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CLIMB OF 1/3 ⟨111⟩ DISLOCATIONS IN GOLD

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ABSTRACT

The annealing behavior of large stacking fault loops in thin foils of gold was studied by transmission electron microscopy. The loops remained angular during shrinkage, maintaining sides accurately parallel to the ⟨110⟩ directions. Climb rates of individual sides of the loops were measured making use of small stacking fault letrahedra as fixed reference points. The climb rate of sides terminating exclusively at 60° corners was frequently less than that for segments bounded by at least one 120° corner. The climb rate of very short sides bounded by two 120° corners was often more than twice that of long sides. Distance to the foil surfaces did not have any influence on climb rate. It was suggested that the rate controlling step for climb of 1/3 ⟨111⟩ dislocations is nucleation of jogs at loop corners. An apparent activation energy of 1.93ev was associated with the climb process.

I. INTRODUCTION

In many FCC metals excess vacancies are eliminated after quenching by nucleation and growth of faulted dislocation loops. An area of intrinsic stacking fault is bounded by a 1/3 ⟨111⟩ imperfect dislocation [Hirsch et al.[1], and Silcox and Hirsch[2]]. When the diameter of the loop is large, >1000Å and the vacancy concentration is near the equilibrium value. the climb force acting on the dislocation is almost exclusively that due to the stacking fault. Therefore, the loops constitute a simple system in which to study dislocation climb; a pure edge dislocation which is acted upon by an almost constant climb force. In a thin foil, shrinkage of the loops takes place by loss of vacancies to the foil surfaces.[3] For slow climb rates, $<10^{-8}$ cm/sec., accurate data on the temperature dependence of climb rate can be obtained because heating of the specimen can be done outside the microscope. During the period of loop shrinkage, the specimen is periodically cooled and returned to the microscope to record loop size. Care must be taken to reproduce accurately the diffraction contrast conditions and magnification. Climb of faulted loops in aluminum has recently been studied using this technique by Dobson et al.[4], and by Tartour and Washburn[5]. Both sets of investigators found that the climb rate was sensitive to the distance between the shrinking loop and the foil surface. In aluminum, the loops exhibit no tendency to maintain angular shapes during shrinkage. In most cases, they become accurately round and the measured shrinkage rates show little scatter from one loop to another when the distances to the surfaces are comparable. These observations are consistent with the idea that the primary rate controlling step for dislocation climb in aluminum is the diffusion of vacancies away from the immediate

neighborhood of the loops as suggested by Siedman and Balluffi[6].

Climb behavior in gold is entirely different. Yokota and Washburn[7] have shown that planar faulted defects which have grown as such by capture of excess vacancies or have been formed by collapse of large stacking fault tetrahedra maintain straight sides parallel to ⟨110⟩, while shrinking. The shrinkage rates are sometimes markedly different for neighboring faulted loops. Even for a given loop, the shrinkage rate sometimes suddenly increases. There is no tendency for loops, close to the surface or even those that are cut by the surface to shrink faster than those near the interior of the foil. It is the purpose of the present work to study this seemingly eratic climb behavior in gold in more detail.

II. EXPERIMENTAL PROCEDURE

The specimen and thin foil preparation procedures have been described in a previous paper[7]. As in the earlier study, specimens suitable for quenching were prepared from 75 micron thick gold sheets of 99.999% purity. These specimens were quenched from 1000°C into an ice-water bath and aged for several hours at a temperature of 150°C. This treatment produced many large stacking fault triangles in the range 1000 to 2000Å, as well as much smaller stacking fault tetrahedra. Thin foil samples suitable for transmission electron microscopy were prepared by electropolishing utilizing a two solution window technique.

A Siemens Electron Microscope operated at 100 kV with a beam current less than 10 μamps was used to make all of the microscope observations. A detachable specimen cap constructed of gold and 100 mesh gold grids were used with the standard Siemens single tilt specimen holder and stage. The detachable cap permitted the annealing of the thin foil specimens in argon in a furnace outside of the microscope. Construction of the assembly entirely of gold minimized the chances of contamination and of plastic deformation which might have resulted from differential thermal strains during heating and cooling. The specimen was fixed stationary in the cap by interweaving the pair of ductile gold grids around the specimen. The hot stage technique in which a specimen is heated directly in the microscope was used in an initial study to determine the gross qualitative features of the annealing process.

Because a single tilting stage was used, the necessary diffraction conditions were obtained by making slight adjustments in the specimen orientation by rotating the grids within the cap before inserting the

specimen holder into the microscope. A number of trials were necessary initially to achieve the desired diffraction condition, but once obtained, it was relatively simple to achieve the same orientation a second time. Recording was on Kodak electron image plates. Locating of the same general areas for repeated observations was facilitated by making use of unusual features such as bend contours, or dirt spots. Then comparison of the field of view with developed plates of previous exposures made possible exact selection of the same areas.

Magnification errors could be corrected in the final printing because in the temperature range studied, stacking fault tetrahedra could be used as fixed reference points. These reference points also permitted independent measurement of the climb rate for each side of a polyhedral loop.

