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ABSTRACT

Results of quantitative measurements of dislocation climb rates in aluminum gold and magnesium oxide are briefly reviewed. Cases have been chosen for which the climb force and the diffusion geometry are known. Individual climbing segments of edge dislocation lines were followed by transmission electron microscopy of the same areas of thin foils after repeated holding times at the climb temperature. In aluminum and in magnesium oxide climb rate was shown to be diffusion controlled. In gold climb rate is emission controlled and varies widely from one segment to another even though the climb force may be the same. It is concluded that impurity pinning plays an important role in this case. In magnesium oxide self-climb by migration of point defects along the core of the climbing dislocation was found to be an important process below about 1300°C.

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INTRODUCTION

At temperatures above about half the absolute temperature of melting nonconservative motion of dislocations becomes important in crystalline materials. Edge and mixed segments can move in the direction at right angles to their Burgers vectors by absorbing or emitting vacancies. Dislocation climb is the cause of recovery in cold worked crystals and frequently is the rate controlling process for high temperature creep. In heavily irradiated metals the climb of edge dislocations due to attraction and absorption of the excess interstitials and vacancies results in void growth and the consequent radiation induced swelling that occurs in the temperature range 0.3 Tm to 0.5 Tm.

In spite of its basic importance to an understanding of high temperature properties of materials there is still little experimental information concerning the climb of dislocations in particular materials under various magnitudes of the driving force and temperature. Particularly the effects of stacking fault energy and impurities on climb rates are still largely unexplored.

For the climb that occurs during recovery or creep the climb forces that act on each individual segment of dislocation are complex and impossible to determine. The local tensile or compressive stress component

acting along the direction of the Burgers vector is the sum of the applied stress and all the internal stresses due to near-by dislocations. This includes adjacent segments of the climbing dislocation itself if it is not straight. For mixed dislocations glide forces acting on the screw component may also make an important contribution to the total climb force. Under the action of climb stresses the local equilibrium concentration of vacancies near a dislocation is changed. Unless this local equilibrium is continuously disturbed by diffusion, climb ceases because rates of emission and absorption become equal. When local equilibrium is nearly maintained climb rate is "diffusion controlled"; it depends on the climb force and on the distances to the near-by vacancy sinks or sources. Alternatively, when the vacancy concentration gradients nearly vanish then climb rate becomes "emission controlled." At a given temperature climb rate is then dependent on the climb force and factors which affect rate of emission or absorption.

Diffusion geometry is usually also complex. In a dislocation tangle some segments act as sources and some as sinks for vacancies. It is also complicated by the fact that the more open structure of dislocation cores provide an easier diffusion path than through the matrix. "Pipe diffusion" along dislocations is particularly important at the low end of the diffusion temperature range. In some cases it becomes the predominant mechanism for dislocation climb. When only pipe diffusion occurs atoms are shifted exclusively along the same or connecting dislocations. Unless the dislocation segment terminates at an external surface of the crystal this kind of climb can not change the total area

of the extra half plane as projected onto the plane perpendicular to the Burgers vector; it has been called "self climb" or "conservation climb."

In materials for which low energy stacking faults exist, climb is further complicated by the fact that dislocations split into partials with a ribbon of stacking fault between. Emission or absorption of point defects may only be probable at nodes or jogs where the partials are recombined.

This paper is a brief review of some recent experimental work on dislocation climb in which the nonconservative motion of individual dislocation segments has been followed in the electron microscope. An attempt has been made to choose cases where the diffusion geometry is relatively simple and the driving force is easy to calculate. The simplest case is an isolated closed loop of edge dislocation in a thin foil. The driving force for climb is its own self energy which decreases with decreasing loop diameter. When the loop encloses a stacking fault the fault provides an additional climb force. If such a loop is vacancy type it shrinks by loss of vacancies to the foil surfaces; an interstitial loop shrinks by absorbing vacancies. By taking a series of electron micrographs of the same areas with intermediate holds in a furnace at accurately known temperature reliable measurements of climb displacement rates are possible. A smoothly operating double tilling specimen stage is essential in order to accurately reproduce diffraction contrast condition from one set of exposures to the next. Recording of stereo pairs makes it possible to measure distances from dislocation line to foil surfaces and is sometimes essential to be able to distinguish between climb and glide motions.

DISLOCATION CLIMB IN ALUMINUM

Figure 1 is a series of pictures obtained as described above showing shrinkage of faulted $\frac{1}{3}\langle 111 \rangle$ dislocation loops in pure aluminum.[1] For large faulted loops in aluminum the climb force is almost constant because the term due to the stacking fault is large compared to the contribution due to change in elastic strain energy.

The elastic strain energy term becomes increasingly important as loop size decreases, and the diffusion geometry also changes as loop diameter becomes small relative to the thickness of the foil. This is shown in Fig. 2 where the measured loop radii for two loops are plotted as a function of time at 353°K. The shrinkage rate accelerates as loop size decreases. Even in this simple case a model which can accurately predict loop radius as a function of time at a given temperature is fairly complex.

In particular simplifying assumptions are usually made concerning the changing diffusion geometry. For the measurements shown in Fig. 2 the validity of the assumption of cylindrical diffusion geometry throughout the measured range of loop size is confirmed by the fact that the measured shrinkage rate can be accurately represented by the model as the sum of a linear rate due to the stacking fault and a constantly accelerating rate due entirely to the change in elastic self energy of the loop. The total shrinkage minus the part expected from elastic self energy change is also plotted in Fig. 2 as solid circles.

