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Authors
Miura, Yasuhiro
Washburn, Jack.

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MOBILITY OF PRISMATIC DISLOCATION HALF-LOOPS IN COPPER

Yasuhiro Miura* and Jack Washburn

Inorganic Materials Research Division, Lawrence Berkeley Laboratory and Department of Materials Science and Engineering, College of Engineering; University of California, Berkeley, California 94720

ABSTRACT

Mobility of punched-out prismatic edge dislocation half-loops in copper crystals was studied by a dislocation etch pit technique. Improved crystal growing procedures yielded copper crystals of dislocation density less than 10^3 cm/cm^3. Both dislocation etch pit pictures and an x-ray transmission topograph proved the low dislocation density of the crystals.

Rows of large prismatic dislocation half-loops (radius $\approx 10\mu$) with Burgers vectors $\frac{1}{2}\langle 110\rangle$ were introduced by a ball indentation on a (111) surface in an originally dislocation-free area of the crystal.

The half-loops were approximately semicircular. It was found that larger loops were more mobile than smaller ones. This was attributed to a lower dislocation step density for larger loops. Corners of steps where the dislocation line turns from one glide plane into the other were assumed to be rounded; that is, a short segment of dislocation line at corners probably lies on planes other than {111}. A higher lattice frictional stress acts to oppose motion of a dislocation on non-close packed planes. Therefore, it is suggested that the corners of steps act as pinning points on gliding dislocation half-loops. In agreement with this model it was found that, within a single half-loop, the side which would be expected to have a higher step density was less mobile.

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*Department of Metallurgy, Kyushu University, Fukuoka, Japan.

I. INTRODUCTION

Resistance to conservative motion of dislocations in face centered cubic metals has been studied by many investigators using a wide variety of techniques. Particularly pertinent to the present work are the experiments of Young.[1-8] He found that some dislocations started moving at a resolved shear stress of $4 g/mm^2$, dislocation multiplication took place at $15\text{-}20 g/mm^2$ and macroscopic yielding occurred at the stress of $35 g/mm^2$. The percentage of grown in dislocations moved by the applied stress increased monotonically with the stress until approximately 75% had been moved at the yield stress. Fresh dislocations were observed to move at lower stresses than grown in dislocations. Because the latter could have been pinned by impurity atoms, he concluded that impurity pinning greatly affects the motion of dislocations even in highly pure crystals (nominally 99.999% Cu).

According to Marukawa,[9] dislocation velocity in low dislocation density copper crystals is much larger than in LiF[10] or silicon iron.[11] He found that the distance moved by a dislocation varies little with the loading time. This suggests that dislocations move rapidly but become stuck after moving through a distance which is a function of the stress.

Petroff and Washburn[12] measured the stress at which individual segments of grown in dislocations began to move, obtaining results similar to Youngs. They attributed the range of critical shear stresses for motion to different jog densities existing on the different dislocation lines. This interpretation was supported by the fact that the critical shear stress for motion of small (heavily jogged) prismatic edge dislocation half-loops was found to be greater than $50 g/mm^2$.

The observations above were all carried out at room temperature using the dislocation etch pit technique to record dislocation movement.

The main difficulties with an etch pit technique are: first, the obtaining of specimens with a low enough dislocation density, secondly, the fact that a single etch pit picture can give no information on the internal dislocation arrangement.

Previous etch pit results have been interpreted without paying much attention to the internal configuration of dislocation lines. More precise information on dislocation motion in copper crystals can only be obtained by careful studies on better characterized dislocations.

In the present work, the origin of the frictional stress which opposes glide motion is studied on dislocations of a known shape; prismatic edge dislocation half-loops of various sizes which are introduced by ball indentations into extremely low dislocation density crystals. The critical shear stress for glide of the half-loops can be estimated from their spacing along the glide cylinder using the theoretical equations of Bullough and Newman.[13] Glide mobility can also be studied by applying a stress which causes the half loops to twist on their glide cylinders.

II. EXPERIMENTAL PROCEDURES

A. Materials

Copper rods of 19 mm diameter with a nominal purity of 99.999% Cu were obtained from Materials Research Corporation. Results of spectrographic analysis were as follows: (numbers are in ppm)

Fe	Ni	Si	Sb	Pb	Sn	Bi	Ag	As	Cr	Te	Se*	S*
0.7	1	0.1	1	1	1	0.1	0.3	2	0.5	2	1	1

*Chemical analysis. Other elements not detected.

B. Growth of Low Dislocation Density Crystals

A necessary requirement of the present work was to obtain specimens with an exceptionally low dislocation density, preferably less than $10^3 cm/cm^3$. Large cylindrical crystals, 32 mm diameter, were grown with a ⟨111⟩ growth direction through a zig-zag path from a seed crystal. The zig-zag path was used to avoid direct propagation of the dislocation substructure in the seed into the grown crystal. All melting was carried out under vacuum. The large crystals were placed on a goniometer and oriented to be cut by an acid saw into 5 mm thick discs with faces parallel to a {111} plane. Thinner discs were cut out for transmission x-ray topography. Discs were polished on an acid lathe and finally cut into parallelepipeds on an acid saw. The geometry of the parallelepipeds is shown in Fig. 1. The polishing solution for the acid lathe consisted of two parts HNO_3, one part H_3PO_4 and one part CH_3COOH glacial. Concentrated nitric acid was used for acid saw cutting. Finally the parallelepiped crystals were electropolished at room temperature in a 50% phosphoric acid-40% water solution.

The dislocation density of as-grown crystals was 1 to 5 × 10^3 cm/cm^3 as revealed by etch pit counts on the (111) faces. Livingston's etchant[14] was used.

For further reduction of the dislocation density, thermal cyclic annealing with temperature varying between 800°C and 1050°C for one week was employed. Livingston,[15] Young,[16] and Kitajima et al.[17] have cycled the temperature to increase the rate of decrease of dislocation density.

After the anneal many areas as large as 0.5 cm^2 were virtually dislocation free and the average dislocation density was about 10^3 cm/cm^3.

Borrmann x-ray topographs[5] were also taken for some crystals to check the dislocation structure inside the crystals and to substantiate the dislocation densities measured by etch pit counts.