II. EXPERIMENTAL RESULTS

1. Shrinkage of Hexagonal Loops

Figure 1 shows several stages in the shrinkage of an irregular hexagonal stacking fault loop in gold. Because the loops remain angular, measurement of shrinkage rates is more complicated than for aluminum where the single parameter, loop diameter, can be used. In the present work, climb of each side of the loops was measured independently. This required three fixed reference points in the vicinity of the loop being studied. By carefully superimposing micrographs taken at each successive annealing stage, the amount each loop side had moved could be determined. Utilizing the above procedure the shrinkage behavior of the loop of Fig. 1 is shown in Fig. 2 as [111] projections of the successive stages. The displacements of all sides of the loop were approximately equal with the exception of the short side DE: Its climb rate was about twice that of the other five sides.

Occasionally jogs on an originally straight side piled up to form a resolvable step as on sides AF and FE. This requires the trapping of a jog so as to start a pile up or bunching of elementary jogs and could result from an impurity atom which might inhibit vacancy formation. The climb behavior of the resulting super jogs suggests that the re-entrant corner does not act as a source of elementary jogs.

The position of the foil surfaces relative to the various sides of the loop did not influence the climb rates. For example, sides AF and CD were closest to and parallel with the foil surfaces, but did not climb faster than sides AB, BC, or EF.

2. Climb of Triangular Stacking Fault Loops

In Fig. 3, compare the shrinkage rate of the triangular and five sided loop. The climb rate for the sides of the triangular loop is negligible compared to that for all sides of the other defect. Numerous observations of this kind lead to the conclusion that the climb of a 1/3 ⟨111⟩ dislocation segment bounded exclusively by 60° corners is quite different from that of one which is bounded by at least one 120° corner. For low and medium stacking fault energy materials such as gold, the 1/3 ⟨111⟩ Frank dislocation loop lowers its total energy by dissociating into a 1/6 ⟨110⟩ stair rod and a 1/6 ⟨112⟩ Shockley partial.

In the case of the 60° corner, the two Shockley partials are on the same side of the plane of the loop; the corner is closed by a 1/6 ⟨110⟩ stair rod segment which connects their junction with the corner of the triangular fault. At a 120° corner the Shockley partials are on opposite sides of the loop plane which necessitates a constricted junction with the two Shockley partials meeting at the plane of the loop.

The slower climb rate for the sides of a triangular loop as compared to the five or six sided loops suggests that the climb rate is controlled by nucleation of jogs at the corners. It should be more difficult to nucleate jogs at the extended 60° corners.

3. Annomalous Rapid Shrinkage Rates for Many Triangular Loops

An annealing sequence showing a number of stacking fault tetrahedra and loops is shown in Fig. 4 and schematically in Fig. 5. For loops 8 and 9 jogs apparently did nucleate at one of the triangular loop corners. This observation appears at first to be in direct contradiction with the conclusion that a 60° corner should be a poor jog nucleation site because the climb rates for the loop sides adjacent to the active 60° corners were of

the same order as those observed, for the long sides of loop 16 which were bounded by 120° corners. However, the active corners of loops 8 and 9 may actually have the configuration CC' shown in Fig. 6. If, as in Fig. 1, the climb rate for a short unresolved side is no more than twice as great as the two adjacent long sides then it will always remain short and might never become resolvable. Those triangular loops such as 12 which did not shrink at all during 10 hours of annealing may have had true 60° corners. Loops 4 and 5 which did not start to shrink until after 2 to 6 hours of annealing respectively initially could have had 60° corners, but somehow a forth side was formed after a time so that they then started to shrink.

Some newly formed short sides were observed to climb at rates far greater than twice that of the long sides. It is not yet clear how this different behavior can be explained. It suggests that impurities may be significantly affecting the climb rates.

Loops 1, 2, 6, 7, and 14 which are cut by the foil surface show that the point of intersection of a 1/3 $\langle 111 \rangle$ dislocation with the surface is not an easy jog nucleation site. Loops 2 and 7 were originally truncated letrahedra, but collapsed to loops after several hours of annealing.

4. Climb Model

A simple model that may explain the climb behavior of Frank dislocations in pure gold can be developed on the assumption that the rate controlling step for climb is nucleation of jogs. Assume that new jogs are always formed at corners and move along the sides by emission of vacancies. The spacing L, between jogs would be determined by the ratio of the frequency of vacancy emission at a jog to that at a corner or approximately:

$$L = \frac{a}{\sqrt{2}} \cdot \frac{2f_m}{f_n} = \sqrt{2}ae^{\frac{U_{fj}}{kT}} \qquad (1)$$

where U_{fj} is the jog nucleation energy, a, is the lattice constant, and f_m and f_n are the frequencies of vacancy formation at jogs and at loop corners, respectively. For sides much longer than L there will always be one or more jogs moving along the side. The steady state jog concentration will be about $\frac{1}{L}$ regardless of whether jogs are being nucleated at one or both ends of the segment; in the latter case, jogs nucleated at one end will be annihilated by meeting a jog that started from the opposite end. The dislocation climb rate will be:

$$V = \frac{\sqrt{3}\,Nv}{4\,L}\; e^{-\frac{U_{fv} + U_{mv} - \frac{\sqrt{3}\gamma a^2}{4}}{kT}} \qquad (2)$$

where U_{fv} and U_{mv} are the vacancy formation energy and vacancy migration energy v is the atomic vibration frequency, N is the coordination number, and γ is the stacking fault energy.