If changes in diffusion geometry can be neglected, these points should fall on a straight line and represent the constant shrinkage rate that would have been due to the stacking fault alone. Using these

corrected shrinkage rates it was possible to accurately compare the rates
for loops near the center of the foil with those near a surface. Climb
rates were found to increase with decreasing distance to the nearest sur-
face resulting in a 20% scatter in measured shrinkage rate. Taking into
account the fact that large faulted loops in aluminum that are less than
about one loop diameter from the surface tend to be lost by conversion to
perfect loops and glide to the surface; this result shows that under the
conditions of the experiment climb rate was essentially "diffusion con-
trolled."

The observed climb rates correspond to a value of the self diffusion
coefficient, D_o = 0.025 cm^2/s and an activation energy for self diffusion
of 1.23 eV. By comparing shrinkage rates for perfect and faulted loops
the stacking fault energy was estimated as 110 ergs/cm^2. This high value
of stacking fault energy is consistent with the circular shape assumed by
shrinking $\frac{1}{3}\langle 111 \rangle$ dislocations in aluminum. The energy gained per unit
length by the splitting reaction

$$\frac{1}{3}[111] \rightarrow \frac{1}{6}[110] + \frac{1}{6}[112]$$
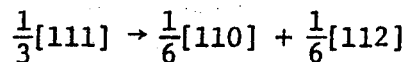
is insufficient to cause appreciable development of straight segments
parallel to $\langle 110 \rangle$ directions.

DISLOCATION CLIMB IN GOLD

Similar experiments have been done on climb of $\frac{1}{3}\langle 111 \rangle$ dislocations in gold.[2] In this case most of the loops are triangular in shape, having been formed by collapse of a tetrahedron.

Figure 3 is a series of pictures of the same area after 0, 1, 2, 4, 6, 8, and 10 hours at 300°C. The behavior is very different from aluminum. The sides all remain straight and parallel to $\langle 110 \rangle$ during shrinking. Climb rate is obviously not diffusion controlled. Some of the loops, including a few that actually were cut by a surface of the foil, did not shrink at all during 10 hours at 300°C. Others decreased in size rapidly, some of them disappearing entirely in a few hours. In this case climb rate was clearly "emission controlled." The observations can be explained by assuming that the critical step is nucleation of a jog at one of the corners.

Figure 4 shows a possible dislocation structure of a loop with four sides. Assuming a stacking fault energy of 40 ergs/cm^2 the dislocations are split to a width of about five atom diameters. Acute corners can be extended. Therefore jog nucleation is a difficult process which, probably occurs with greatest frequency at 120° corners such as at C. The fact that the sides remain straight during shrinkage suggests that once formed at a corner a jog emits vacancies rapidly enough to travel many interatomic distances before another jog is nucleated. In most cases one or more sides of the loop failed to climb at all during the 10 hours. This was determined by utilizing small tetrahedra which remained unchanged throughout the series as fixed reference points. The results of the measurements are shown in Fig. 5. Sides that were terminated by at least

one 120° corner always climbed. Those that appeared to end at two 60°

corners sometimes climbed and sometimes did not. It was concluded that in

the former case one corner was really a pair of 120° corners associated

with a short unresolved side. For such a short unresolvable side between

two 120° corners to persist during shrinkage it is necessary for its climb

rate to be exactly twice that of the two neighboring sides. This would

be expected if the distance traveled by a jog before nucleation of a second

jog that moves off along the same side is greater than the length of the

side. It is clear that in some cases short sides grow in length during

shrinkage as on loops 3, 13, 15, 17, and 18 in Figs. 3 and 5.

Some interesting questions remain to be answered concerning the

mechanism of climb of widely split dislocations where climb is clearly

emission controlled. In the case of gold Escaig[3] has shown that 60°

corners should not be sharp as shown in Fig. 4. Vacancies should be

emitted at 300°C. Therefore the apparent stability of some such corners

must have been due to the presence of particular impurity atoms with a

large binding energy to one of the unique sites near the point where the

three stair rod dislocations meet. In silver, in which $\frac{1}{3}\langle 111 \rangle$ dis-

locations are even more widely split, preliminary results show that

shrinkage of loops still begins at about the minimum temperature for self

diffusion. Therefore the mechanism of jog nucleation has not been

adequately explained.

SELF CLIMB OF DISLOCATIONS

Dislocation climb sometimes occurs almost entirely by transfer of

atoms from one point to another along the core of the climbing dislocation.

This process is "conservative climb" in the sense that the total area of the extra half plane associated with the dislocation is unchanged.

Recently quantitative measurements have been made which permit comparison of the temperature dependences and diffusion constants associated with self climb and ordinary nonconservative climb of dislocations in magnesium oxide.[4,5] In this case dislocation climb involves transport of both Mg^{++} and O^{--} ions. However because of the relatively large difference in ionic radii (O^{--} = 1.32 Å, Mg^{++} = 0.66 Å) it is likely that oxygen ion diffusion is the rate controlling process for dislocation climb. Two coplanar edge dislocation loops with identical Burgers vector attract each other strongly when the separation of their centers is not much more than the diameter of the larger loop.[6] At temperatures below about 1300°C in magnesium oxide two such loops climb toward each other without change in diameter. Several examples are shown in Fig. 6. Loop pair separations were measured with intermediate holding at several different temperatures. To maximize the precision of measured distances between dislocations, pictures were taken with both $\bar{2}00$ and 200 diffraction conditions and the dislocation was assumed to lie midway between the positions of the respective diffraction contrast images. Steriomicroscopy was done at each step so that loop pairs close to the center of the foil were always chosen for measurement. Assuming that the observed climb occurred entirely by pipe diffusion and that the only driving force was the interaction energy change that accompanies a change in loop separation, a value for the activation energy for pipe diffusion of 60,300±3500 cal/mol was obtained from the measured displacement rates of seven pairs of loops at several different temperatures. The observed climb rates corresponded

to a value of $D_o^P a^P$ of $(7.5\pm4.3)\times10^{-18}$ cm^4/sec. Taking a^P, the cross sectional area of the dislocation pipe, as 10^{-15} cm^2 the pipe diffusion coefficient D_o^P is 5×10^{-3} cm^2/sec.