C. Punching of Prismatic Dislocation Half-Loops

Spherical glass beads approximately 300μ in diameter were dropped on the (111) surface of the copper single crystals through a vertical glass tube. The indentation punches out rows of prismatic dislocation loops probably along all six ⟨110⟩ directions,[18] three of which are parallel to the surface, the other three being directed into the crystal. The rows of punched half-loops along the three ⟨110⟩ directions on the surface are revealed by etching. Each single half-loop is represented by a pair of etch pits. Variation of the size of glass spheres and the height from which they were dropped gave different sizes of loops.

D. Prismatic Dislocation Half-Loops

A typical rosette pattern is shown in Fig. 2. Each of the six branches, extending along the ⟨110⟩ directions, consists of a row of prismatic edge-dislocation half-loops.

E. Geometry of Etch Pits

The edges of the triangular dislocation etch pits which were formed {111} surfaces lay along ⟨110⟩ directions. There are two different combinations of ⟨110⟩ which make equilateral triangles on a (111) plane. Comparison of a microscope picture of an etch pitted surface with an x-ray Laue back-reflection picture of the same specimen viewed from the same direction showed that the etch pit forms in such a way that its pyramidal planes are tilted relative to the surface in the same direction as the three intersecting {111} planes.

III. RESULTS

A. Shape of Half-Loops

The shape of half loops under the surface was determined by alternately etching and then polishing away a known thickness of surface layer (Fig. 3). Typical plots of the distance between a pair of pits vs the depth below the original surface are shown in Fig. 4. Annealing at a high temperature did not result in any appreciable change in observed shape. Loops were always found to be approximately semicircular. However, they were frequently observed to be rotated off from the pure edge orientation.

B. Mobility of Prismatic Dislocation Half-Loops

The critical shear stress acting along the glide cylinder at the position of the last loop of a row was calculated as the sum of shear stresses due to its first, second and third neighbors ($\tau_c = \sum_{i=1}^{3} \tau_i$).

Bullough and Newman's expression,[13] for the shear stress on the glide cylinder due to the presence of a loop was assumed to be a good approximation for half loops. Rows of loops chosen for calculation were those that were of regular and uniform shape and did not have any obvious obstacles ahead on their $\langle 110 \rangle$ directions or any unpaired dislocations along the row. Therefore the only obstacle to further glide of the last loop in a row was assumed to be the frictional resistance to glide.

Critical shear stresses were calculated for a series of loops of different sizes as punched out, and also, after being annealed at 550°C for thirty minutes. The latter should give the critical shear stress required for glide at 550°C. Plots of τ_c as a function of $1/r$ are shown in Figs. 5 and 6. τ_c represents an upper limit for the true critical shear stress, because it was assumed that the specimen crystal was perfect except for prismatic dislocations. From Figs. 5 and 6 it is observed that τ_c increases when the size of the half-loop decreases and τ_c decreases as temperature increases, implying that glide involves thermally activated escape of the dislocation from barriers. Figure 7 shows the effects of applying a bending stress. The twisting motion of some half-loops was observed by etching before and after loading. When the size of a loop is large, interaction between its opposite sides is weak. The two ends intersecting the surface act almost like isolated individual straight dislocation lines. Figure 7 shows that one side of a half-loop is generally more mobile than the other.

IV. DISCUSSION

A. Geometry of Half-Loops

Because the punched-out half-loops were approximately semicircular, they must be jogged. On the atomic scale the edge of the extra half plane of atoms must have numerous steps. The fine structure of a half-loop shown in Fig. 8 will be assumed for the following discussion. The fact that half-loops were approximately semicircular instead of rhombus shape is most likely related to their mechanism of formation. However no prominent change in shape was observed even when loops were annealed at high temperature. This suggests that loop energy would not be significantly reduced if the loop were to adopt a more angular shape. On a fine scale far below the resolution of the etch pit technique, the steps are probably of different sizes depending on loop size. A large loop should be like a photographic enlargement of a small one. This follows from a consideration of the mechanism by which a dislocation half-loop is likely to have been formed during the indentation:

When a hard ball is dropped on the specimen surface, the maximum shear stress arises at some distance below the contact surface. It is reasonable to assume that underneath the contact surface with the glass ball a plastically deformed region will be formed, surrounded by an elastically deformed region. In this situation dislocation half-loops are nucleated and punched-out partially relaxing the local high stresses. The probable mechanism of loop formation is nucleation of a small glide loop somewhere on a (111) plane in the heavily stressed region with its Burgers vector $(a/2)\langle 110\rangle$. The screw component of the loop, instead of continuing to move on its original plane and in so doing leaving the region of high stress would tend to sweep around the half-spherical stress contour by a number of successive cross slippings, forming a pair of prismatic loops; an interstitial half-loop and a vacancy half-loop. The interstitial loop would glide away along the ⟨110⟩ direction parallel to its Burgers vector and the vacancy loop would be annihilated within the plastically deformed region under the ball indenter. The shape of loops formed in this way should depend on the shape of the stress contours and on the stress gradient; larger loops should tend to contain longer steps resulting from less frequent cross slip.

B. Minimum Energy Orientation of Half-Loops

It is expected that, for a prismatic dislocation loop, the orientation for minimum energy on its glide cylinder will not be that of the shortest total dislocation length which is the pure edge orientation, but will be an orientation that is tilted due to the interactions between opposite segments. Bullough and Forman[19] have considered the orientation dependence of the elastic strain energy of a rhombus shaped dislocation loop quantitatively. Energy variations accompanying rotation about ⟨110⟩ and ⟨001⟩ axes were computed. Their results show that a shallow minimum in the energy exists away from the pure edge orientation for a large range of loop sizes. The sizes of loops studied in the present experiments are such that $10^3 < a/r_o < 10^5$ (a = diameter of loop, r_o = radius of dislocation core). Bullough and Newman's results show that the energy of a loop in this size range does not change much when it is rotated within ± 20° from the pure edge orientation about either a ⟨110⟩ or an ⟨001⟩ axis. This explains the observed scatter in the orientation of half-loops. Only frictional stress must be overcome to rotate a half-loop to any orientation within about 20° away from the plane perpendicular to its Burgess vector.

Therefore the tilt of a particular set of loops probably depends on the stress distribution that existed when the loops were being punched out and on the relative mobilities of the two sides of the loop. There are always some irregularities of the surface of the crystal and of the glass ball used for indentation. Therefore the stress field should seldom be exactly symmetrical.

Grown in defects, both point defects and dislocations may also serve as obstacles to the gliding half-loops and can cause rotation away from the pure edge orientation.