When a side is appreciably shorter than L, the climb rate should be greater than (2) for two reasons: On a side shorter than L, it may not be possible to have complete splitting of the Frank dislocation into a Shockley partial and a stair rod. In this case, the constriction energy term in U_{fj} decreases. Also, jogs nucleated at a corner between a side which is shorter than L and one which is longer than L will not have an equal probability of moving off along either side as was assumed in (1); the short side will be preferred.

5. Activation Energy for Dislocation Climb

From a measurement of the climb rate of a given dislocation segment at two or more temperatures, an activation energy for climb can be determined. Abrupt changes in the temperature were made during the course of annealing and the climb rates before and after the change were compared. The curve of

Fig. 7 was obtained in this way utilizing three temperatures. Because of the linear rate of climb of the loops, the entire annealing time at each temperature could be used to determine the rates. The identical slopes of the curve at 400°C for the beginning and end sections suggests that irreversible changes in the mechanism of climb had not taken place during the experiment. The log of the climb rates are plotted against the reciprocal of the absolute temperature in Fig. 8. The apparent activation energy for dislocation climb was 1.93 ev and the pre-exponential factor was 2.78×10^{13} Å/sec. $U_{fv} + U_{mv}$ from high temperature self diffusion measurements is 1.80 for gold. If this value is assumed, it leave 0.13 for the jog nucleation energy at a 120° corner of a 1/3 ⟨111⟩ dislocation. The corresponding spacing between jogs at 300°C would be about 80Å. The experimental observations show that an increase in climb rate occurs for sides at least an order of magnitude longer than 80Å, which suggests that 0.13 may be an underestimate of the true jog nucleation energy.

IV. CONCLUSIONS

Some generalizations can be made concerning the annealing behavior of stacking fault loops in gold:

i. Stacking fault loops in gold remain angular during shrinkage and do not tend to become rounded as do hexagonal stacking fault loops in aluminum.

ii. The climb rate of a loop side is independent of its length above a critical length. It is suggested that this critical length may be associated with the spacing between jogs.

iii. Within the temperature range investigated, the rate of climb of sides bounded exclusively by 60° corners is much less than that of sides terminated by at least one 120° corner.

iv. The distance to the foil surface does not affect the rate of shrinkage. Even sides cut by the surface climbed at normal rates. Therefore, jogs are not easily nucleated where a Frank dislocation terminates at a surface.

v. The apparent activation energy for dislocation climb in gold is 1.93 ev, which when compared with the high temperature self diffusion value of 1.80 ev gives 0.13 as the jog nucleation energy for a 1/3 ⟨111⟩ dislocation at a 120° corner.

vi. Impurity pinning of jogs is suggested by some of the experimental observations.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

Fig. 1 - Shrinkage of a large six sided stacking fault loop.

Fig. 2 - Relative displacements of the individual sides of the loop shown in Fig. 1.

Fig. 3 - Comparison between climb rates for a triangular and a five sided loop.

Fig. 4 - Sequence at 300°C showing tetrahedra and triangular loops after. a) 0, b) 2, c) 4, d) 6, e) 8, f) 10, and g) 12 hours of annealing.

Fig. 5 - Relative displacements of individual sides of some loops in Fig. 4.

Fig. 6 - Dislocation configurations at sharp 60° corners A and B, and at 120° corners C and C'.

Fig. 7 - Temperature dependence of climb rate.

Fig. 8 - Estimate of apparent activation energy and pre-exponential factor from data of Fig. 7.