For measurement of ordinary climb rate, isolated loops were chosen which on holding at temperatures above about 1200°C shrink by diffusion to or from the foil surfaces. Figure 7 shows a typical series on which measurements of climb rate were made. As in the case of aluminum these $\frac{1}{2}\langle110\rangle$ loops in MgO were found to be climbing under diffusion controlled conditions. Climb rate for a given loop depended on its distance from the foil surface. Since these are perfect loops the driving force is only the change in loop self energy. The temperature dependence of climb rate corresponded to an activation energy of 110,000±4200 cal/mol and the rate of climb to a value of the diffusion coefficient, D_o, of $1.37\pm0.26\times10^{-2}$ cm^2/sec.

It seems most reasonable to associate this value of activation energy and diffusion coefficient with intrinsic oxygen diffusion in MgO. If the activation energy for self climb is also associated with oxygen ion migration the ratio between activation energies for pipe and volume diffusion of oxygen in MgO is 0.55 which is about the same as the corresponding ratio in face centered cubic metals.[7]

CONCLUSION

The types of experiments that have been reviewed permit the measurement of dislocation climb rates under conditions where the driving force can be accurately calculated and the diffusion geometry is known.

Further application of such measurements, particularly to the investigation of solute and impurity atom effects on climb and self climb,

should help to increase understanding of more complex processes such as recovery and creep where dislocation climb is often the rate-controlling process.

ACKNOWLEDGMENTS

REFERENCES

1. Jean-Pierre Tartour and Jack Washburn: Phil. Mag., 1968, vol. 18, 1257.

2. J. Washburn and M. J. Yokota: Cryst. Lattice Defects, 1969, vol. 1, 23.

3. B. Escaig: Cryst. Lattice Defects, 1970, vol. 1, 211.

4. J. Narayan and J. Washburn: Phil. Mag., 1972-I, in press.

5. J. Narayan and J. Washburn: Acta Metallurgica, 1972-II, in press.

6. F. Kroupa, J. Silcox, and M. J. Whelan: Phil. Mag., 1961, vol. 6, 971.

7. D. W. James and G. M. Leak: Phil. Mag., 1965, vol. 12, 491.

FIGURE CAPTIONS

Fig. 1. Shrinkage of faulted $\frac{1}{3}\langle 111 \rangle$ dislocation loops in aluminum; pictures taken at 0, 213, 793, 1304 minutes at 101.8°C. Loops with sharp stacking fault fringe contrast are near one foil surface, those with uniform fault contrast are near foil center.

Fig. 2. Measured shrinkage rates for two $\frac{1}{3}\langle 111 \rangle$ loops in aluminum; at 80°C (triangles) and shrinkage rate that would have been expected due to the stacking fault alone (circles). ⬦

Fig. 3. Stacking fault tetrahedra (distorted square defects) and triangular $\frac{1}{3}\langle 111 \rangle$ dislocation loops in gold. Pictures taken after 0, 1, 2, 4, 6, 8, and 10 hours at 300°C. Some tetrahedra collapse to become triangular loops (10, 17, 18); some loops shrink rapidly (3, 13, 17, 18) and some not at all (7, 12).

Fig. 4. Dislocation configuration for a four sided $\frac{1}{3}\langle 111 \rangle$ dislocation loop in a face centered cubic crystal of low stacking fault energy. Cross hatched areas are intrinsic stacking faults.

Fig. 5. Measured displacements of individual sides of some loops in Fig. 3.

Fig. 6. Self-climb of $\frac{1}{2}\langle 110 \rangle$ loop pairs 1 through 5 in magnesium oxide; A → B 76.1 min at 1195°C; B → C 38.4 min at 1195°C; C → D 30.2 min at 1239°C (many intermediate pictures not shown).