C. Glide of Half-Loops

The critical shear stress (τ_c) for loop motion increases with the reciprocal of the loop radius and decreases at high temperature (Figs. 5 and 6). These observations suggest that the density of pinning points along the dislocation is a function of the radius of the loop.

Possible dislocation pinning mechanisms are:

1. Pinning by impurity atoms or vacancy clusters
2. Long range interactions with grown in dislocations
3. Surface pinning due to surface roughness
4. Pinning at steps on the dislocation line.

Pinning by foreign atoms has been reported to be important even for high purity copper (99.999% Cu). Young[1] explained the observed critical shear stress of 4 g/mm^2 for grown in dislocations in 99.999% Cu in terms of a Cottrell atmosphere. He observed that fresh dislocations moved at a critical shear stress even lower than $4g/mm^2$. However, there are other possible differences between grown in and freshly multiplied lengths of dislocation. One possibility is a difference in jog density.

It is difficult to see how impurity pinning could explain the size effect observed in the present experiments; the short range interaction between a solute atom and a dislocation should not be sensitive to its macroscopic radius of curvature. Pinning by vacancy clusters is subject to the same objection. The X-ray transmission topographs of annealed crystals had dark spots which, according to Young,[6] are vacancy clusters. The density of these spots was about comparable with the density of dislocation etch pits. Therefore they were not numerous enough to have been the primary barriers to dislocation glide.

The long range elastic stress due to grown in dislocations was also negligible in the highly perfect crystals used for the present study (the density was less than $10^3 cm/cm^3$ and over areas larger than the indentation rosettes the crystals were often dislocation free).

Any contribution from internal stress to the critical shear stress for glide of a half-loop should also be independent of loop size.

Pinning of the end of the dislocation at the crystal surface could make a size dependent contribution to the critical shear stress because on an atomic scale even a carefully electropolished metal surface has some surface roughness and a layer of oxide.

When a dislocation intersecting a crystal surface glides, new surface is created along the path of the dislocation wherever the Burgers vector has any component at right angles to the surface. Also some change in the dislocation length and deformation of the oxide must occur. The applied stress must supply extra energy for these processes. However, for the large loops considered in these experiments surface pinning could not be the rate controlling mechanism.

In section A the shape of the half-loops was described as macroscopically semicircular but with jogs or steps which were assumed to exist where the dislocation changed from one glide plane to another (fig.8). For such a dislocation the core energy at jogs or at the ends of super-jogs might be expected to be higher than that in straight segments on {111} planes. Figure 9 shows the two different kinds of step corners, an acute corner LMN, and an obtuse corner PQR, corresponding respectively to those on the more heavily stepped side and on the less stepped side of a dislocation half-loop.

In neither case is it likely that the corners would be atomically sharp. It would require the difference in energy between a dislocation segment lying on {111} and a segment on a non-close-packed plane to be unreasonably large. Therefore, it is expected that the dislocation will locally shorten its length by slightly rounding corners. The segment of dislocation LN will lie approximately on a {100} plane while PR will be on {110}. Assume that a length equal to five Burgers vectors lies on a plane other than {111} at each corner.

If the step lengths are shorter for smaller half-loops, the fraction of total dislocation length which lies on planes other than {111} increases as loop diameter decreases. Then for smaller loops a greater fraction of the dislocation line is forced to glide on {110} or {100} rather than on {111}. Glide on non-close-packed planes is associated with a higher Peierls-Nabarro stress. If this Peierls-Nabarro stress at jog corners provides the primary frictional resistance to glide then the critical stress for motion would be expected to be temperature dependent as was observed.

The critical shear stress for glide of a half-loop with an infinitely large radius (1/r extrapolated to zero in Fig. 6) is only a few grams per square millimeter, which is in good agreement with the reported value by Young and Petroff and Washburn for the minimum shear stress to move the most highly mobile grown in dislocation segments in copper.

D. Twist of Half-Loops on the Glide Cylinder

According to the half-loop model in Fig. 8 dislocation segment, α(ABCD---) is expected to have more jog steps than dislocation segment, β(A'B'C'D'---). If the crystal is stressed in bending with the axis along <112> the segment α and the segment β are expected to move in opposite directions along the glide cylinder. If jog steps act as the primary pinning points, the dislocation-β should be more easily moved because it has fewer steps. This behavior was observed in the great majority of loops and so constitutes independent evidence that step density is an important factor in determining mobility of an edge dislocation.

CONCLUSIONS

1. The critical stress necessary to cause motion of a loop along its glide cylinder decreases with increasing loop size.

2. Because all the loops that were studied were found to be macroscopically semicircular in shape they must be jogged or stepped with the longest segments probably lying on {111} because of dissociation into shockley partials.

3. The effect of size on loop mobility can be explained if it is assumed that the primary resistance to glide occurs at the corners of steps where the dislocation turns from one {111} glide plane into another. Considering the probable conditions of half loop formation the number of such step corners per unit length should decrease with increasing loop size.

4. Application of a stress which tends to twist a loop on its glide cylinder showed that one side is more mobile than the other. This observation can also be explained if it is assumed that step corners provide the primary resistance to glide. A geometrical argument shows that steps must be more numerous on the same segments that were observed to be less mobile.

5. Qualitatively the results are consistent with the assumption that at jog corners where a dislocation bends from one {111} glide plane into another it is slightly rounded so that at these points the dislocation is undissociated for a length of several interatomic distances and is forced to glide on non-close-packed planes.

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FIGURE CAPTIONS

Fig. 1. Size and geometry of specimen.

Fig. 2. Punched-out rosette of prismatic dislocation half-loops.

Fig. 3. Shapes of half-loops revealed by alternately etching and then removing known thicknesses by electropolishing.

Fig. 4. Plot of results from Fig. 3 for two loops: (a) assuming one side of the loop lies exactly on {111}, (b) assuming loop is symmetrical.