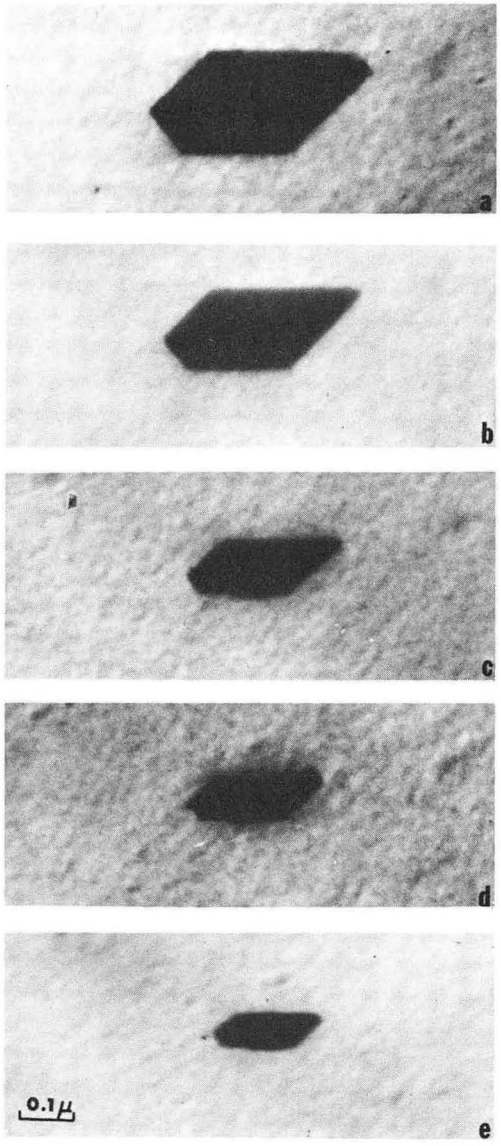

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Fig. 1

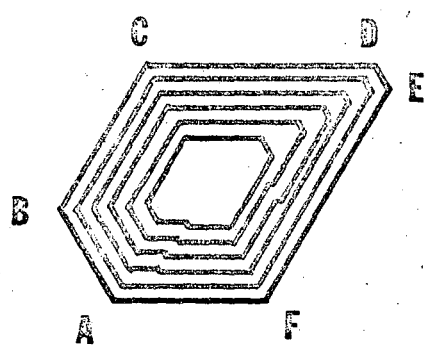

Fig. 2

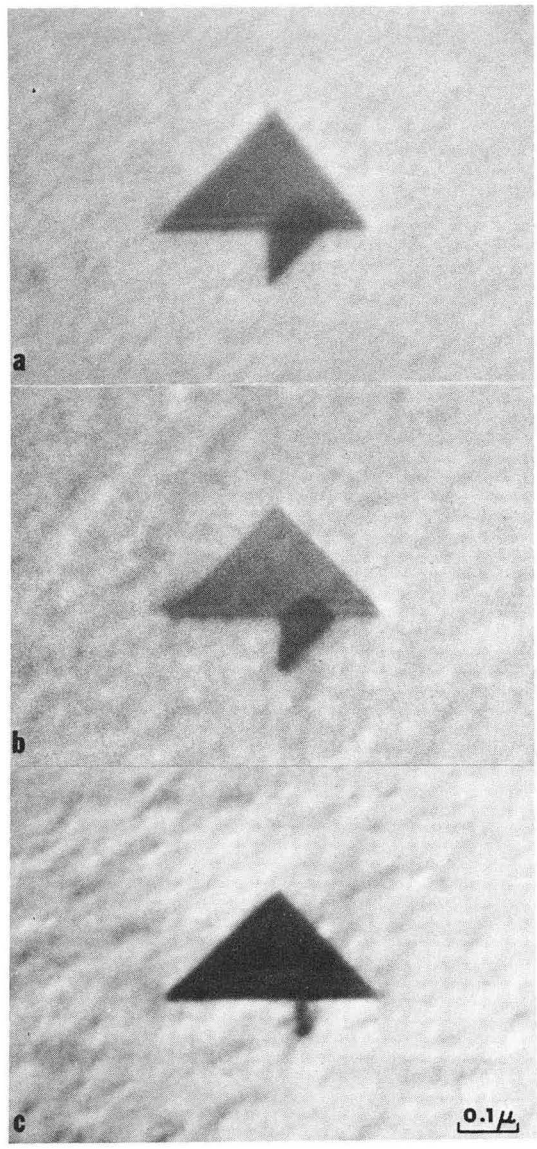