Fig. 7. Climb of isolated $\frac{1}{2}\langle 110 \rangle$ dislocation loops in magnesium oxide by diffusion to or from a foil surface A → B 42.1 min at 1368°C; B → C 45.9 min at 1291°C; C → D 150.1 min at 1202°C; D → E 46.2 min at 1243°C. Many intermediate pictures not shown.
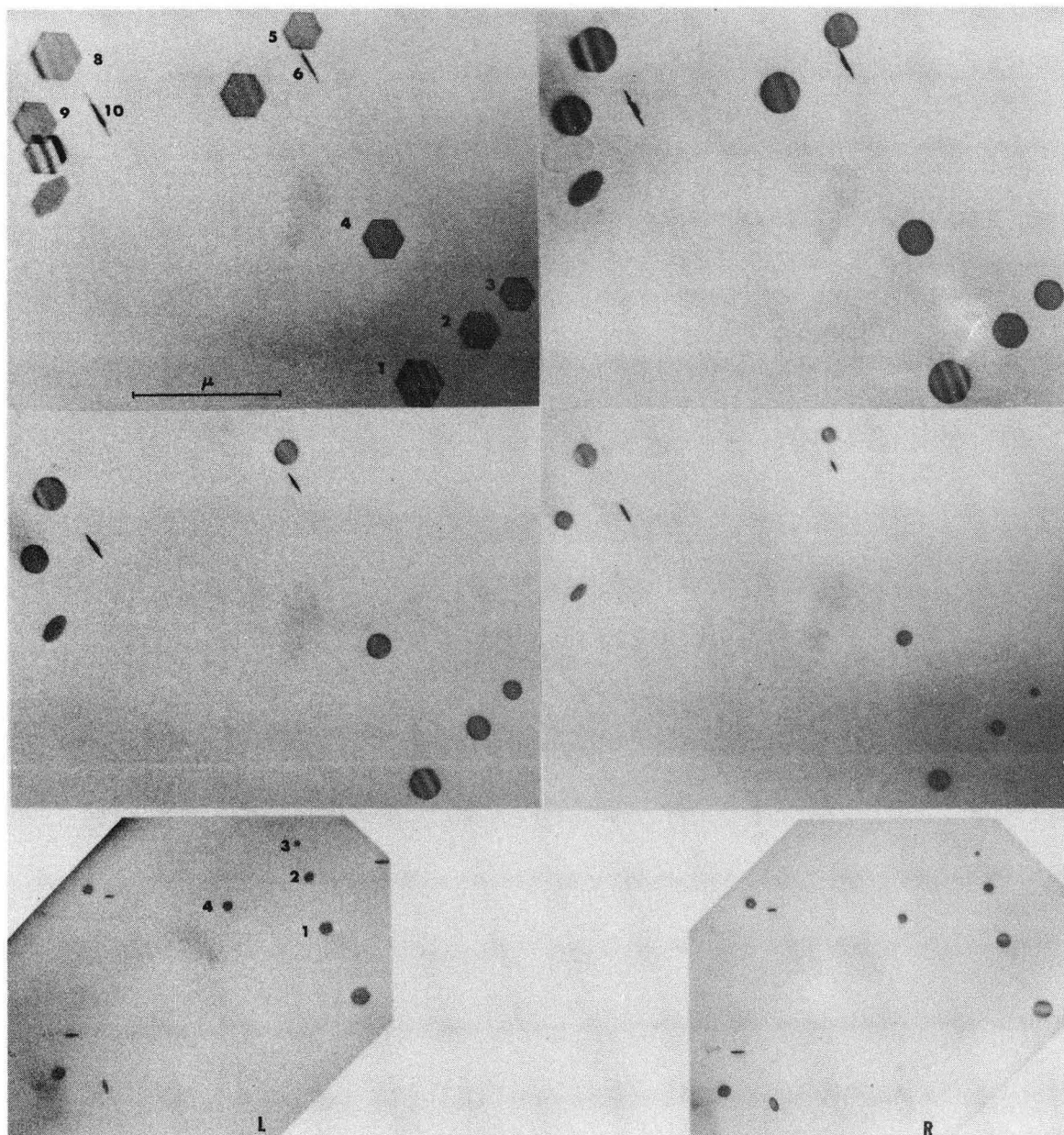
XBB 681-413

