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ANNEALING OF VACANCIES IN QUENCHED ALUMINUM-MAGNESIUM ALLOY

Berkeley, California

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ANNEALING OF VACANCIES IN QUENCHED ALUMINUM-MAGNESIUM ALLOY

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(Master of Science)

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ABSTRACT

Vacancy climb of prismatic helicoidal dislocations and prismatic dislocation loops in aluminum-5 wt.% magnesium was studied using transmission electron microscopy. From both bulk annealing and thin foil heating stage experiments, the magnesium-vacancy binding energy E_b was estimated to be 0.10-.20 eV.

Helices and loops appear to grow in thin foils just as they do in bulk specimens, which is possible if local vacancy supersaturation can be maintained around the climbing dislocations. Above 250°C, loop growth can be described by an activation energy, $E_{(\overset{\circ}{r})} = 0.95 \pm 0.05$ eV, which leads to a value of E_b of the order of 0.10-0.20 eV. The estimated binding energy accounts for the supersaturation. Some of the observations indicate that helical dislocation turns can degenerate into loops. The degeneration of helices into loops could account for the high density of columns of loops observed in quenched aluminum alloys.

INTRODUCTION

The number of vacant lattice sites N_v in a pure metal can be estimated by an equation of the form, $N_v/N = \exp(-E_f/kT)$, where N is the total number of atoms in the system, E_f the energy to form a single vacancy, k the Boltzman constant, and T the absolute temperature. For pure face-centered cubic metals E_f is of the order of 1 eV, and the calculated (and observed) vacancy concentration is approximately 10^{-4} near the melting temperature. The equilibrium concentration at room temperature is about 10^{-13}. Thus, a large vacancy supersaturation results when the metal is quenched from near the melting point to a temperature where vacancies are not very mobile.

On aging a quenched specimen at temperatures where vacancies are mobile, the supersaturation will be eliminated as vacancies cluster to form dislocation defects,[1-3] annihilate at free surfaces, or "sink" at dislocations. The vacancy migration energy, E_m, is the energy barrier for an atom to jump into an adjacent vacant site. With the formation energy and migration energy it is then theoretically possible to describe the quench-aging sequence in pure metals, i.e., the number of vacancies quenched-in and the rate at which they annihilate.

One might expect vacancy kinetics in a two component system to differ from that in pure metals. Solute atoms in the lattice should perturb both E_m and E_f to some degree as shown for silver-base alloys.[4] In aluminum alloys, using resistivity methods, the copper-vacancy binding energy has been estimated to lie between 0.15 and 0.25 eV,[5] and the zinc-vacancy binding energy is estimated to be of the order of 0.06 eV.[6] In addition to vacancy-solute atom interaction, the solute atom clustering, precipitation and solute atom-dislocation interaction might be expected

to affect the ultimate quench-aged structure.

Prismatic dislocations in the form of helicoidal lines are found in binary aluminum-base alloys after quenching from near the melting point.[7-9] Helical dislocations have been observed in non-metallic systems.[10-12] Helices can form by climb of dislocations which are primarily of screw character. Columns of prismatic loops are often observed to be closely associated with helices. The two types of arrays, helices and columns loops, have parallel axes in the ⟨110⟩ direction. In some cases, within limits of resolution, the two arrays appear to have a common axis. The columns of prismatic loops are thought to originate from helices rather than being a product of independent nucleation.

Little is known about the exact mechanism of helix formation. Impurity atoms in solid solution appear to be a very important prerequisite. Also, the concentration must be higher than some minimum value as evidenced by a tendency for random loop formation in low solute content aluminum base-copper and aluminum base-magnesium alloys.[8] Impurity species is important too, e.g., helices are seldom found in aluminum base-zinc alloys containing up to 30% Zn.[8] It appears that solute atoms are effective in two primary ways; solute atom-dislocation interaction and solute atom-vacancy interaction. Precipitation or clustering may cause pinning of the screw dislocations so that they can act as stationary vacancy sinks. That is, stationary in the sense that glide is inhibited. Once pinned the screw dislocations could readily climb into helical form. Secondly, vacancy-solute atom interaction may be necessary to give a vacancy supersaturation that is sufficiently high for the formation of stable helices.

The purpose of the present work was to study quench-aging of vacancies in the presence of solute atoms. Aluminum-5 wt.% magnesium alloy was chosen because the -phase, which might obscure the climb effects of interest, does not occur as an observable precipitate during isothermal anneal. For this reason the Al-4% Cu alloy is not satisfactory.[13]

EXPERIMENTAL PROCEDURE

A. Specimen Preparation.

Specimens were taken from Al-5% Mg (nominal) strip which had been cold rolled to 0.005 inch thickness with intermediate anneals. The chemical analysis for the two heats which were used is given in Table I.

TABLE I
CHEMICAL ANALYSIS

	Mg	Cu	Si	Fe	Mn
Heat (A)	5.11	.003	.003	.003	.004
Heat (B)	5.12	.003	.014	.007	.001

Heat "A", 5.63 atomic percent Mg, was used for all work including the heating stage experiments. Heat "B", 5.65 atomic percent Mg, was used to repeat observations on the quenched and 20°C aged condition. There was no observable difference between the two alloys.

The specimens, 4 ins. by 1 in. by 0.005 ins., were held at temperature, 550°C ± 5°C, for one-half hour prior to quenching into ice-brine at approximately minus 15°C. The four inch strips were cut in half after quenching to make two specimens. The quenched condition includes a post-quench age of no less than 24 hours at approximately 20°C. Some specimens were quenched from 520°C into 78°C water with no significant difference in structure when compared to the 550°C quench. These samples

were subsequently used for long time aging treatments.

A standard "window method" for making thin foils from aluminum base alloys was used.[14] The electrolyte, 1 part perchloric acid to 4 parts ethyl alcohol, was maintained at -15° to -20°C during polishing for best results. Specimens from which bulk observations were made were saved and subsequently used for heating stage experiments. The specimens were examined in a Siemens Elmiskop Ib electron microscope operated at 100 kV.

B. Bulk Annealing.

Aging of bulk specimens (0.005 ins. thick) was carried out at 100°, 183°, and 300°C for 60 minutes to study helix and loop growth. To find out where precipitates would first form, two aging treatments were used. A 94 hour age at 100°C was used to bring about precipitation without annealing out the loops and helices. The second, "B" age, was a sequence of temperatures as follows; 20 hours at 200°C plus 30 hours at 150°C plus 20 hours at 100°C.

The density per unit volume of prismatic loops and helical turns was determined for the quenched and the 60 min. 183°C age treatments. The two types of defects, columns of loops and helices, were treated separately. The volume element was chosen to include only a single loop column or helix. The area, which is illustrated in Figure 2, was taken as the projected defect length times the center-to-center distance of two adjacent defects. A foil thickness of 5,000Å was assumed to hold for all measurements. It was possible to measure the thickness in one case, which was 5,000Å.

C. Heating Stage.

Thin foil annealing experiments were carried out using a Siemens Heating Stage in the Siemens Elmiskop Ib. A 12-volt storage battery

served as the constant voltage power supply. Current to the platinum furnace was regulated through a variable resistance and monitored with a watt meter. The Siemens calibration curve for specimen temperature as a function of power input is shown in Figure 1. Temperature is estimated to be accurate to ±15°C.(15)

Loop growth was recorded using a Paillard-Bolex H16 movie camera to photograph the fluorescent screen. The camera was driven by a Bolex Electric Motor, Type MC 17, which allowed continuous exposures of up to 100 feet (4,000 frames). The combined camera and motor speed settings gave a resultant film speed of 10 frames per second, or 400 seconds total for a maximum continuous run.

The data were recorded on Kodak Tri-X film using a Bell and Howell f0.95 lens full open. Exposure was only just adequate in many cases, but Tri-X film represents an upper limit to the loss of detail one can tolerate.

EXPERIMENTAL RESULTS

A. Bulk Annealing.

Structure attributed to the as-quenched condition is thought to develop during quench and room temperature storage prior to electropolishing. Adjacent regions of the specimen included poorly developed helices, relatively uniform helices and columns of prismatic loops as shown in Figure 2. Some loops which appear to be of random origin were also present. The large variation in structure was always found. The dislocation structure illustrated in Figs. 2a and 2b is representative of regions of the foil within the grain interior away from grain boundaries. Figures 2c and 2d show the dislocation structure adjacent