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Fig. 3

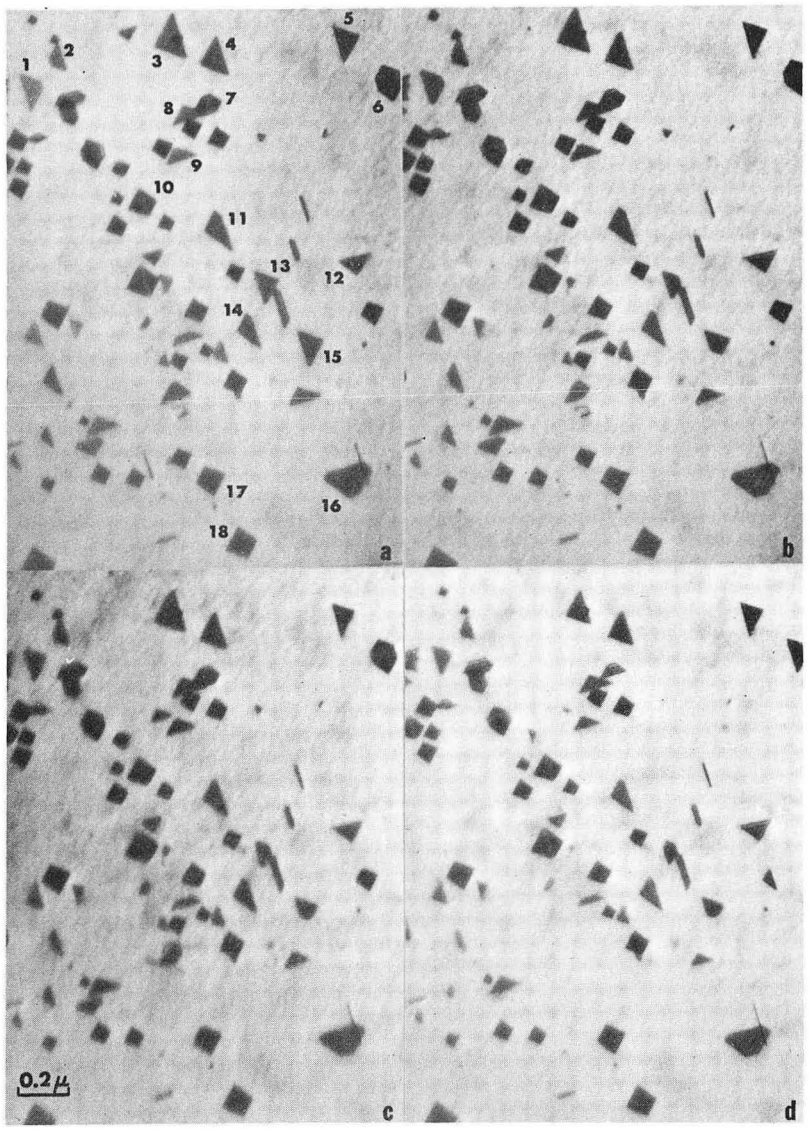

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Fig. 4 a-d