Fig. 1
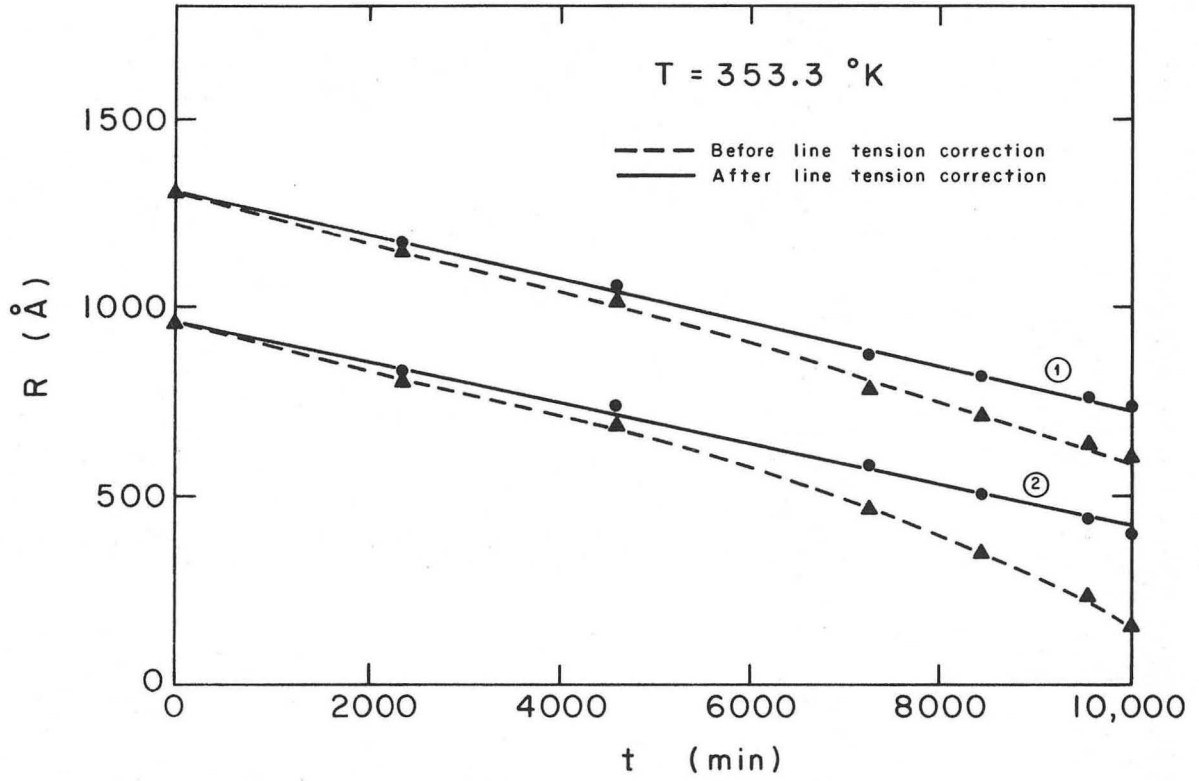
Fig. 2

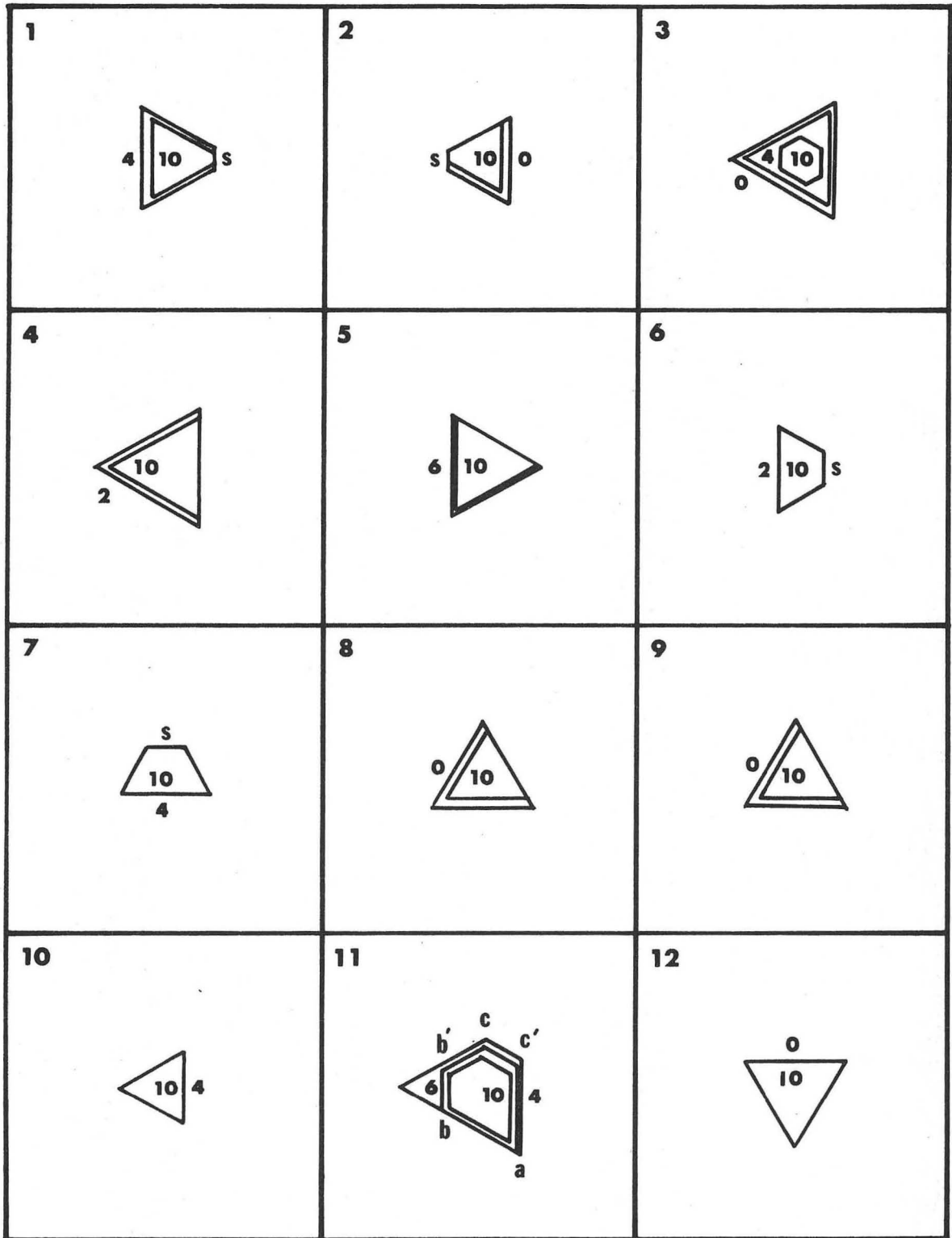