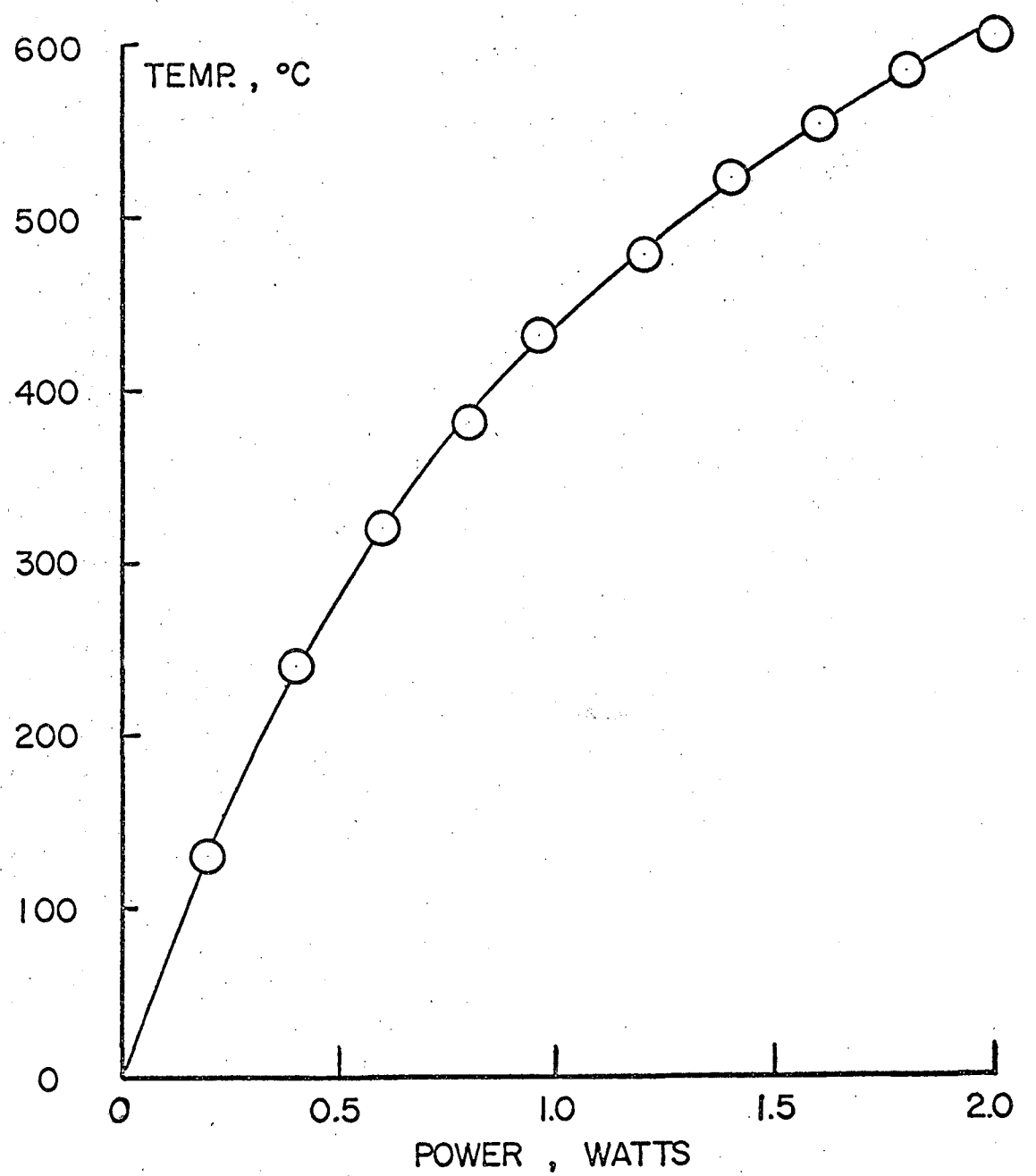
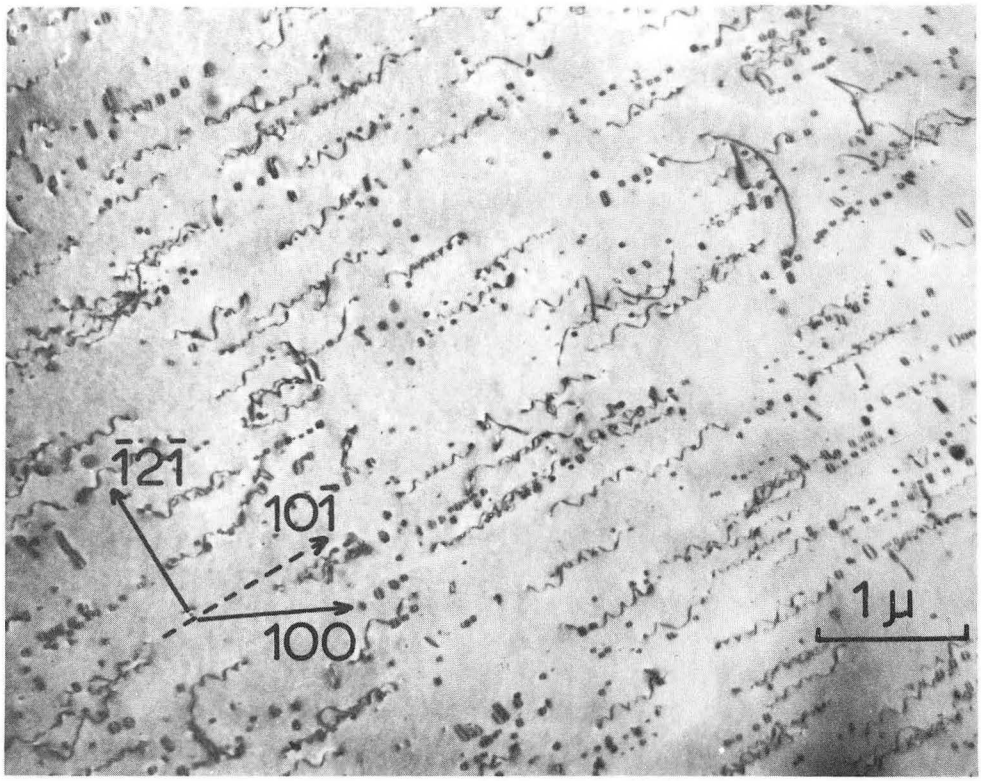


FIGURE 1. Temperature calibration curve for the Siemens heating stage.



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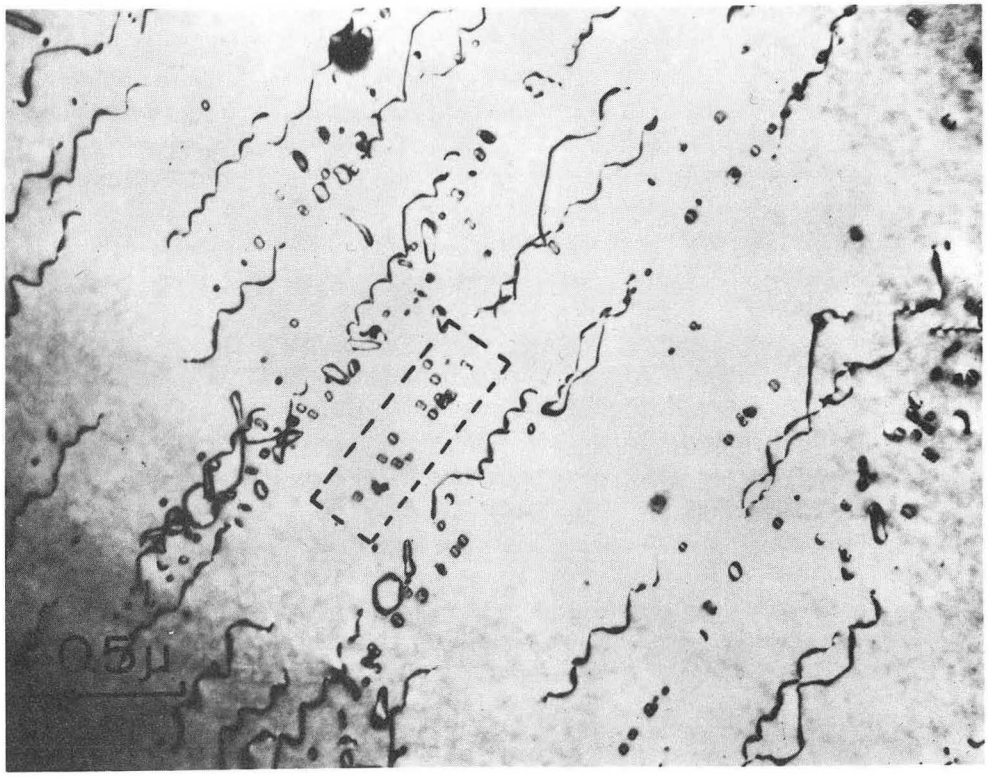
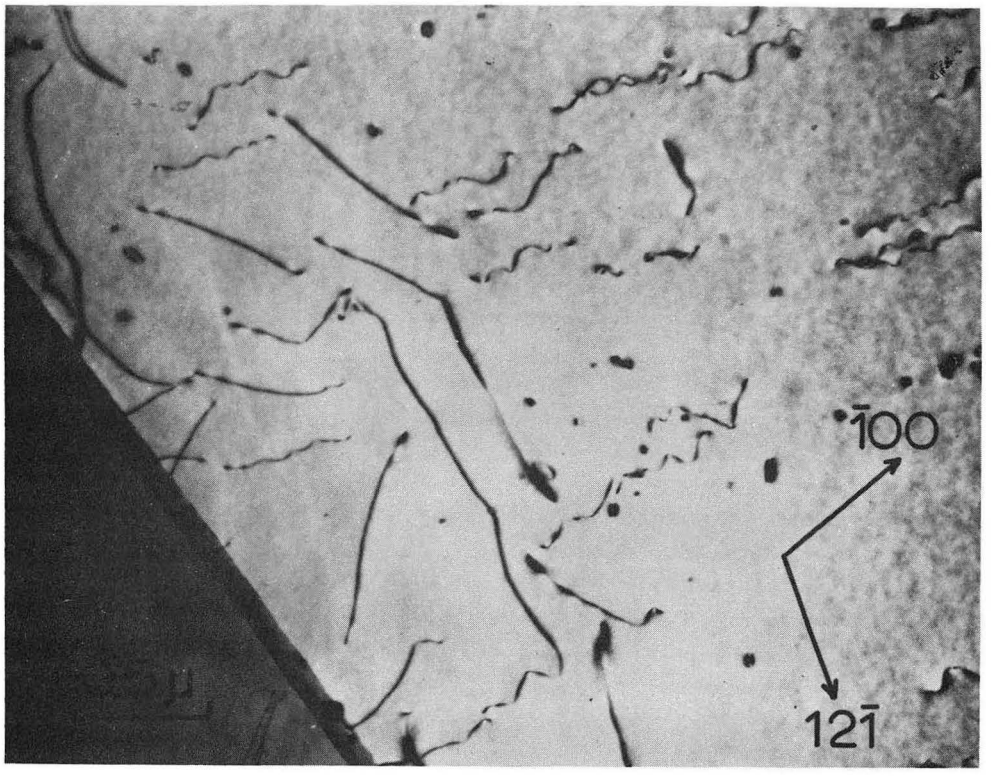


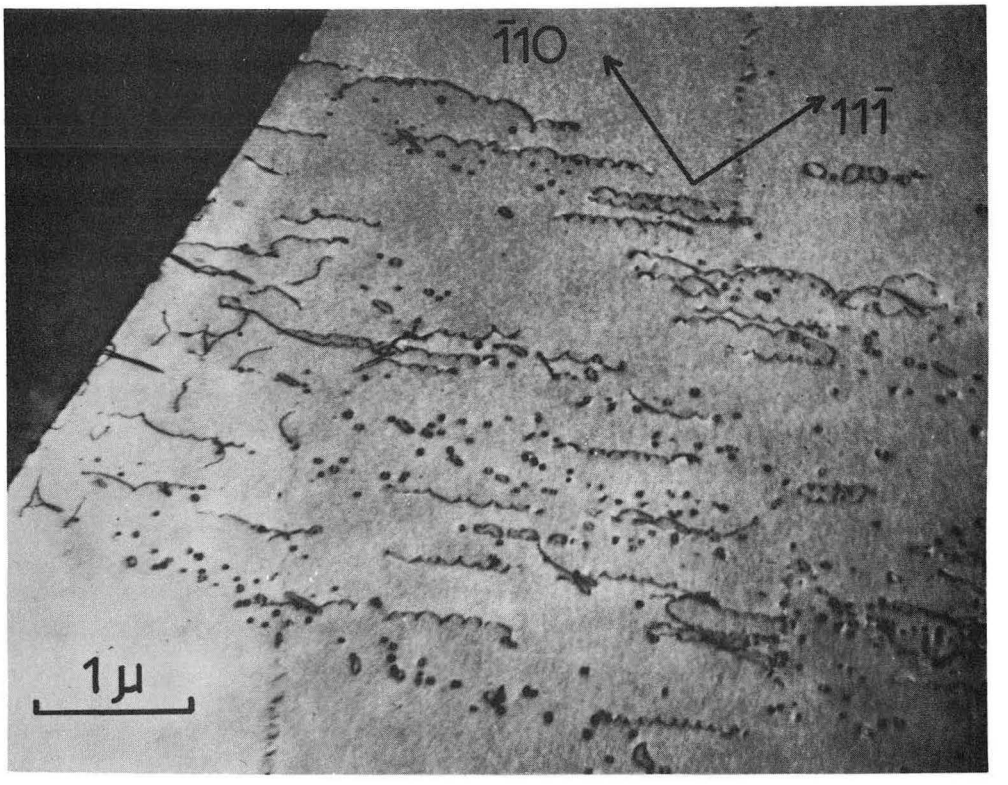
-b-

ZN-3970

Fig. 2. Al-5% Mg quenched from 550°C into ice-brine, aged at 20°C.