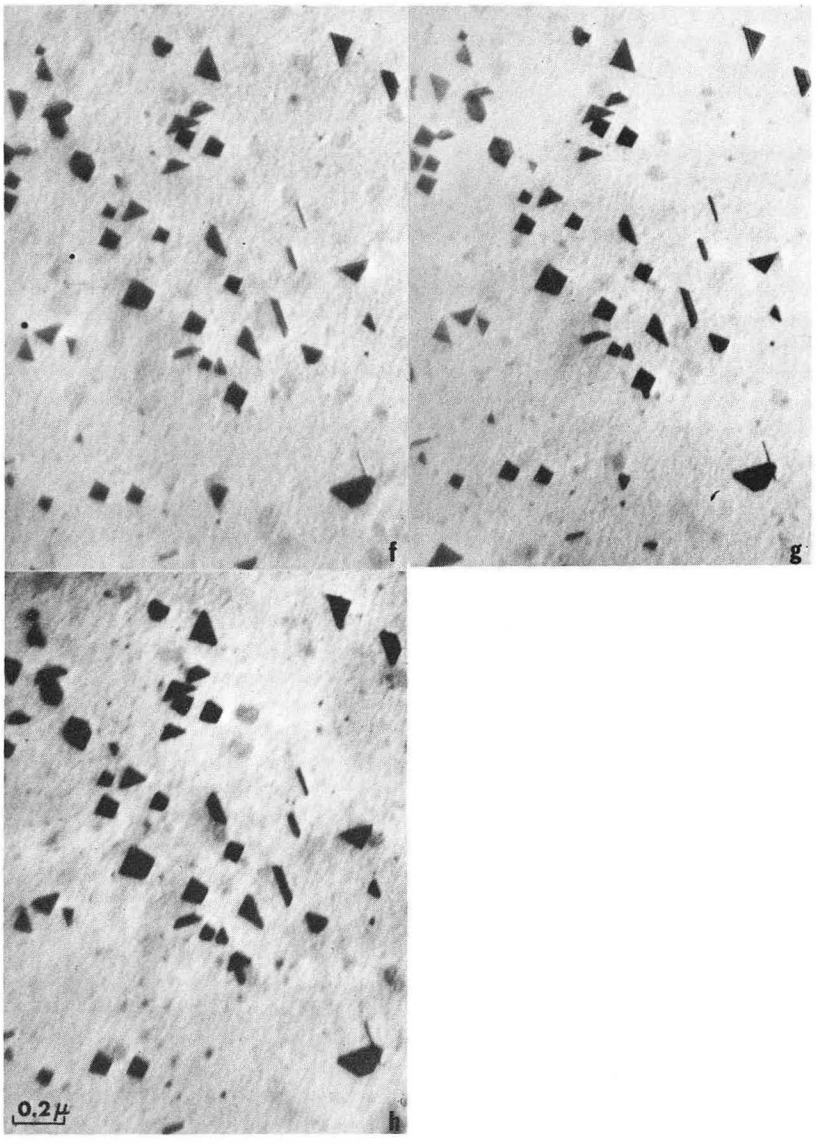

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Fig. 4 f-h

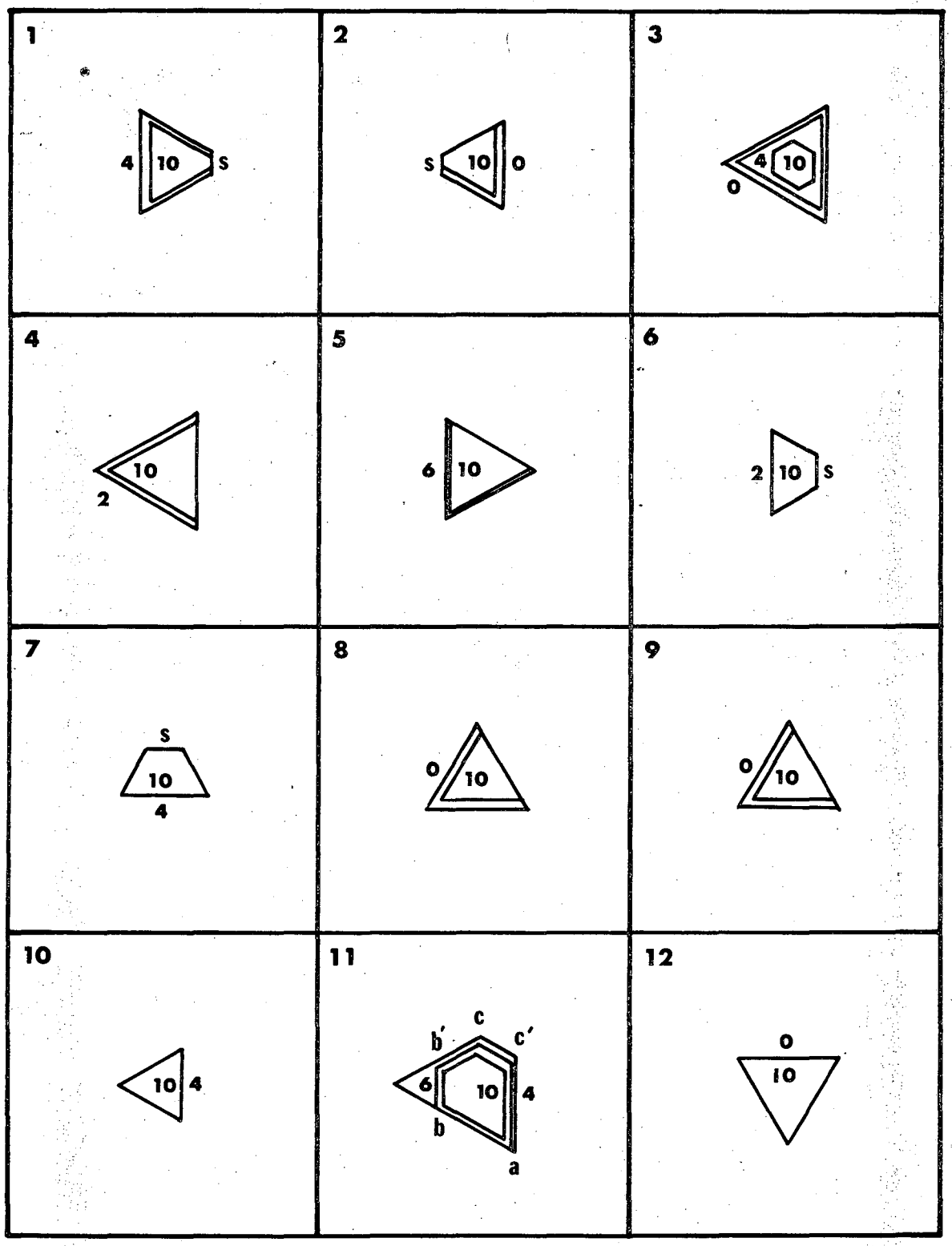

Fig. 5 XBL 686-1126

