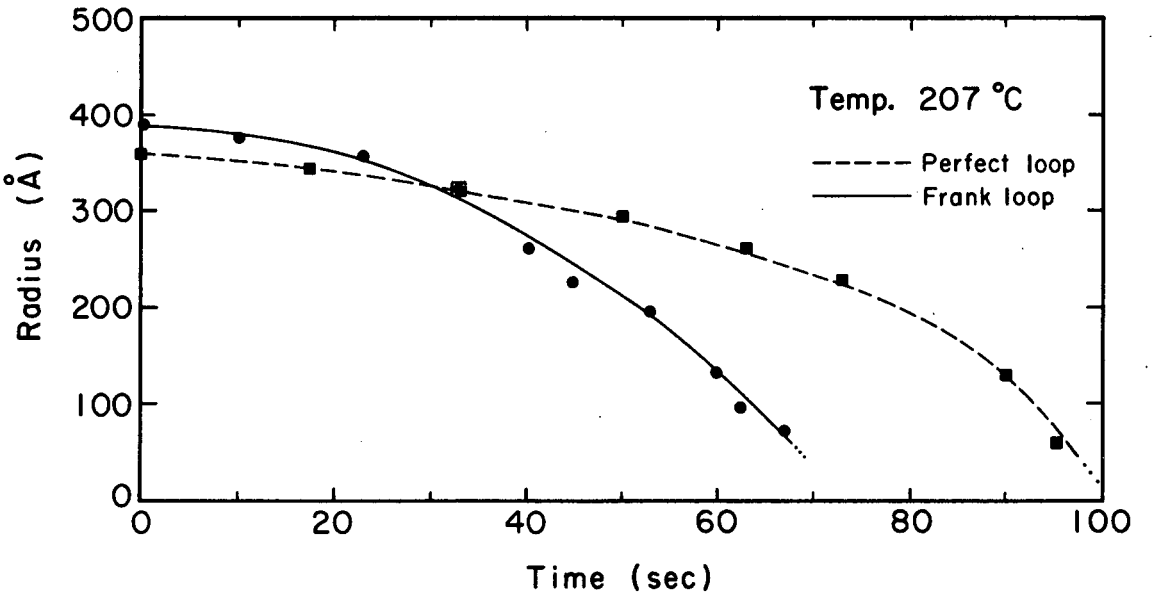
ZN-4820

Fig. 4



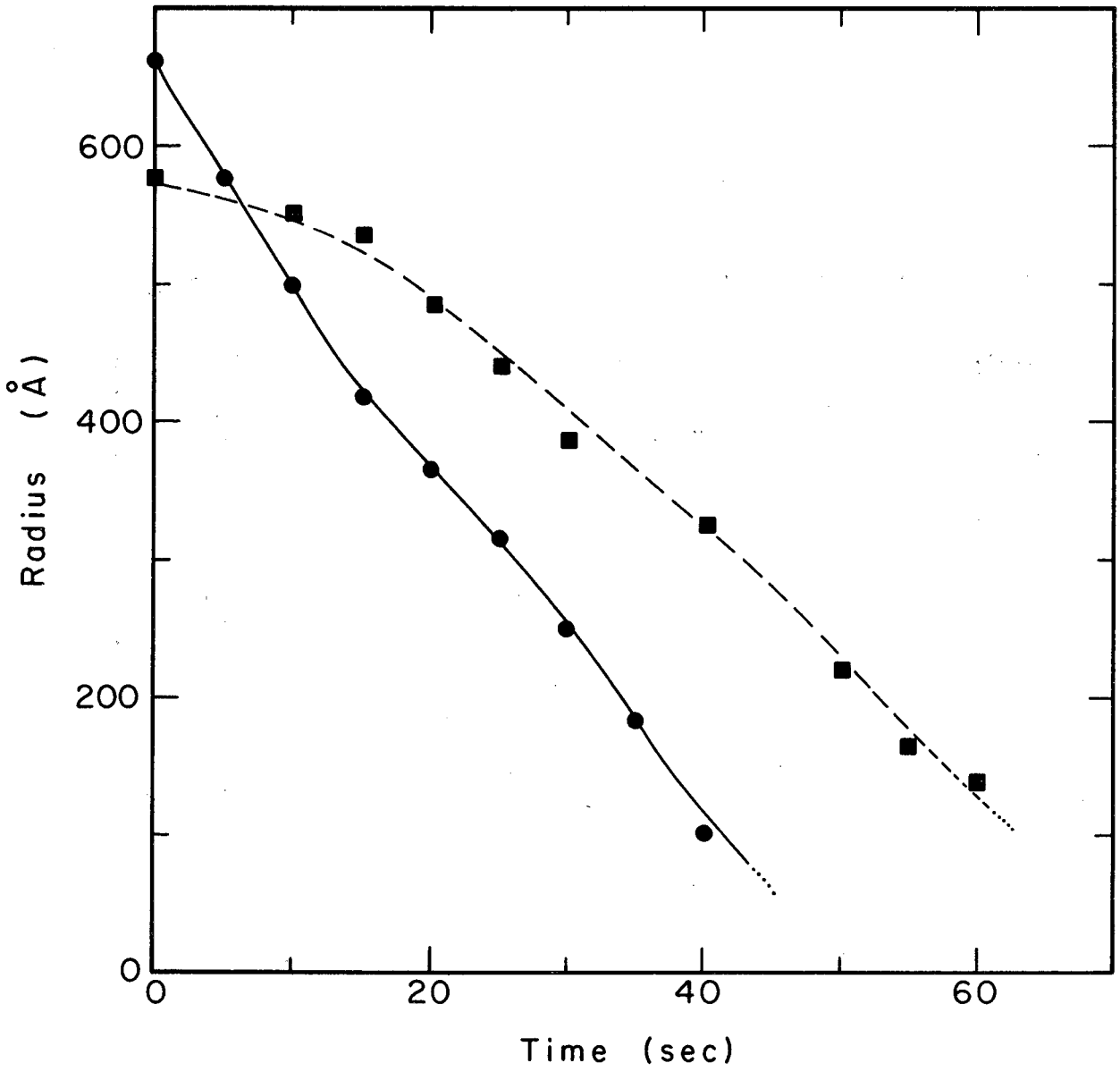
ZN-5292

Fig. 5