-c-



-d-

ZN-3971

Fig. 2. Al-5%Mg quenced from 550°C into ice-brine, aged at 20°C.

to grain boundaries. The observation that helices occur in bands was common, (see Fig.2d).

The tendency for loops and helices to become angular during aging is illustrated in Figure 3 which represents a 60 minute age at 183°C. The spacing and radii are still the same for both loops and helices. The helical axis (at X, Fig. 3) is parallel to the [011] direction and meets the (001) foil surface at a 45° angle. The column of loops at Y in Fig. 3b is also parallel to the [011] direction.

The density of dislocation loops and the density of helical dislocation turns in specimens quenched from 550°C and aged at 20°C and 183°C are given in Table II. The loops and helical turns were counted within

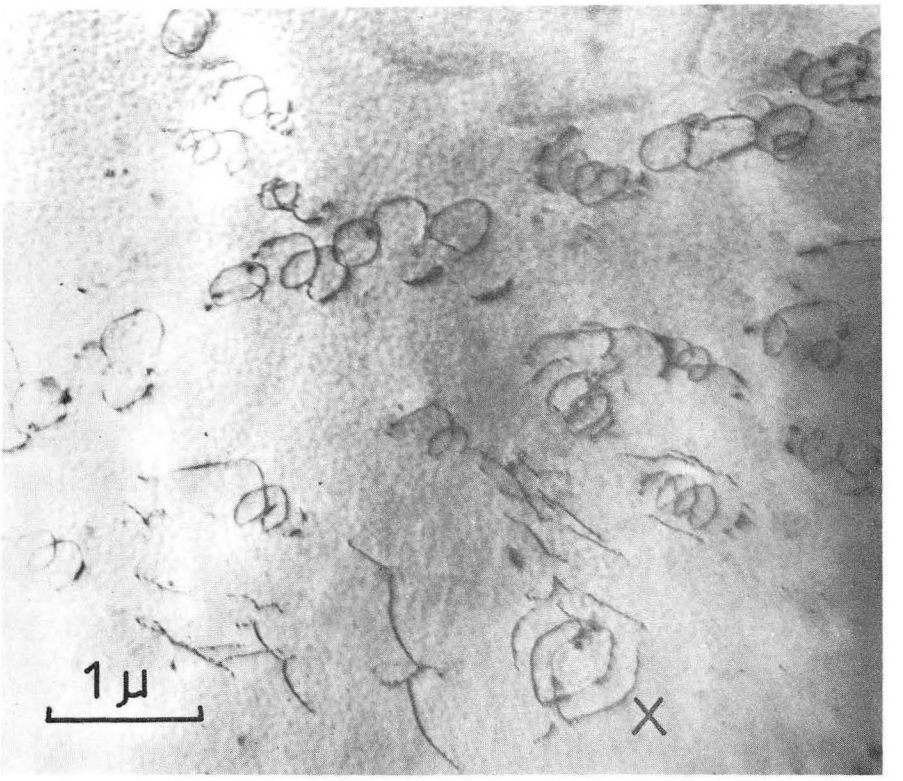
TABLE II
CALCULATED VACANCY CONCENTRATIONS

Specimen Treatment		N* Loops/cm^3	Diameter* Å	C_v'
Quenched 550°C aged at 20°C.	Helices	7.1×10^{13}	483	3.7×10^{-5}
	Loops	1.2×10^{14}	360	3.6×10^{-5}
Quenched 550°C and aged 60 min. at 183°C.	Helices	1.1×10^{13}	2740	1.9×10^{-4}
	Loops	1.4×10^{13}	2890	2.6×10^{-4}
Single large helix		7.1×10^{12}	6250	6.3×10^{-4}
Single large loop		3.6×10^{12}	6250	3.1×10^{-4}

*N is Turns/cm^3 and Diameter is that of circumscribing cylinder in the case of helices.

separate volume elements, as described in the section of Experimental Procedure, and can not be added together to give a total density. The loop density refers only to those loops occuring in columns. The vacancy concentration C_v' necessary to produce a given density of loops N can be estimated by the equation,

$$C_v' = \pi r^2 \sigma N$$



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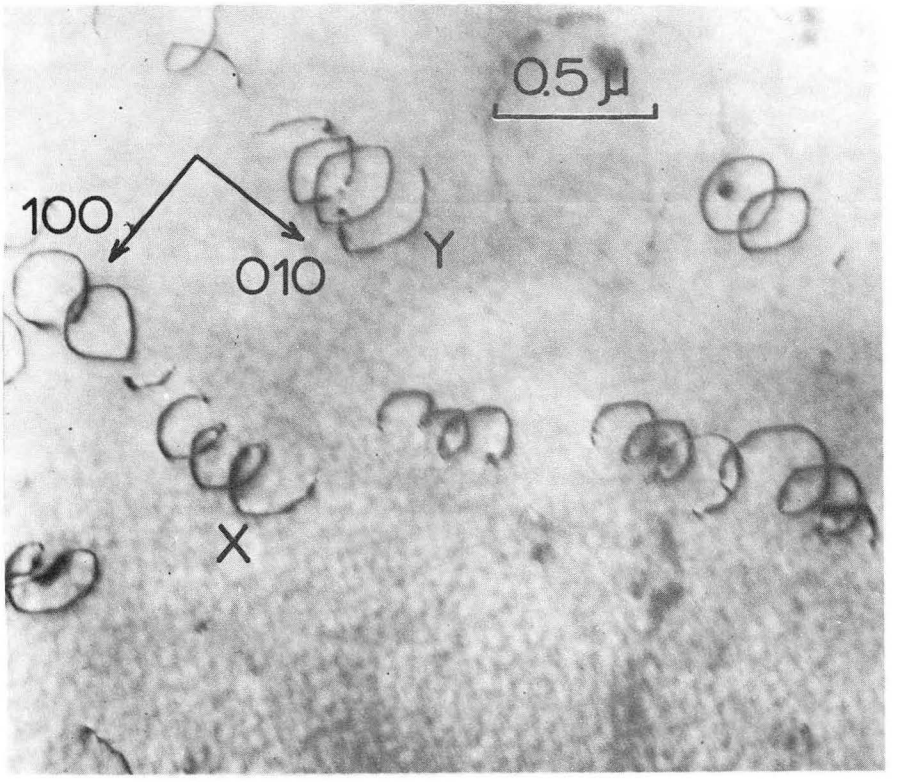


-b-

ZN-3972

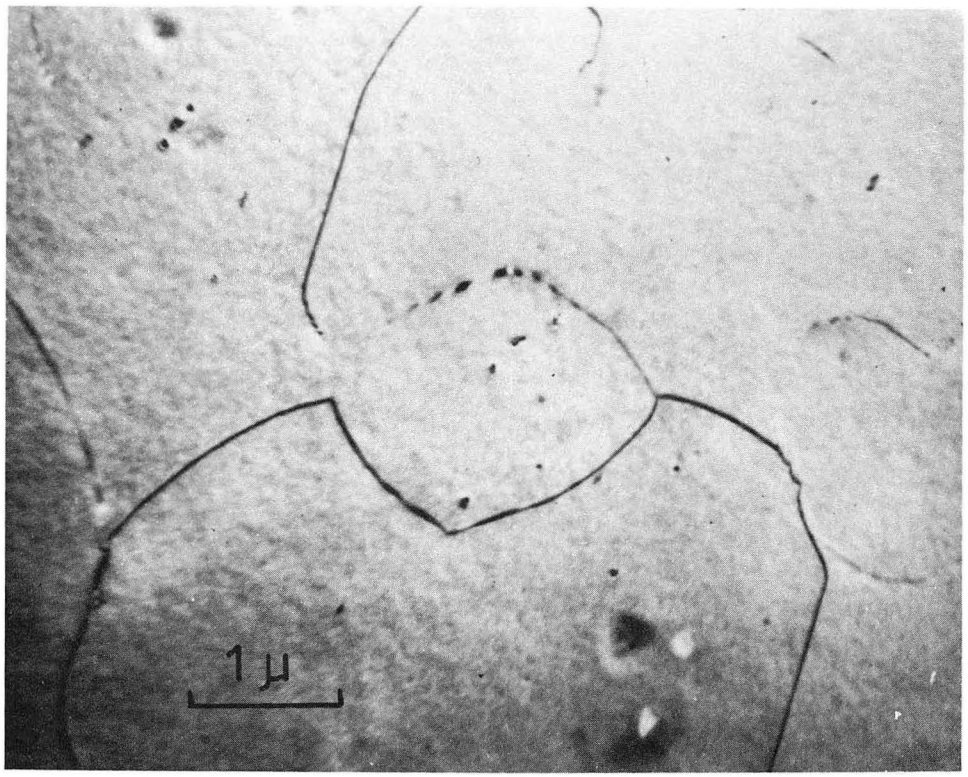
Fig. 3. Al-5% Mg quenched from 550°C into ice-brine, aged 60 minutes at 183°C.

where r is the average loop radius, and b the Burgers vector. The same relationship can be applied to helices by assuming that a helicoidal line can be approximated by a column of loops. In the case of helices, N is the number of turns per unit volume, and the radius is that of the circumscribing cylinder.

The vacancy concentration associated with helices is approximate y the same as that with columns of loops as shown in Table II. The concentration has increased from 3.7×10^{-5} in specimens quenched from 550°C and aged at 20°C to 2.3×10^{-4} in specimens aged for 60 min. at 183°C. The single large loop and single large helix, each with diameter equal to 6250Å, are included to represent an upper limit on the vacancy concentration calculated for specimens aged at 183°C.

Very few dislocations were found after aging 60 minutes at 300°C. The structure is shown in Figure 4. Those dislocations present are arranged in a three-dimensional array, e.g., Figure 4a. No helices were observed, but some trace of large loops, or what appeared to be segments of loops after being cut by the foil surface, were observed.