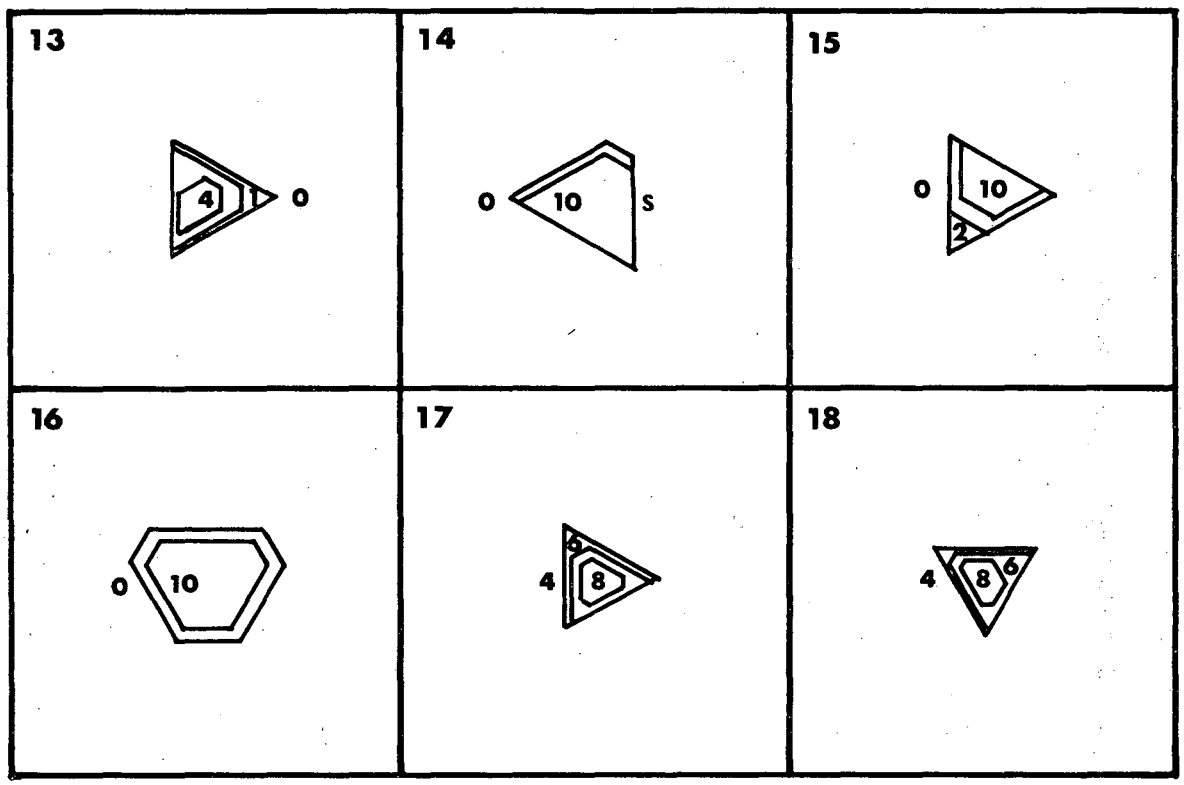

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Fig. 5 (cont.)

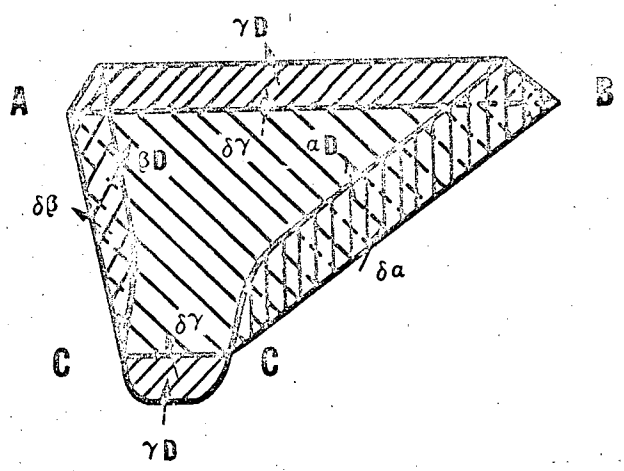
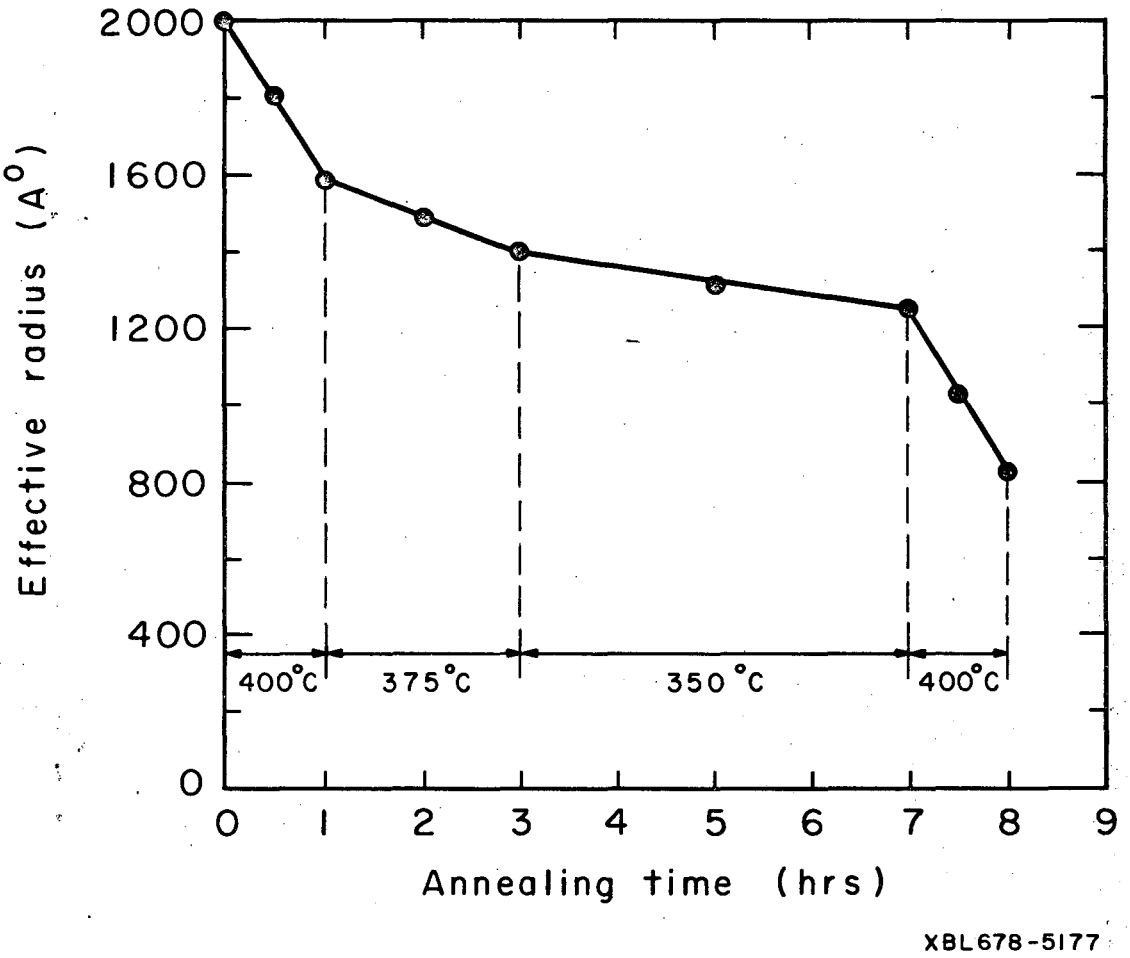