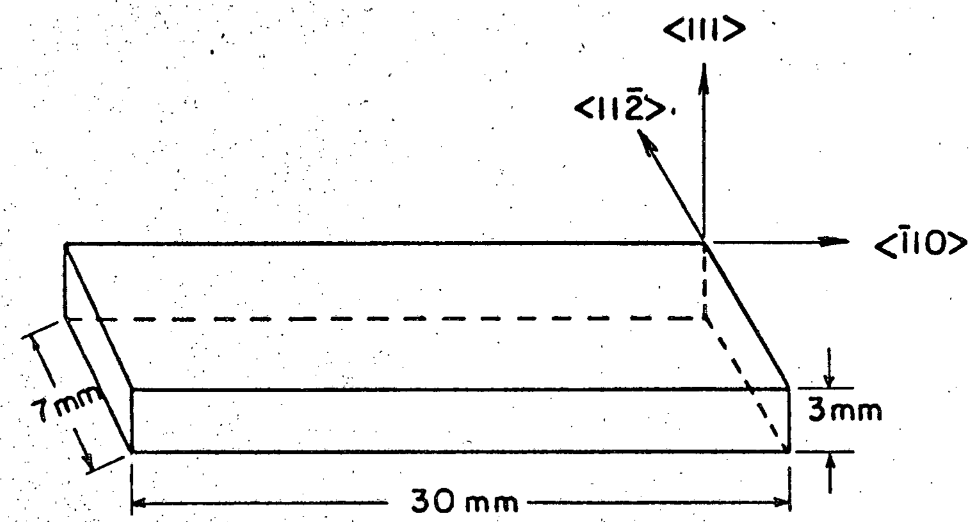
Fig. 5. Critical shear stress for glide (τ_o) as a function of reciprocal of the loop radius (1/r) at room temperature. The point represented by a square box is from Petroff and Washburn.[12]

Fig. 6. Critical shear stress for glide (τ_c) as a function of reciprocal of the loop radius (1/r) for larger loops at room temperature and at 550°C.

Fig. 7. Behavior of half-loops under a twisting shear stress. At 15 g/mm^2 applied stress only the less jogged sides of some of the half-loops have moved, (a,b,c). In d at τ = 20 g/mm^2 both sides of loops have moved.

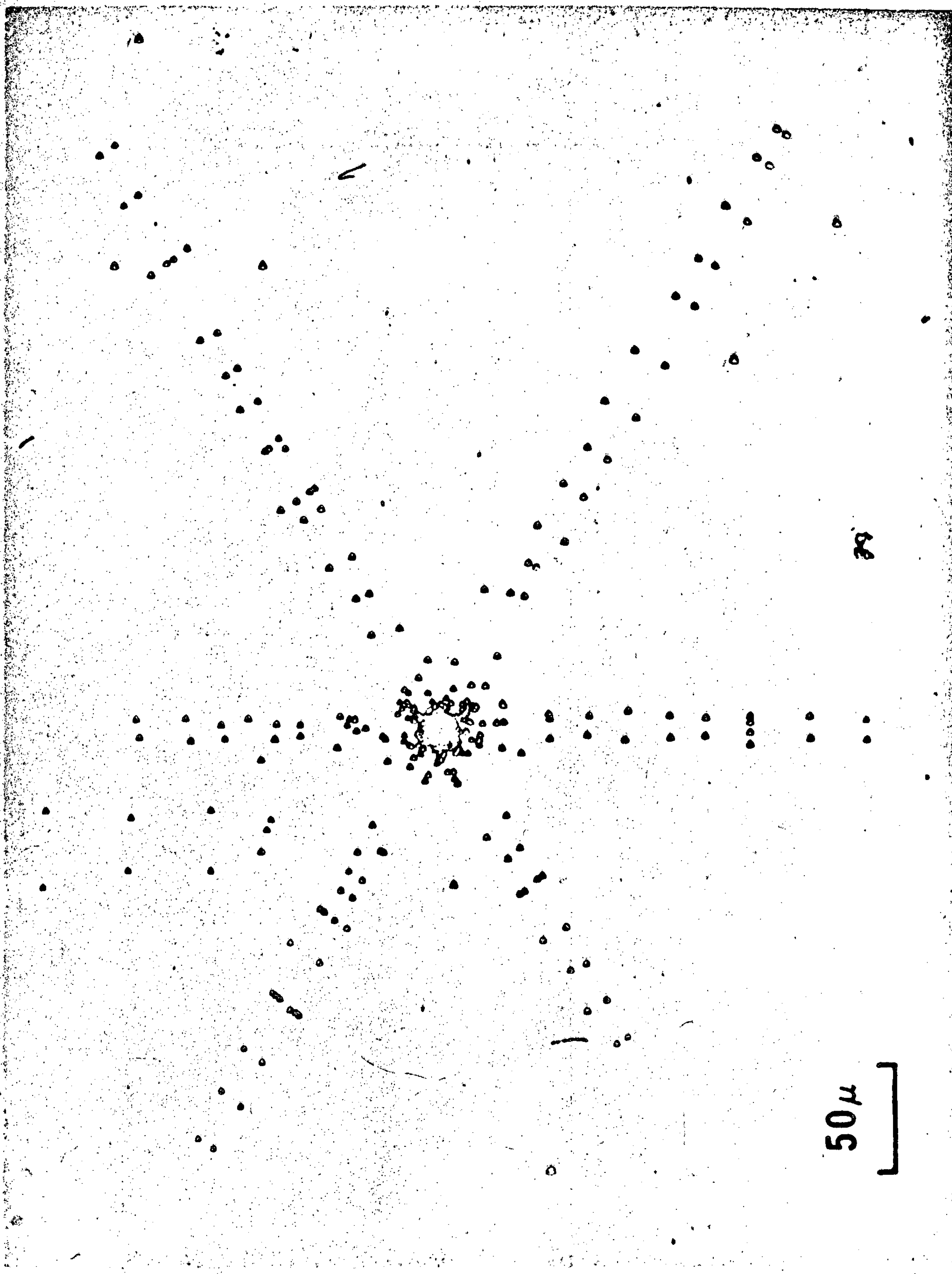
Fig. 8. Assumed fine structure of half loops.

Fig. 9. Proposed atomic scale rounding of obtuse and acute step corners.