Several long time aging treatments were used to obtain some information on Beta-phase precipitation. These are illustrated in Figures 5 and 6. The only consistent indication of precipitation is that at grain boundaries, e.g., Figure 6. The contrast, which was sensitive to tilting, is thought to be due to strain fields associated with precipitates. This contrast was also observed after annealing 94 hours at 100°C, but on a much finer scale. It is important to note that Beat-precipitate has a high strain energy associated with it even at grain boundaries as evidenced by the dislocations being generated and by the contrast.



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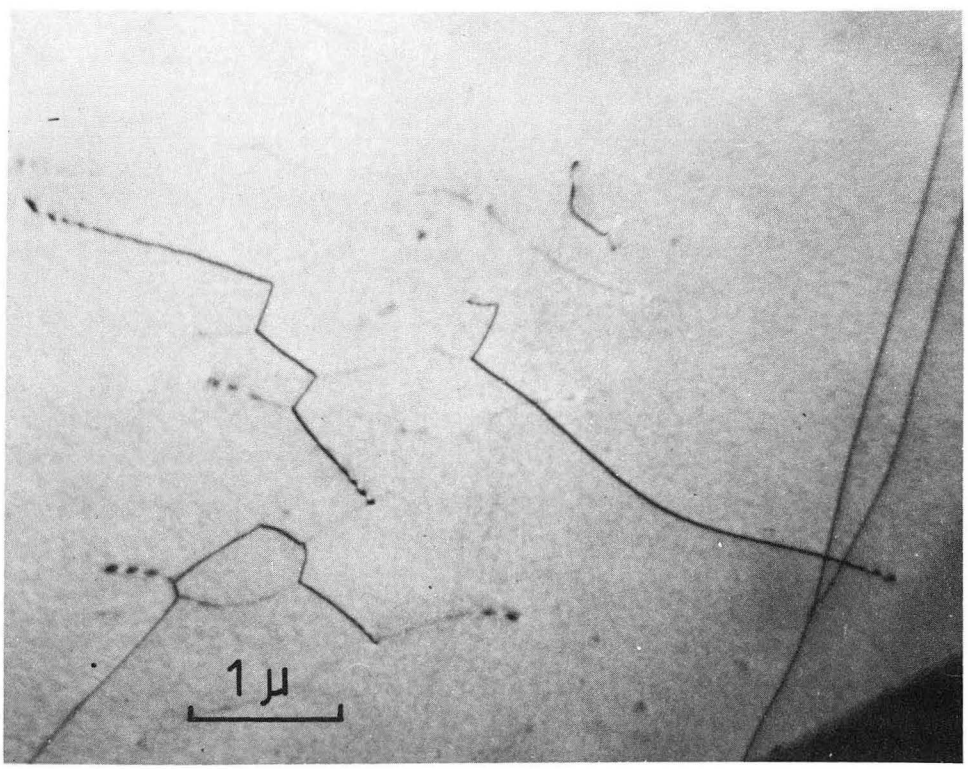
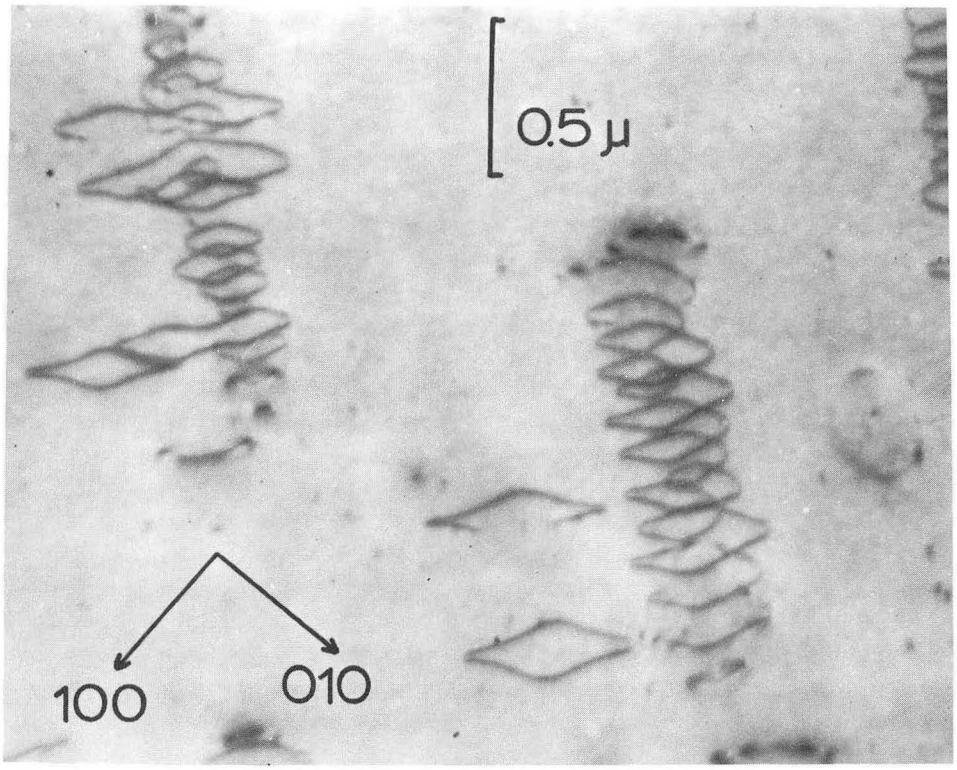


-b-

ZN-3973

Fig. 4. Al-5% Mg quenched from 550°C into ice-brine, aged 60 minutes at 300°C.



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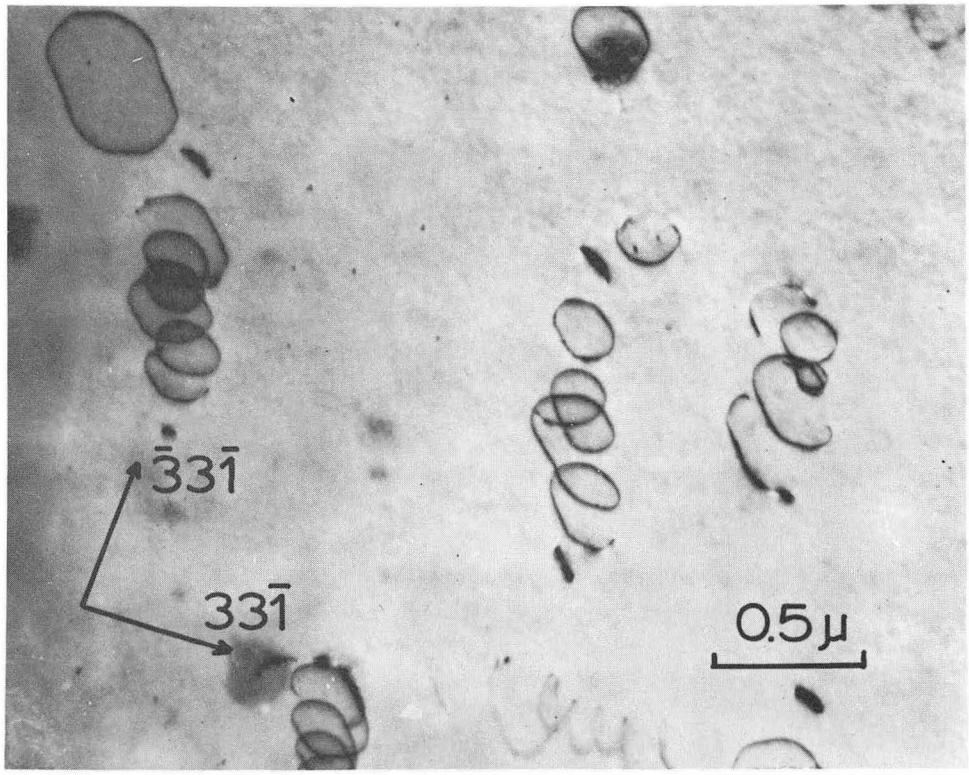
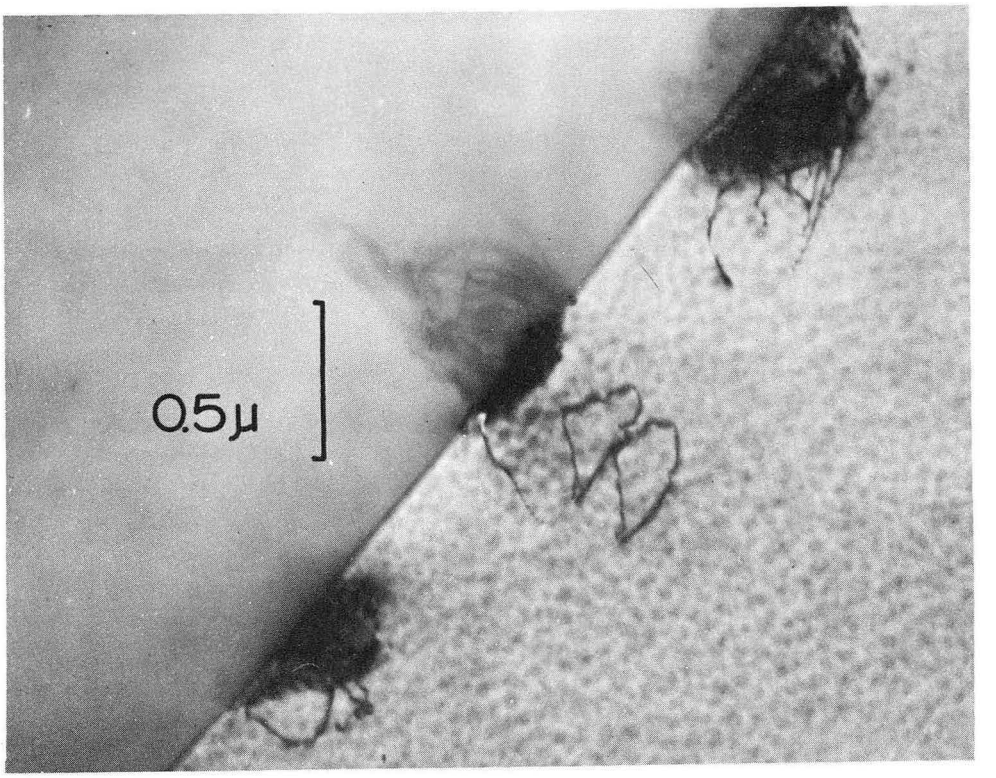


-b-

ZN-3974

Fig. 5. Al-5% Mg quenched from 520°C into 78°C water, aged 94 hours at 100°C.