Fig. 6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1200
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400
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Effective radius (A°)
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350°C
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1
2
3
4
5
6
7
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9
Annealing time (hrs)
XBL678-5177

Fig. 7

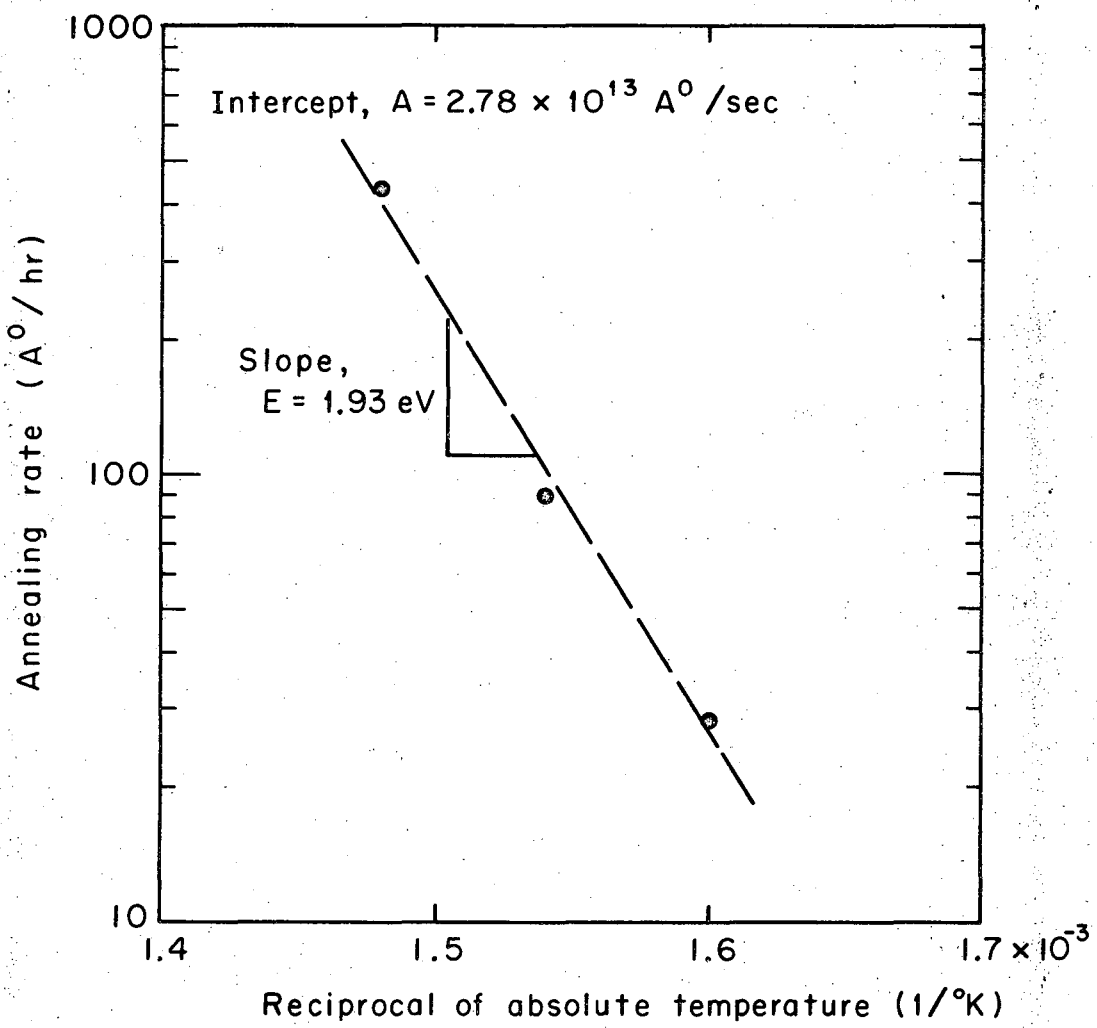

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Fig. 8

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