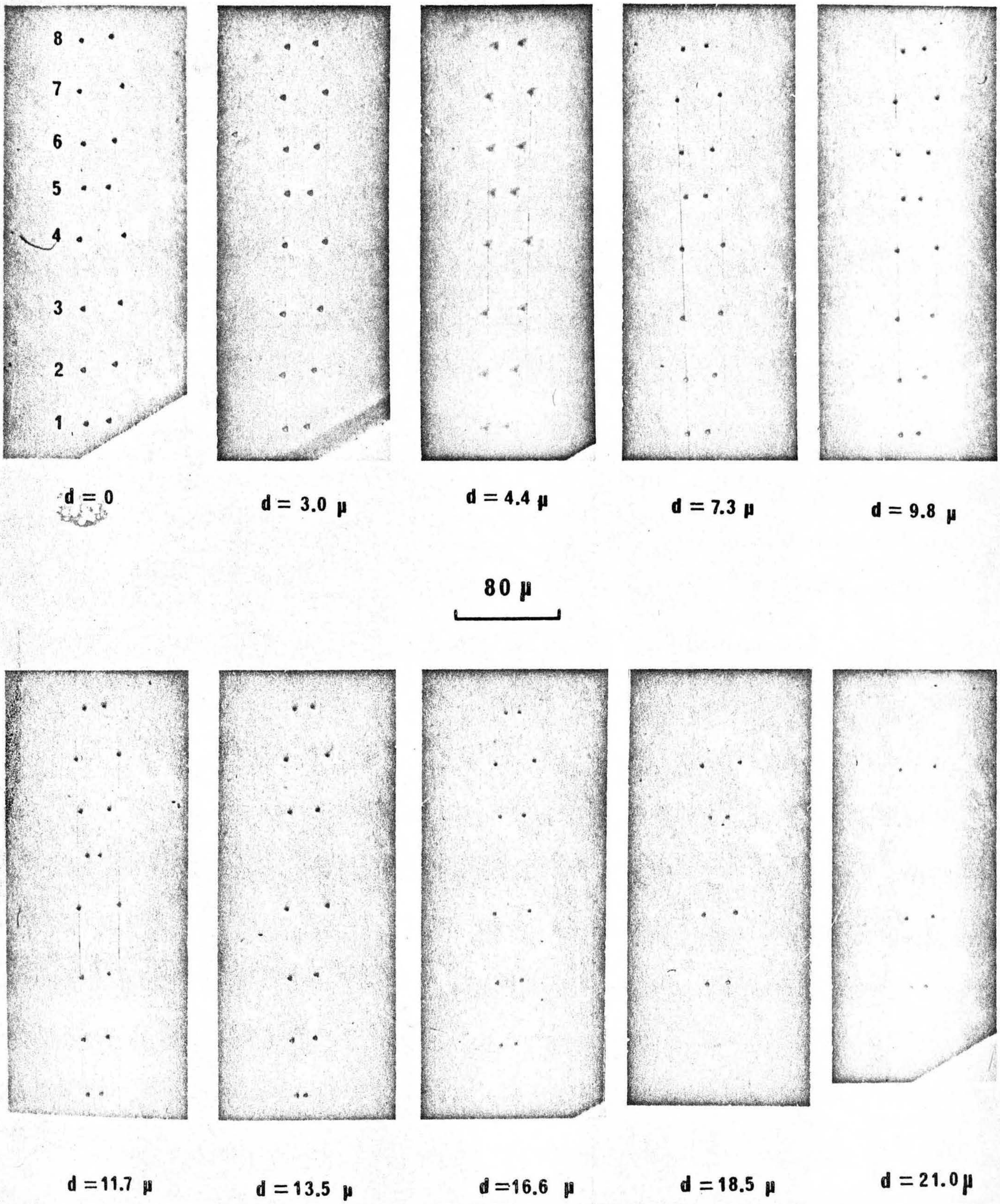
XBL 705-966

Fig. 1



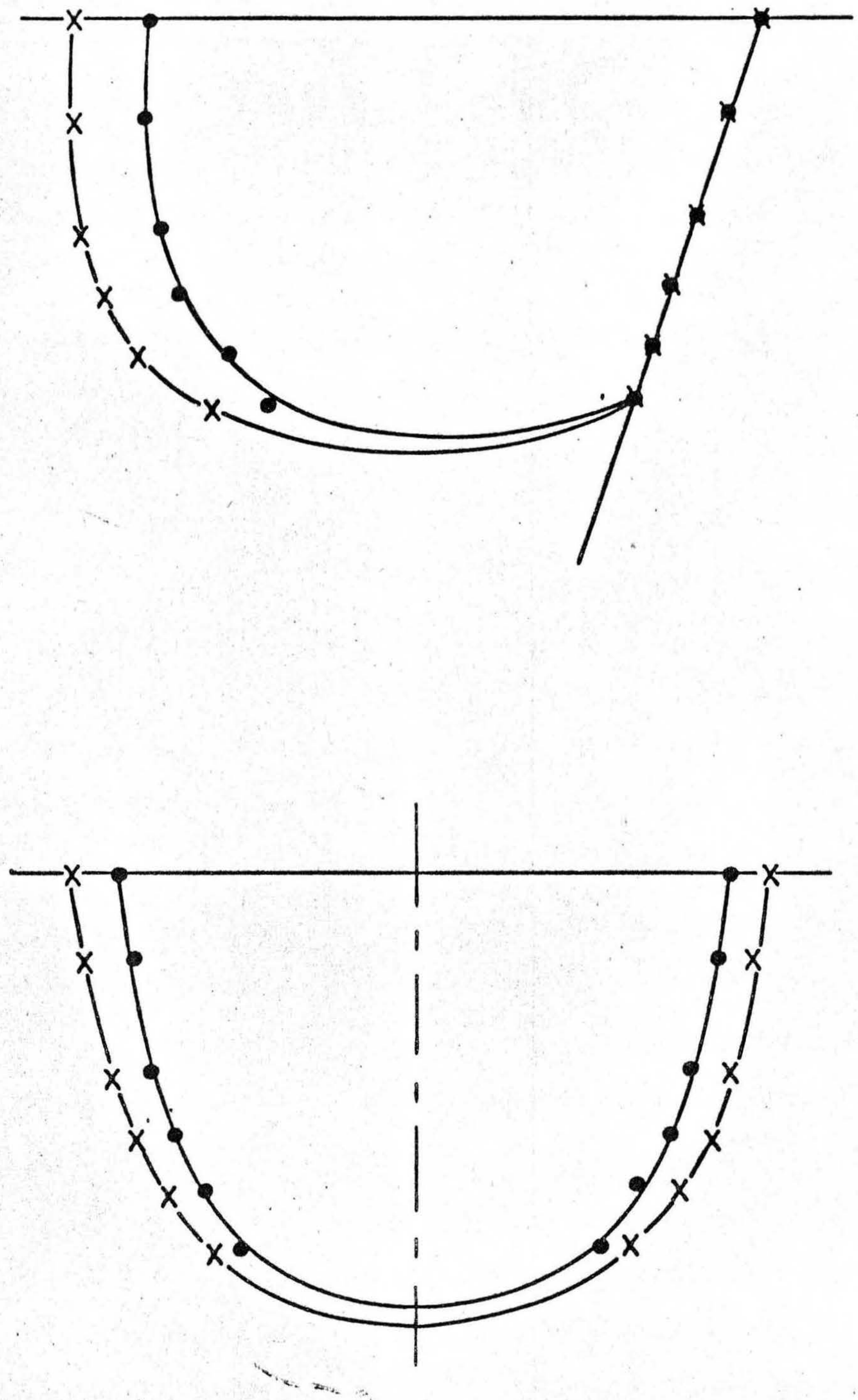
XBB 704-1999

Fig. 2



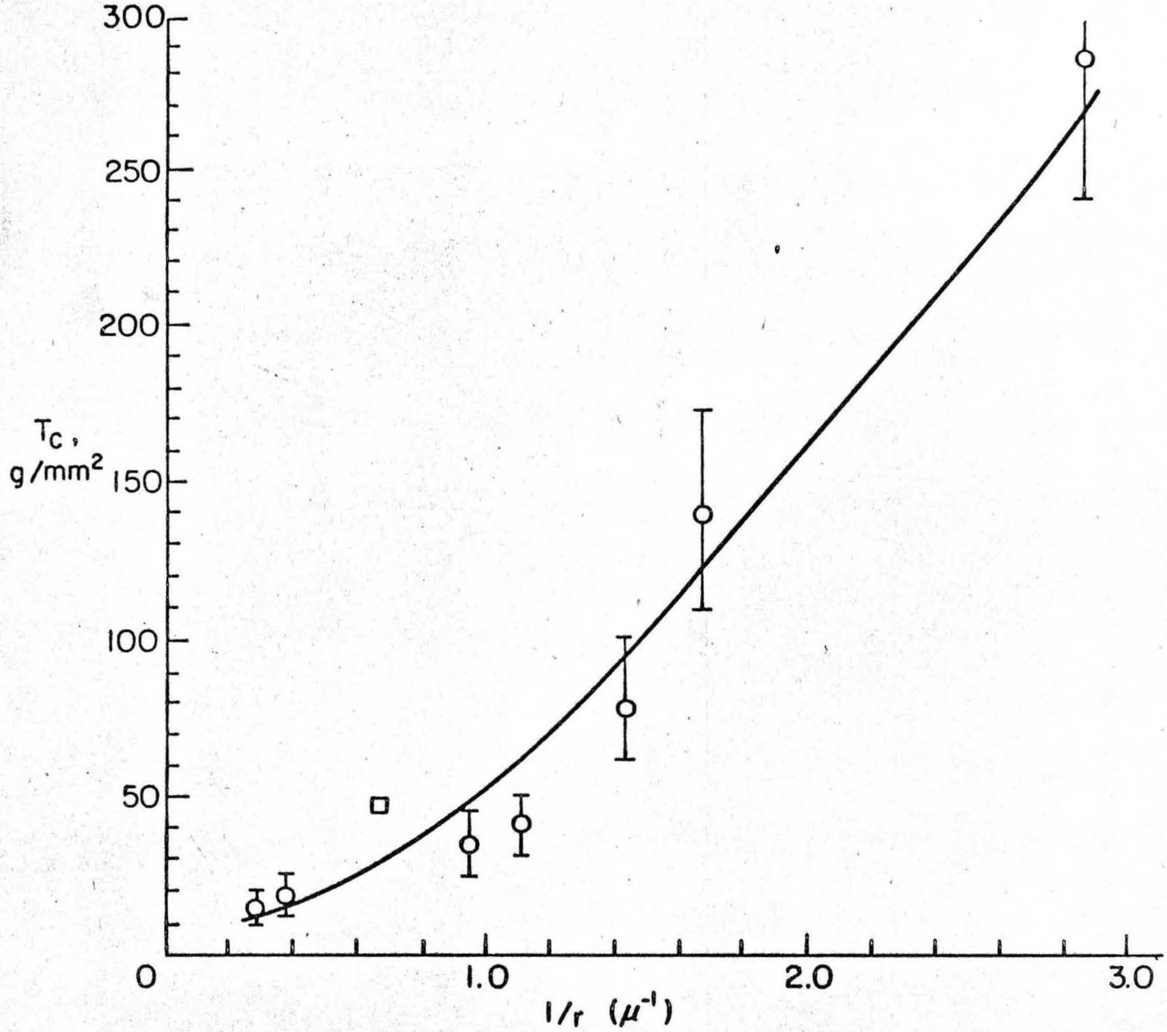