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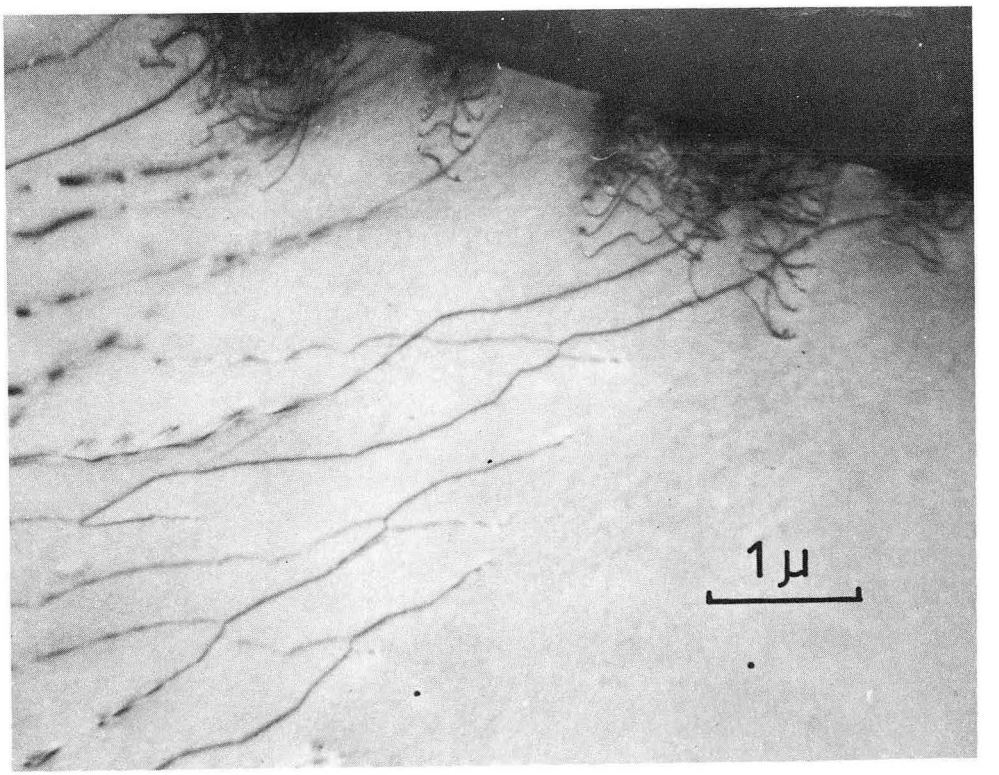


-b-

ZN-3976

Fig. 6. Al-5% Mg quenced from 520°C into 78°C water, aged according to "B" schedule in Experimental Procedure.

B. Heating Stage.

Bulk annealing experiments indicate that loops and helices are much more stable in Al-5% Mg, than are loops in pure aluminum. Thus, one would hope to obtain information on vacancy diffusion in the presence of magnesium atoms from loop growth rate. Also, one would hope to study the degeneration of helices in thin foils, provided the foil bulk is adequate for completion of such a reaction.

The climb of prismatic loops as a function of temperature is summarized in Figure 7. Above 250°C, loops were always observed to grow these data represent isolated loops with initial radii ranging from 200Å to 1500Å. Loop growth above 250°C occurs with an energy 0.95 ± 0.05 eV. This is much smaller than the value 1.3 eV for loop climb in pure aluminum.[16] The low value of $E_{(\dot{r})}$ in Al-5% Mg and the observation that loops grow in thin foils tend to support the hypothesis of a strong magnesium-vacancy binding energy.

An example of loop growth is shown in Figure 8 and the change in radius with time in Figure 9. Below 250°C, with the exception of a single case, loops were also observed to grow. However, the data are not sufficient for kinetic analysis.

An example of helix growth is given in Figure 10. No kinetic data for helices is available. However, it is important to note that helices tend to become angular just as in aged bulk samples.

The only case of loop shrinkage was observed at 240°C. The decrease in radius with time follows a parabolic growth law, as shown in Figure 11. The time (t) for a given loop to shrink to r=0 can be related to the activation energy for vacancy diffusion,[16]

$$t = t_o \exp(E/kT)$$

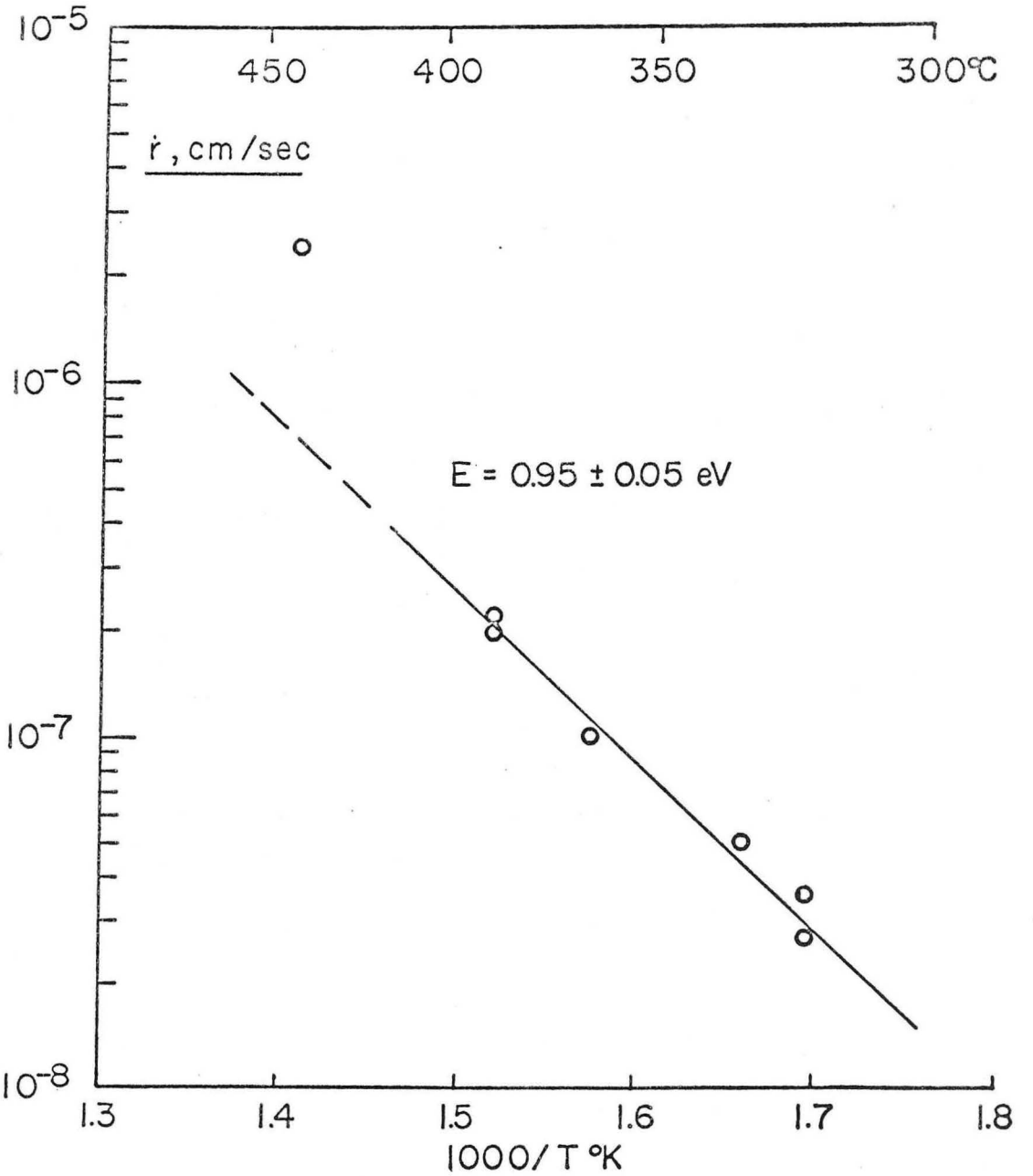
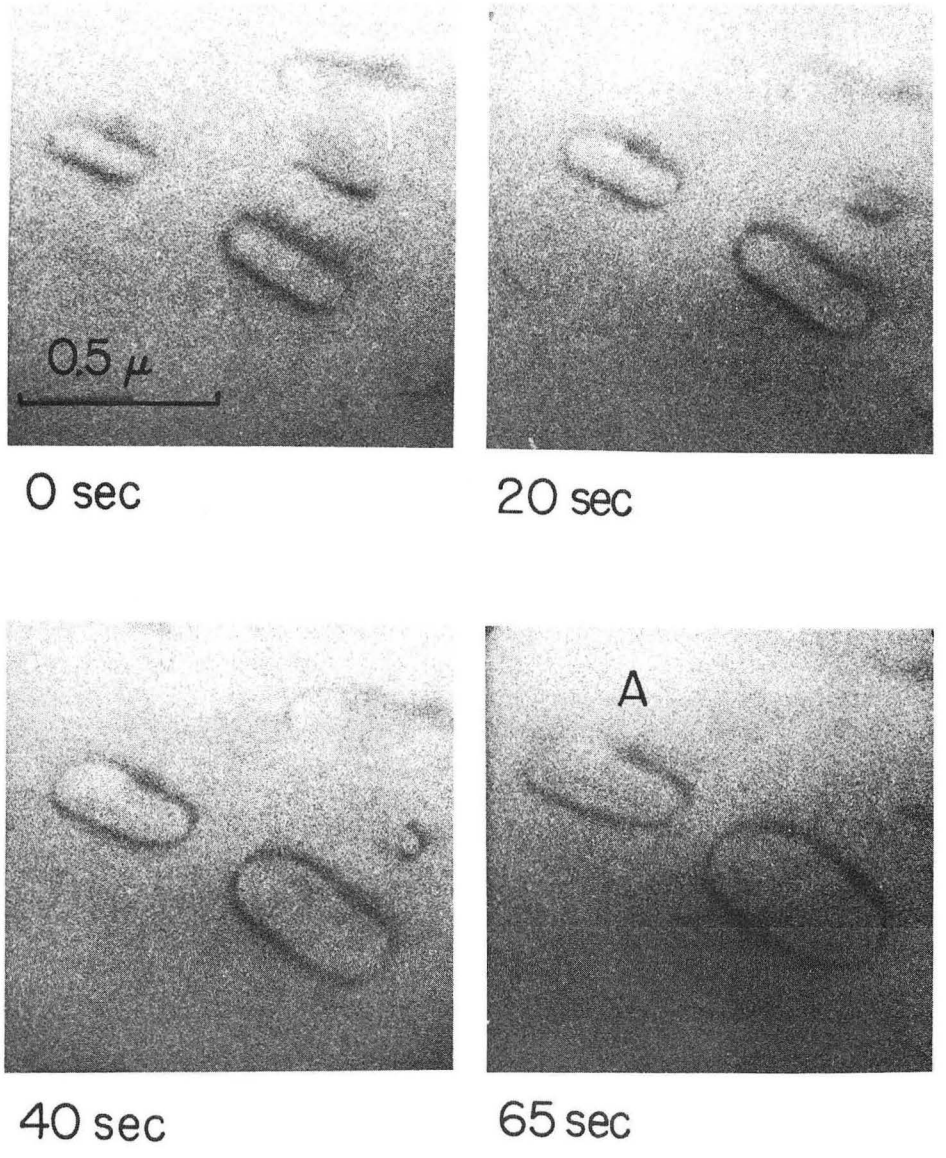


FIGURE 7. Rate of increase of dislocation loop radius vs reciprocal of the absolute temperature.