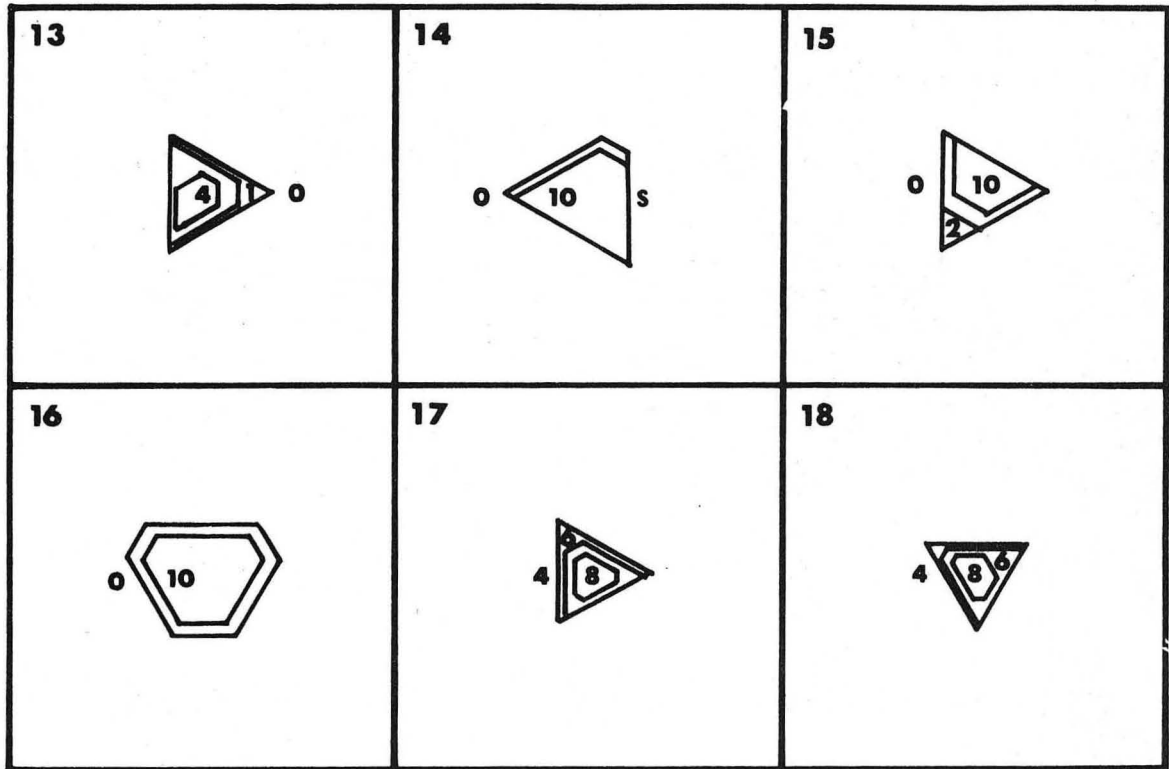XBB 686-3939A
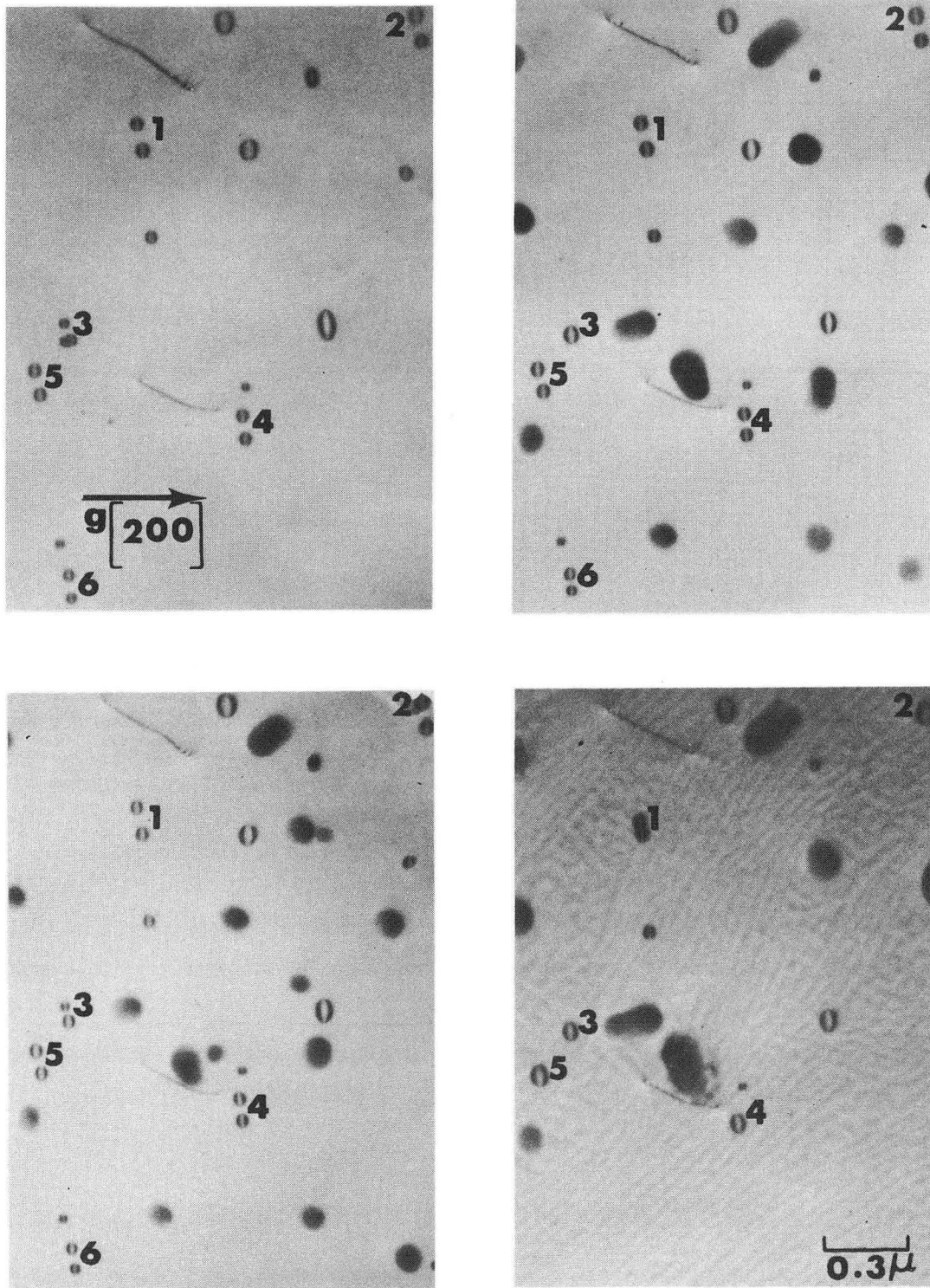
Fig. 3