XBB 704-2001

Fig. 3



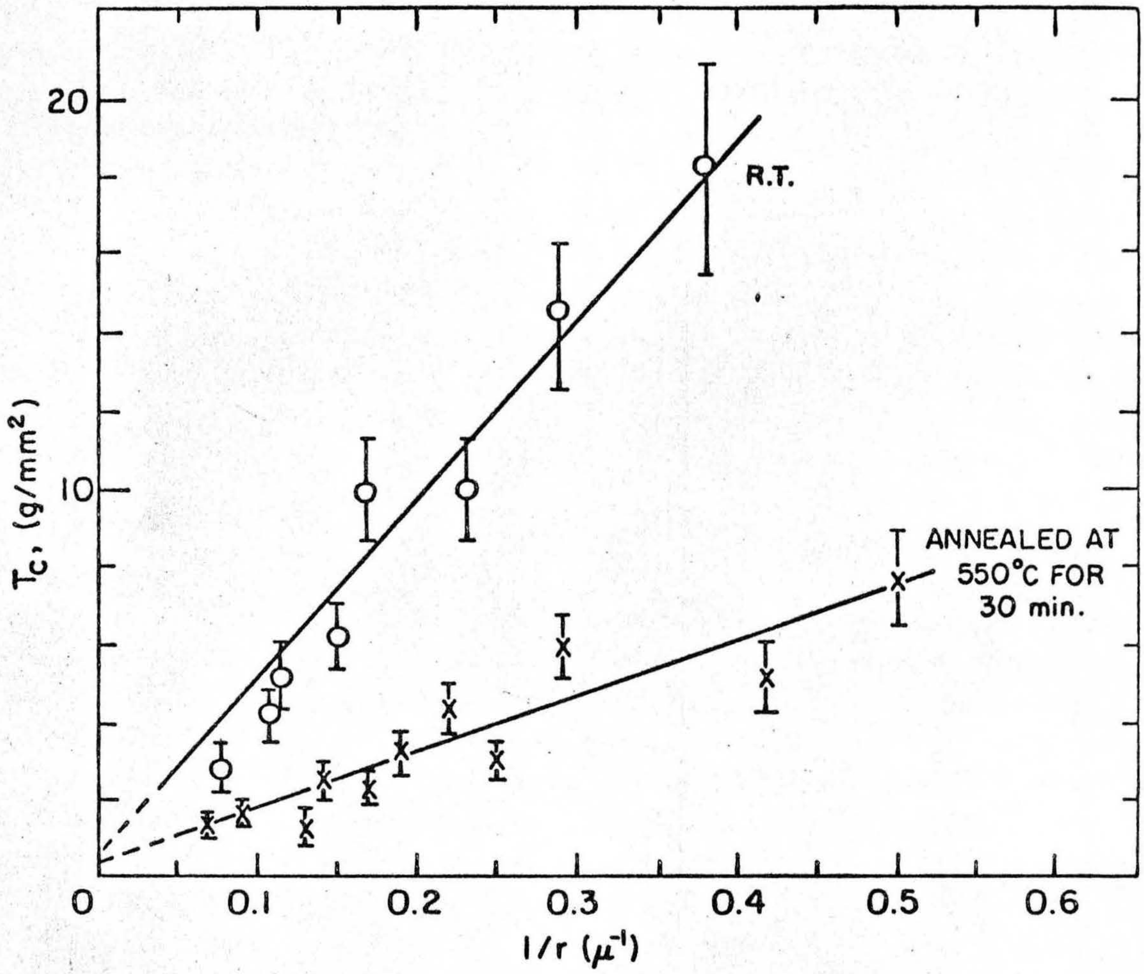
XBL 705-964

Fig. 4



XBL 705-955

Fig. 5



By the method of least squares

R.T. $\tau_c = 49.1\ (1/r) - 0.2$