ZN-3266

Fig. 8. Heating stage sequence showing loops growing at 365°C. Note the loop cutting a surface at "a" after 65 seconds.

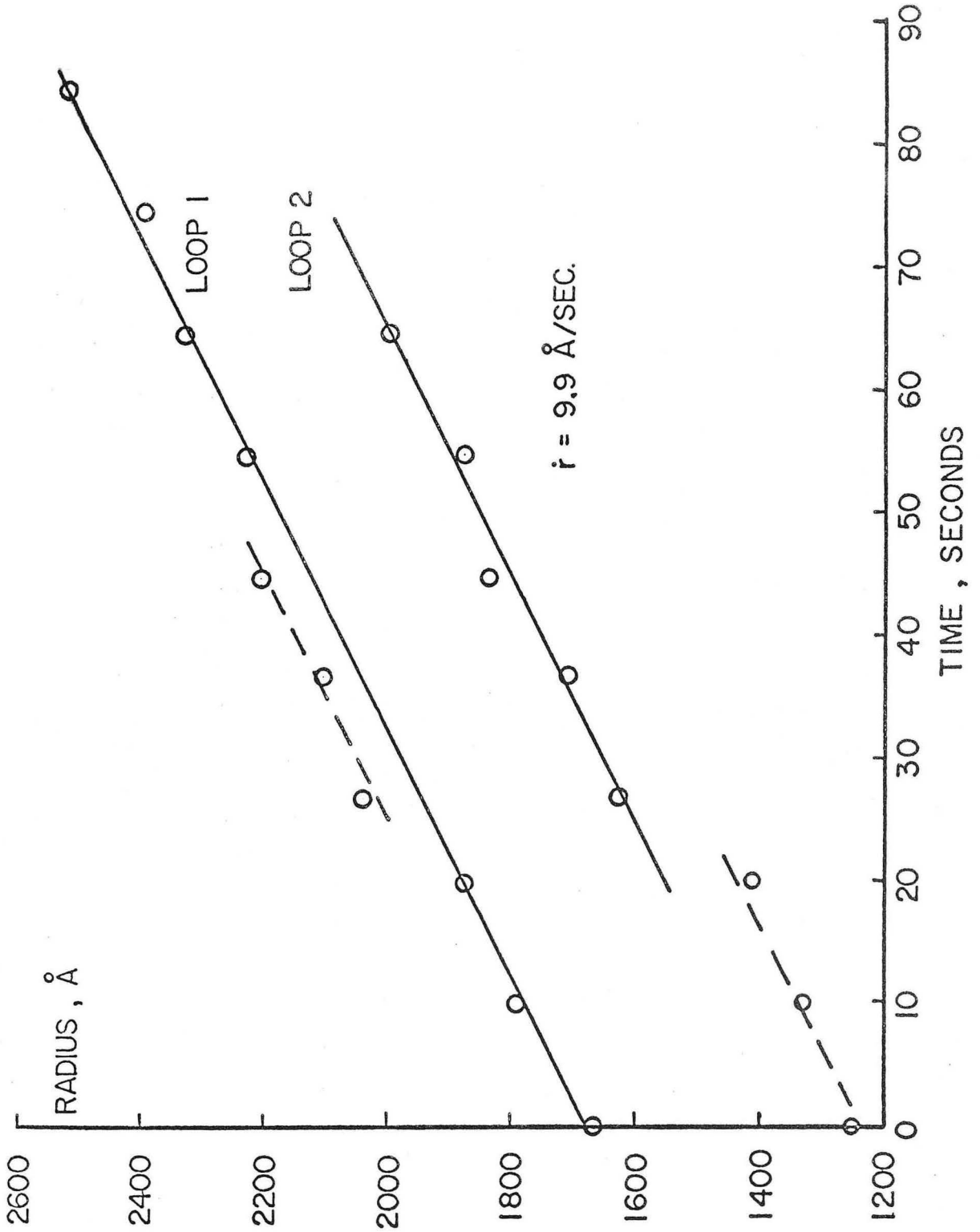
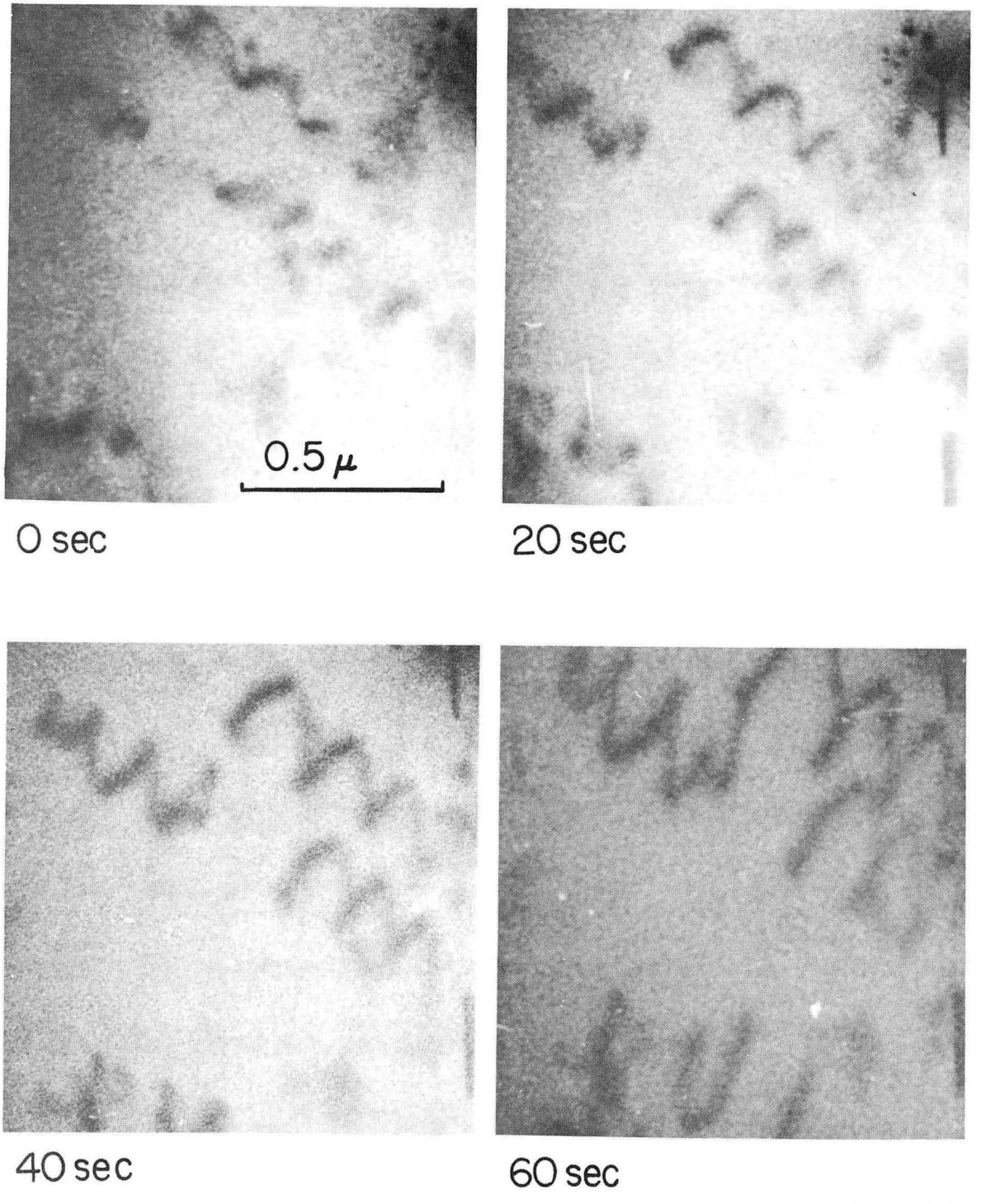


FIGURE 9. Prismatic loop growth from the sequence shown in Fig. 8.



ZN-3267

Fig. 10. Heating stage sequence showing helical dislocations growing at 280°C. The helices are attached to a foil surface at each end.

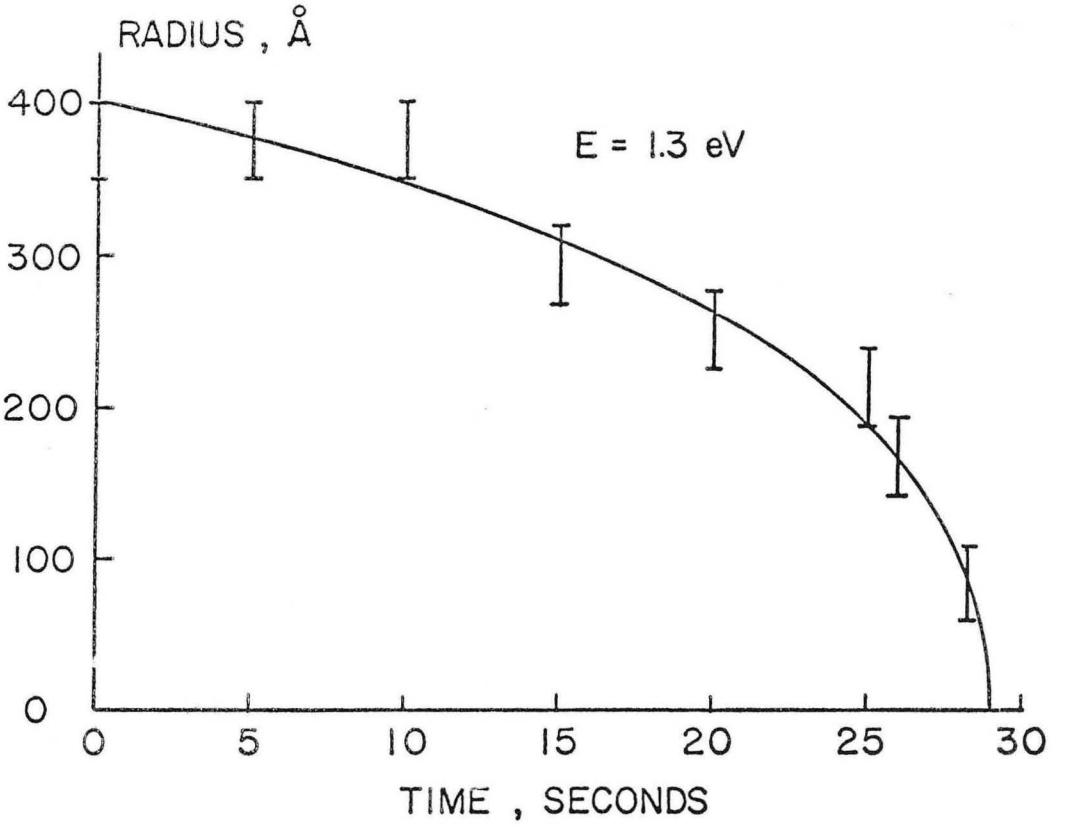


FIGURE 11. Decrease of prismatic loop radius with time at 240°C.

where, $E = E_m + E_f$

$t_o = r_o^2(z\nu b^2\alpha)^{-1}$

$r_o = 400\text{Å}$ at $t = 0$

$t = 29$ sec. to $r = 0$

$z = 11$

$\nu = 10^{13}$

$b = 2.86\text{Å}$

$\alpha = 50$ (estimated from Ref. 16)

$kT = 0.044$ eV at 240°C

The value calculated from Figure 11 is, $E = 1.3$ eV. This value is that for $E_m + E_f$ in pure aluminum which is consistent with the results. That is, the loop decreased in size indicating an inability of the material to support supersaturation. One would expect such to happen, if for example, the region around the loop was low or lacking in solute content.