0.2 μ
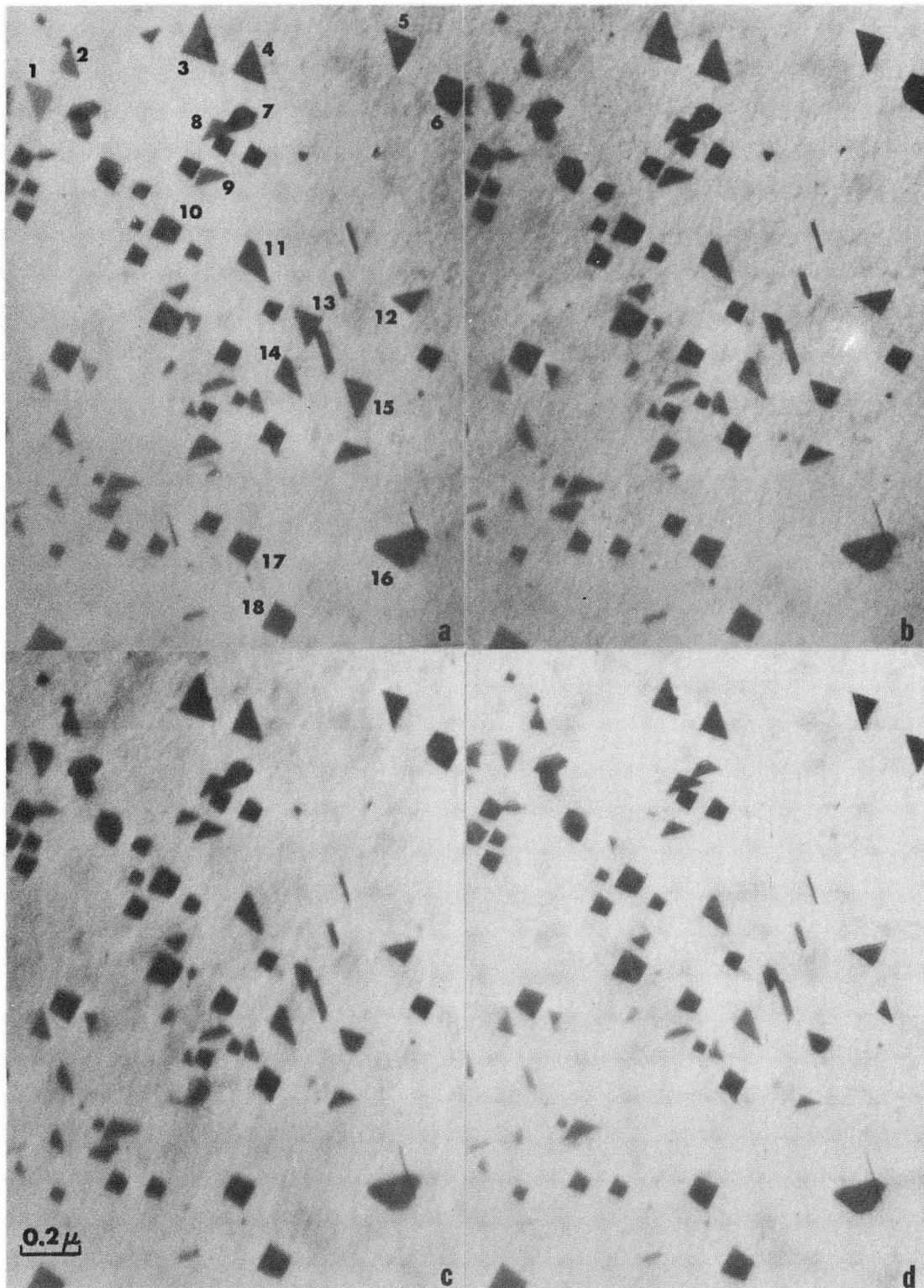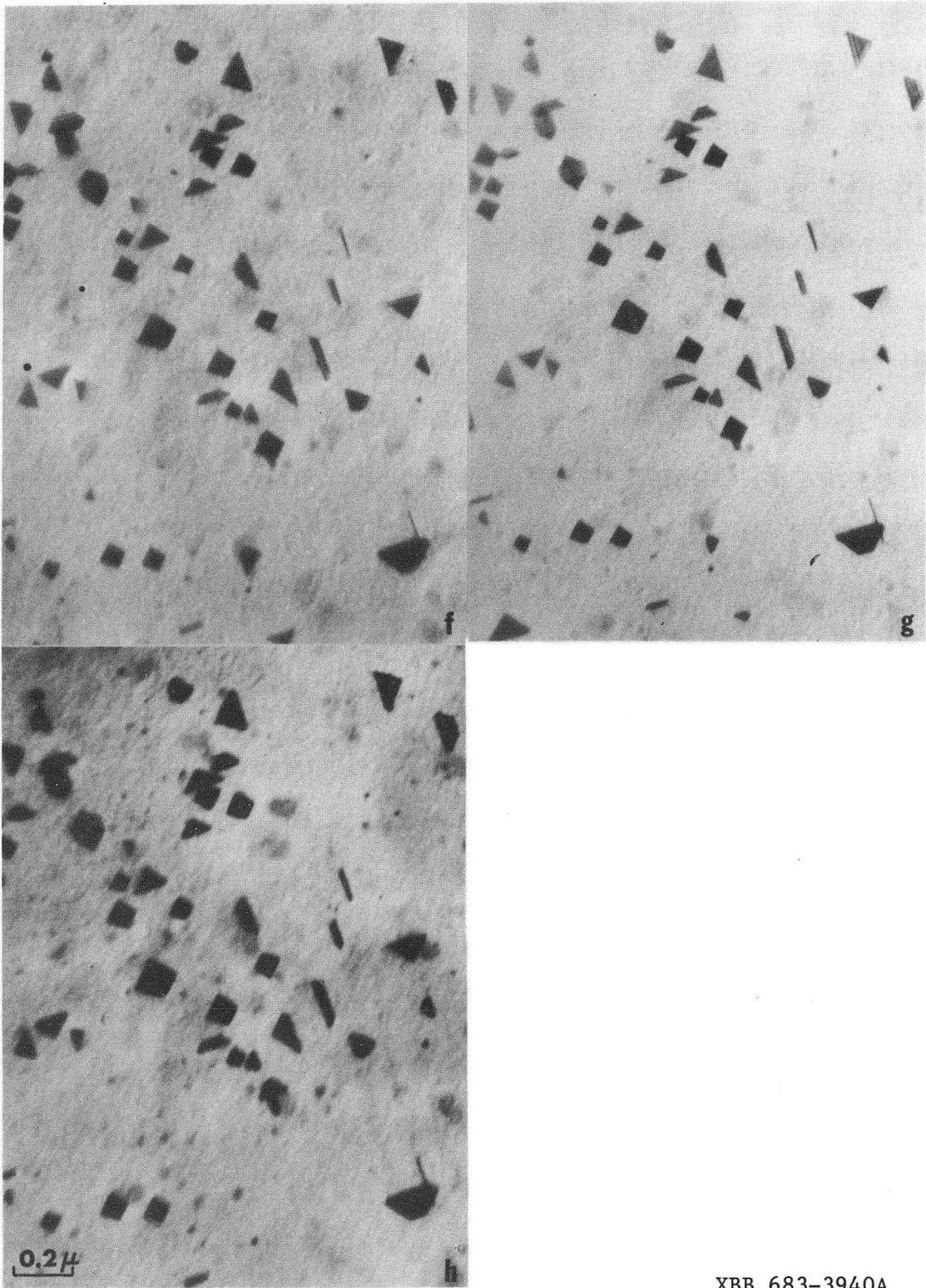
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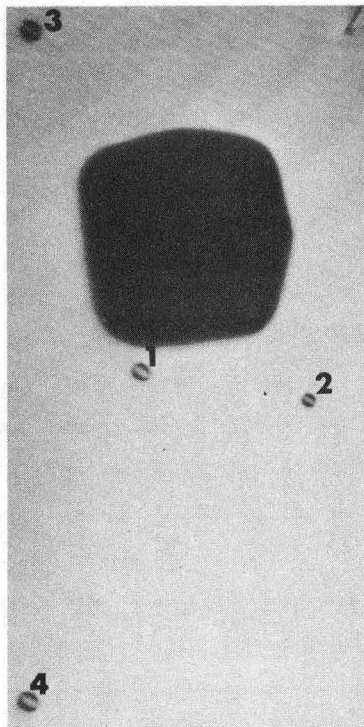