550°C $\tau_c = 14.1\ (1/r) + 0.4$

XBL 708-1676

Fig. 6

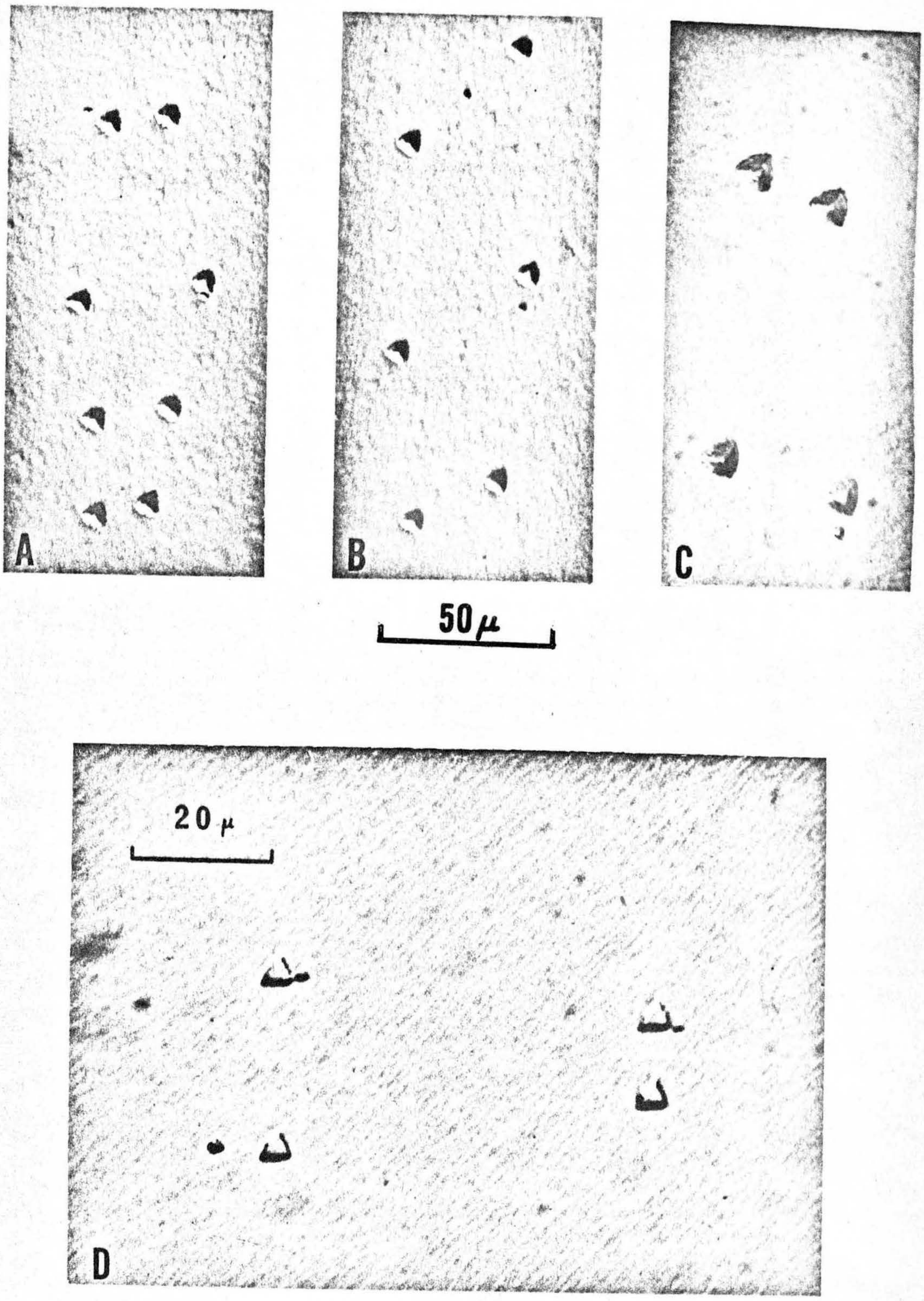


Fig. 7

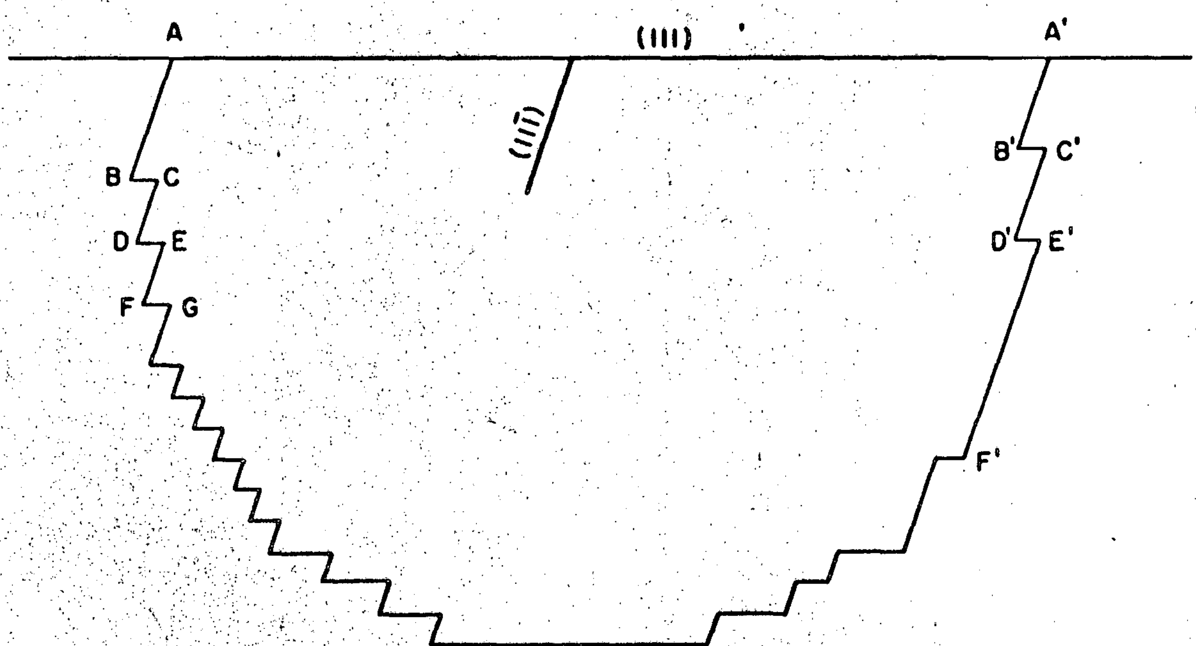