DISCUSSION

A. Vacancy Supersaturation.

The apparent vacancy concentration associated with helices and columns of loops is about 3.7×10^{-5} in specimens quenched and aged at 20°C. This is an order of magnitude lower than that determined for pure aluminum[1,17] and Al-1% Mg[8] using similar techniques. The unalloyed aluminum was quenched from 580-600°C, but the Al-1% Mg alloy was quenched from 550°C. The calculated initial vacancy concentration, obtained by measuring the density and sizes of loops, after aging 60 minutes at 183°C was increased to 2.3×10^{-4}, that is, to a value one would expect for Al-5% Mg quenched from 550°C assuming $E_f = 0.76$ eV,

and E_b to be of the order of 0.15 eV. A low concentration estimated from quenched specimens aged only at room temperature indicates that these specimens contain uncounted vacancies, or a vacancy supersaturation.

The vacancy concentration C' in an alloy can be estimated if the vacancy-solute atom binding energy is known,[18]

$$C' = A \exp(-E_f/kT)(1-12c+12c \exp(E_b/kT))$$

where, E_f = vacancy formation energy in pure metal
E_b = vacancy-solute atom binding energy
c = atomic fraction solute atom
A = entropy factor
kT = Boltzmann factor

If C' is measured and, $C = A \exp(-E_f/kT)$ for pure metals, is either calculated or measured, the ratio C'/C can be used to estimate E_b by,

$$\exp(E_b/kT) = \left(\frac{C'}{C} - 1+12c\right) \frac{1}{12c}.$$

Using $C = 1\times10^{-4}$ and values for C' from Table II for 60 min. at 183°C, the binding energies in Table III were calculated. A reasonable lower limit for E_b is 0.1 eV. An isolated large helix which was included

TABLE III
ESTIMATED VACANCY-MAGNESIUM ATOM BINDING ENERGY

Specimen Treatment	C'/C	E_b
Q550° & aged 60 min. at 183°C		
Av. both loops and helices	2.3	0.08 eV
Single large loop	3.1	0.10
Single large helix	6.3	0.16

to represent an upper limit gives E_b = 0.16 eV. The volume element used for large helix was the same as that used for the other density determinations. A value of E_b = 0.2 eV has been estimated from climb sources in Al-Mg alloy.[19] Climb sources are found in Al-Mg alloy

after a slow silicone oil quench which should allow more vacancies time to precipitate.

The ratio of the concentration of vacancies associated with magnesium atoms to the concentration associated with aluminum atoms can be calculated as a function of the solute atom-vacancy binding energy. This calculation has been made for Al-5 at.% Mg at both 550°C and 20°C for E_b = 0.1 eV and 0.2 eV.[20] The ratio at 20°C for E_b = 0.1 eV is approximately 10^2, or, assuming the total quenched in concentration in Al-5% Mg to be $\sim 1x10^{-3}$, there are approximately $1x10^{-5}$ unassociated vacancies just after quenching. The ratio at 20°C is an order of magnitude higher for E_b = 0.2 eV giving $1x10^{-6}$ unassociated vacancies immediately after quenching. These concentrations are lower than the measured concentration of $3.7x10^{-5}$ (from Table II). The fraction of vacancies not associated with magnesium atoms should come out of solution first, followed by vacancies which are associated with magnesium atoms and come out, as magnesium-vacancy pairs sink at dislocations.

A binding energy in the range, 0.1-0.2 eV, gives reasonable correlation with vacancy concentrations measured for Al-5% Mg quenched from 550°C if the vacancies remaining in solution are considered. Those remaining in solution after only room temperature aging appear well accounted for after an additional 60 minute anneal at 183°C. The fraction of vacancies referred to as associated and thought to remain in solution still represents a considerable room temperature supersaturation, i.e., the concentration in equilibrium at 20°C is of the order of 10^{-11}. Rather than decay slowly, the high concentration could be maintained if vacancies exist in tightly bound magnesium-

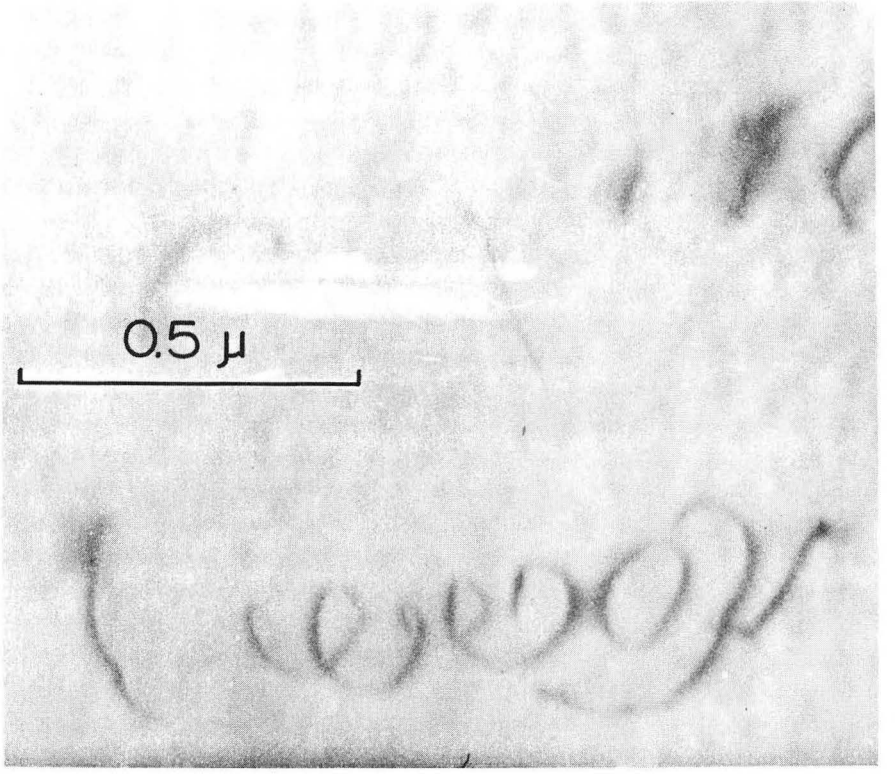
vacancy clusters at 20°C. It is also possible that uncounted vacancies exist in small unresolvable loops.

B. Helix Climb

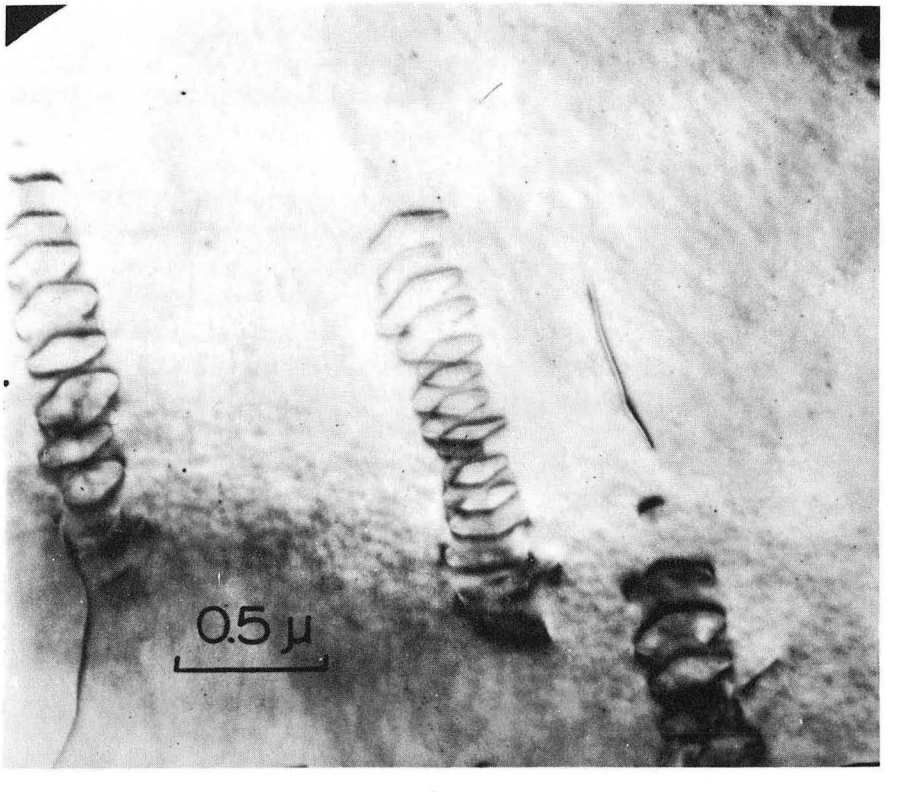
Columns of prismatic dislocation loops which are observed in quenched Al-5% Mg appear to have formed from helices by some mechanism. The interaction of a gliding screw dislocation with a helix having the same Burgers vector, and the interaction of adjacent helices of opposite sign have been discussed.(7,12,21,22) The latter case would result in prismatic loops having twice the diameter of original helices, as is found in electron micrographs. One would also expect to find some trace of gliding screw dislocations after the first reaction had occurred. These mechanisms do not appear to account for many of the present observations.