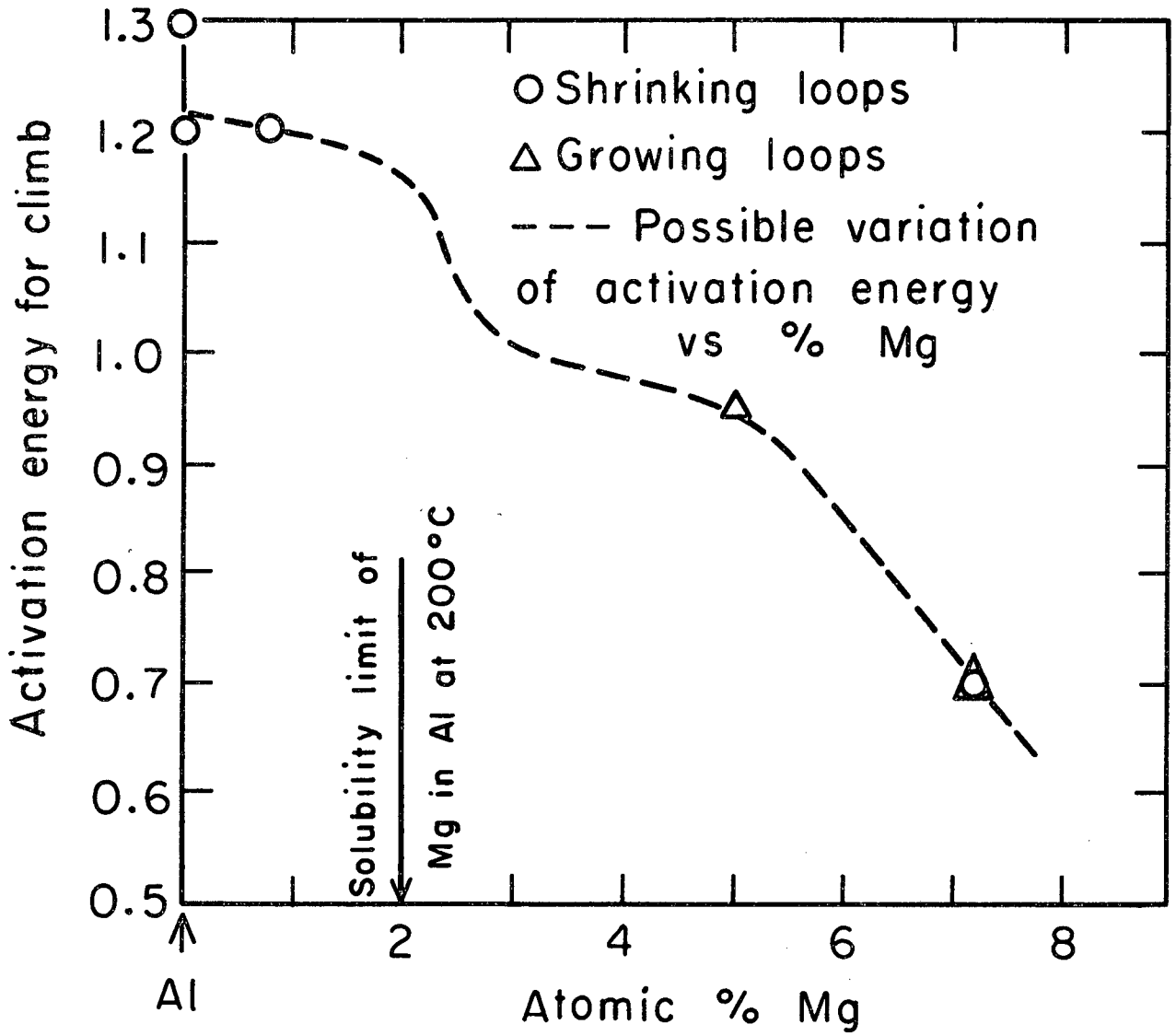
MU-35239

Fig. 6



MUB-8036

Fig. 7



MUB-5171

Fig. 8

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