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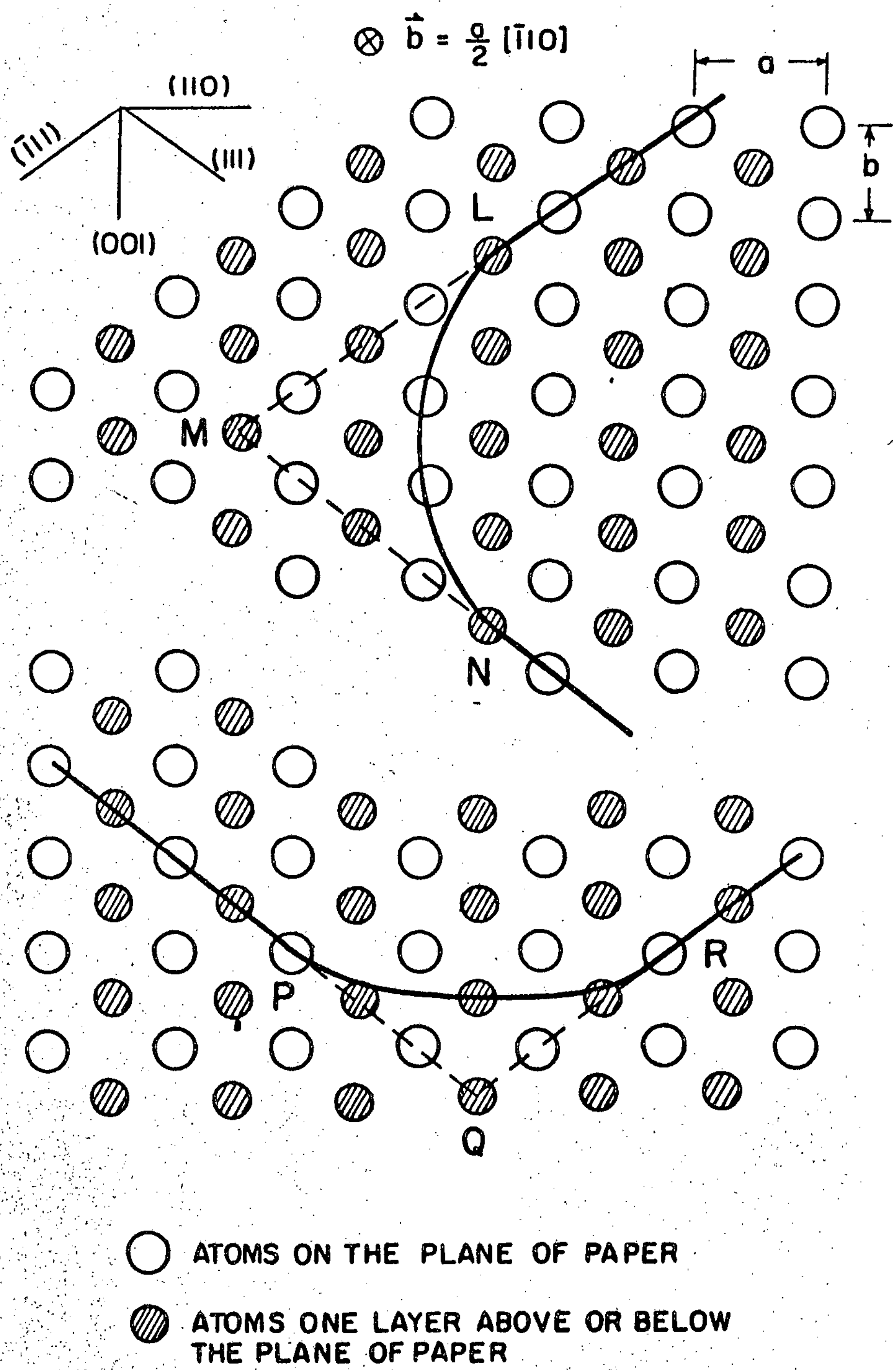
Fig. 8



XBL 707-1367

Fig. 9

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