Two examples of loops which have formed within helices are shown in Figure 12. In both cases the segment of dislocation which is repelled by the loop after helix degeneration is evident. Figure 13 shows a helix along which adjacent line segments appear ready to combine at X. The straight segment Y is parallel to the [110] trace and may be pure screw. Figure 14 is a heating stage sequence suggesting degeneration of a single helical turn into a loop. The extra line segment, at Z, pulled away and the loop subsequently grew. These results imply that the degeneration of helicoidal lines into prismatic loops can occur without interaction with a second dislocation.

The three cases illustrated in Figures 12 and 13 are not uniformly developed, a feature which may be important to loop formation. Local fluctuations in vacancy or solute atom concentration, or stress fields may allow one segment of helix to climb at a faster rate than neighboring



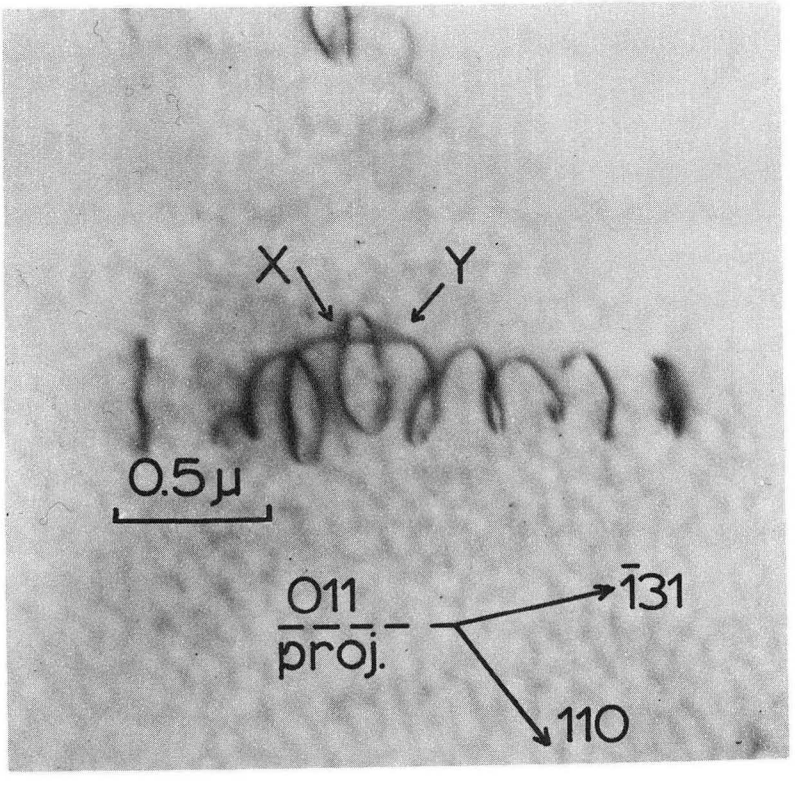
-a-



-b-

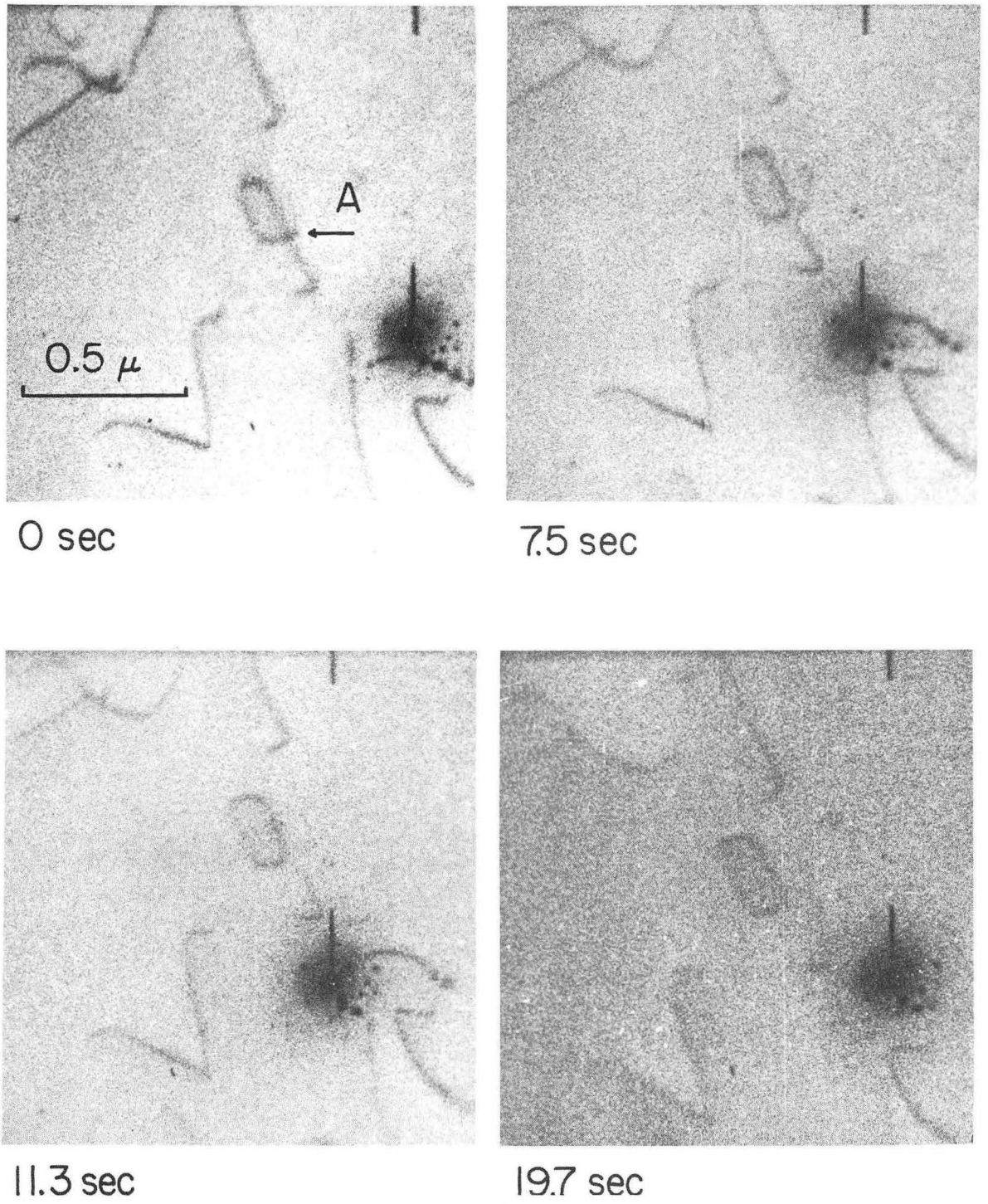
ZN-3975

Fig. 12. Loops along helicoidal axes in Al-5% Mg. (a) quenched from 550°C into ice-brine, aged 60 min. at 183°C and, (b) quenched from 520°C into 78°C water, aged 4 hours at 100°C.



ZN-3977

Fig. 13. Al-5% Mg quenched from 520°C into 78°C water, aged 60 minutes at 200°C.



ZN-3268

Fig. 14. Heating stage sequence showing a possible loop formation from single helical turn at "A". Foil temperature 280°C.

segments. A larger than normal turn, having a larger radius of curvature, should be less stable under the influence of existing stress fields. Considering the stress field associated with grain boundary precipitates, e.g., Figure 6, one might expect magnesium atoms clustered on a single dislocation to exhibit a similarly large stress. This is consistent with the observation that helices become angular and appear to increase in edge character along portions of the helical turn, during isothermal annealing as shown in Figures 3 and 5.

The effectiveness of stress in the process of forming columns of loops may be more important during quenching. Data in Table II indicate equivalent vacancy concentrations for both helices and columns of loops. One would expect that columns of loops originated from helices by a combined climb and glide process and not exclusively by climb. Some of the glide could be produced by quenching strains. Helices often appear in what could be described as bands emanating from grain boundaries, see for example Figure 2.

Uniform growth of helices in thin foils is shown in Figure 10. The helices are growing at 280°C, which is above the solubility limit for magnesium. It would seem more reasonable for a helix which terminates at both ends at a foil surface to act as a vacancy source and straighten out, at least near the emergent points. The vacancy concentration necessary to cause the observed growth could be supplied by vacancy-magnesium clusters which have been discussed.

C. Prismatic Loop Growth.

The activation energy for loop climb above 250°C was determined from isothermal loop growth data to be, $E_{(\dot{r})} = 0.95 \pm 0.05$ eV (Fig. 7). By assumming that the vacancy formation energy E_f' and vacancy migration

energy E_m' for the alloy are primary contributors to loop climb, then, $E_{(\dot{r})} = E_f' + E_m'$. If the vacancy-magnesium binding energy is equally effective in both E_f and E_m, which represent the most simple case, then $E_{(\dot{r})} = E_f + E_m - 2E_b$ where E_f and E_m refer to pure aluminum. Loop climb in pure aluminum was found to have an activation energy in the range 1.2-1.3 eV,[16] which gives good correlation with the activation energy for self diffusion in pure aluminum, E_D = 1.3 eV. (Average of three values reported in the literature: 1.2 eV,[24] and 1.33 $\pm$ 0.05 eV).[25]

Correlation of $E_{(\dot{r})}$ from the present work with E_D from the literature is reasonable, but not exact. For example, assuming E_b = 0.1 eV one would expect to measure $E_{(\dot{r})}$ = 1.1 eV which is higher than that obtained. A binding energy of the order of 0.17 eV would give good correlation. The assumption $E_{(\dot{r})} = E_f' + E_m'$ is valid for loop climb in thin foils, if the vacancy source is some form of vacancy-magnesium cluster or unresolvable loops. If, in the most simple case, it was possible for vacancy-magnesium pairs to provide the vacancy source for growth, the energy $E_{(\dot{r})}$ would include only E_m'. The binding energy, using $E_{(\dot{r})}$ = 0.95 eV and E_m = 0.54 eV, would be approximately 0.4 eV which does not agree with results from bulk annealing experiments.

The single case of negative loop climb observed during this study gave $E_{(-\dot{r})}$ = 1.3 eV which is in good agreement with the literature if the interpretation given is valid. That is, the loop may have been surrounded by a solute deficient matrix so that supersaturation was not possible.

CONCLUSIONS

1. The magnesium-vacancy binding energy was estimated to be 0.1 eV (lower limit) from bulk annealing experiments. The uncounted vacancies remaining after only aging at 20°C are thought to exist as magnesium-vacancy clusters or small unresolvable loops.

2. The magnesium-vacancy binding energy was estimated to be 0.15 eV from thin foil annealing experiments.

3. Dislocation loops and helices appear to grow in thin foils just as they do in bulk specimens.

4. Helices appear to degenerate into columns of prismatic loops without interaction with a second dislocation.

5. The helical dislocations and prismatic loops did not act as preferential sites for precipitation during the aging treatments used. The only observable consistent precipitation occurred at grain boundaries.

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