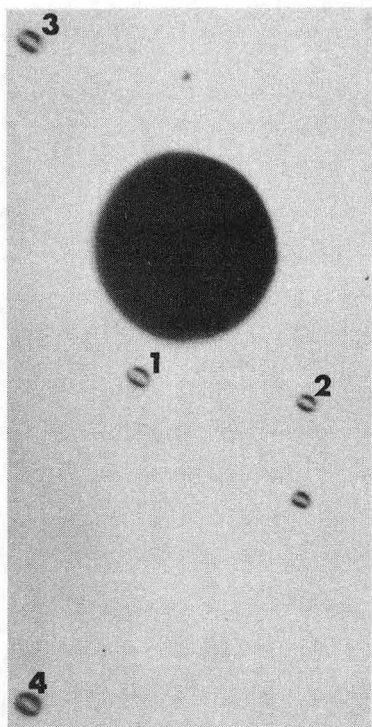
Fig. 3 (cont.)
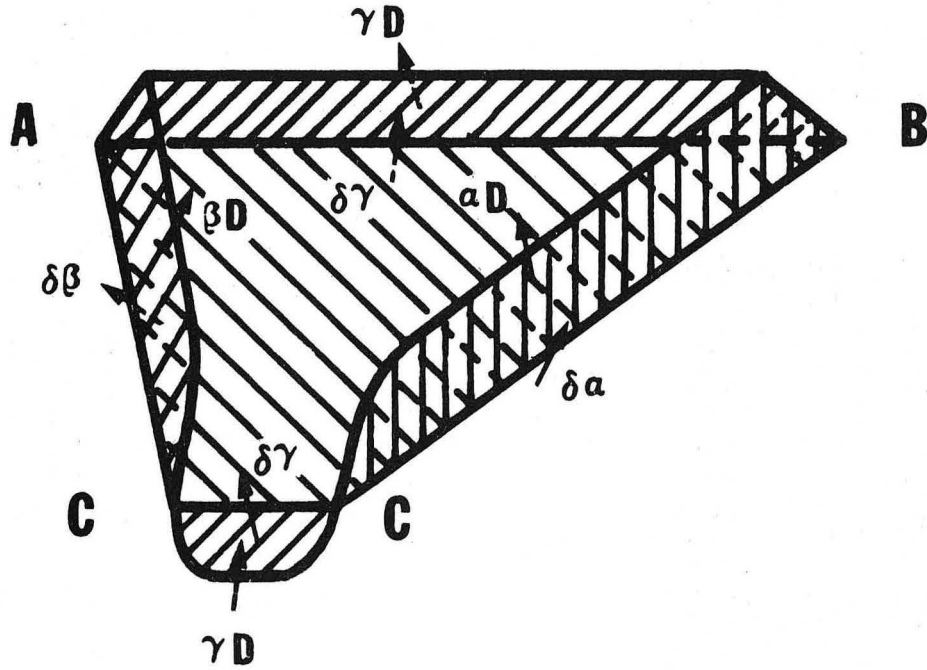
XBL 729-6917

Fig. 4

Fig. 5

XBL 686-1125

Fig. 5 (cont.)

g[200]

XBB 721-3

Fig. 6

XBB 721-118

Fig. 7