Submitted to Materials Science
and Engineering

LBL-1406
Preprint c.2

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January 1973

Prepared for the U. S. Atomic Energy Commission
under Contract W-7405-ENG-48

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TRANSMISSION ELECTRON MICROSCOPIC STUDIES OF PRISMATIC SLIP IN MAGNESIUM OXIDE

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SUMMARY

The movement of dislocation loops in separate pairs was studied at relatively lower temperatures where bulk diffusion is insignificant. This was done by repeatedly photographing the same area of a sample under identical diffraction conditions in the microscope after annealing the sample outside the microscope. The dislocation loops move both by glide and climb due to interaction. A systematic study of glide or prismatic slip of dislocation loops is reported.

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I. INTRODUCTION

Dislocation loops are formed by irradiation[1,2] and quenching[3,4] due to condensation of vacancies or interstitials. Dislocation loops are also formed by moving dislocations containing sessile jogs[5,6] and by a "punching effect" in crystals containing small precipitates.[7] Since long-range elastic field of a dislocation is not significant,[8] two loops can interact with each other only if they are close enough. The movement of the loops as a result of interaction can occur by self climb and/or glide. Self climb or conservative climg, which occurs at lower temperatures compared to usual climb involving bulk diffusion, is determined by core or pipe diffusion. Pipe diffusion parameters determined by the measurements of self climb rates of coplanar loops has already been reported by the author.[9] Movement by glide necessitates overcoming the Peierls force and may also be further hindered by interactions with impurity atoms and other point defects. The work of Bullough and Newman[10] on the equilibrium spacing of a row of prismatic dislocation loops generated by a center of pressure (prismatic punching) suggests that the ease of movement of a loop by glide can give a direct measure of the Peierls force. In ionic crystals, where Peierls force is much higher than metals, the glide of dislocation loops due to mutual interaction is possible only at high temperatures. In MgO this temperature is above one thousand degrees centigrade. Since so far it is not possible to do in-situ hot stage experiments above 1000°C in the electron microscope, a technique of annealing electron microscope sample outside the microscope without contamination was developed. The same area of a sample could be

photographed under identical diffraction conditions after different annealing treatments. Using this technique glide or prismatic slip of dislocation loops due to interaction with other loops in separate pairs or because of interaction with free surfaces, was studied.

II. EXPERIMENTAL PROCEDURE

Thin slices of thickness (∿ 1 mm) of MgO single crystals were cleaved carefully along {100} planes. Crystals contained the following impurities: Al - 0.06%, Fe - 0.03%, Ca - 0.03%, Mn - 0.002%, Cr - 0.002%, Cu < 0.001%, Si < 0.001%. The surface damage introduced during cleaving was removed by chemical polishing in H_3PO_4 at 150-160°C. These crystals were plastically deformed (bending) and subsequently annealed to create dislocation loops. After initial thinning of these crystals, a jet polishing technique[5] was used to obtain electron microscope samples. Using the stereo technique, the position of various loops in thin foil was determined. Surface dirt particles served as convenient reference points. After selecting the areas of interest in the sample, they were photographed under the same diffraction conditions after various annealing treatments done outside the microscope. A Siemens 100 KeV microscope was used. The details of sample preparation and contamination-free annealing techniques are described elsewhere.[11]

III. RESULTS

Figure 1 shows coalescence of dislocation loops 6 and 7 primarily by glide or prismatic slip, and coalescence of dislocation loops at 5 occurring mainly by self climb. The annealing treatments in Fig. 1 A through E were: A → B, 34 min at 1200°C; B → C, 39 min at 1237°C; C → D, 65 min at 1200°C; D → E, 45 min at 1200°C. The changes in the sizes of isolated dislocation loops is negligible showing that bulk diffusion effects in this temperature range in MgO are very small. Both dislocation loops 6 and 7 had the b-vector 1/2[101] and were determined to be vacancy type. The separation between glide axes of the two loops was roughly equal to the sum of the radii of the two loops. It is noteworthy that the interaction and consequently the glide rate is enhanced as the loops come closer to each other (see Figs. 1C and 1D). This is expected from Foreman and Eshelby's[12] formula as discussed below. Figure 2 at 1 shows another example where two vacancy type small loops, b = 1/2[101], on parallel glide cylinders are slipping due to mutual interaction. Their separation of glide axes is roughly equal to the sum of their radii. Following are the annealing treatments in Fig. 2: A → B, 20 min at 1250°C; B → C, 19 min at 1250°C; C → D, 49 min at 1100°C. In Fig. 3, dislocation loop 1 is moving along its glide cylinder due to interaction with the big loop 2. Both the loop and the dipole have b = 1/2[101] and probably of vacancy type. The axis of the glide cylinder of the smaller loop is approximately one radius of the loop away from the nearest side of the loop. As discussed in Section IV, this case can be approximated by the loop moving due to influence of single edge dislocations. This approximation is

easier to handle in rate equations. Following are the annealing treatments in Fig. 3: A → B, 45 min at 1100°C; B → C, 45 min at 1100°C; C → D, 21 min at 1200°C; and D → E, 34 min at 1200°C. At 3 in Fig. 3 there is another small loop gliding under the influence of the big loop.

Figure 4 shows an example where probably an interstitial loop having common glide axis with bigger vacancy loop, b = 1/2[101], slips and annihilates. The remaining loop near the point of annihilation (Fig. 4B) is like a dipole and it breaks up into loops by pipe diffusion[13] on further annealing (Fig. 4C). The annealing treatments of Fig. 4 are: A → B, 27 min at 1050°C; B → C, 34 min at 1050°C.

In Fig. 5, the dislocation loop at 1, b = 1/2[101], was very close to the surface and also intersecting the surface at one end. Figure 5B shows how the part of the dislocation loop closer to the surface has slipped out because of surface forces. At 3 there is another loop intersecting the surface which is slipping out. At 2, the part of the dipole which is near to the surface has slipped out. Following are the annealing treatments from Fig. 5A through C: A → B, 10 min at 1086°C; B → C, 19 min at 1086°C. In Fig. 6, the half loops, b = 1/2[101], at 2, 3, 4 and 5 (part of the dipole) have slipped out of the foil as annealing is continued. Annealing treatments were as follows: A → B, 19 min at 1086°C; B → C, 40 min at 1086°C, C → D, 41 min at 1086°C.

IV. DISCUSSION

Following Foreman and Eshelby,[12] for the case of two infinitesimal or widely spaced loops the total force on the loop for glide motion is

$$\frac{\partial E_{int}}{\partial Z} = \frac{\mu b_1 b_2 A_1 A_2}{4\pi(1-\nu)} \frac{3\cos\theta}{R^4} (3 - 30\cos^2\theta + 35\cos^4\theta) \tag{1}$$

where b_1, b_2 and A_1, A_2 are the Burgers vectors and areas of the two dislcoation loops respectively, θ is the angle that R makes with the loop normal. R is the separation of centers of two loops.

We can write

$$\sin\theta = \frac{S_p}{R} , \qquad \cos\theta = \frac{Z}{R} , \tag{2}$$

where S_p is the separation between axes of glide cylinders of the two loops.

Using relations of Eq. (2), one can write Eq. (1) as

$$\frac{\partial E_{int}}{\partial Z} = \frac{\mu b_1 b_2 A_1 A_2}{4\pi(1-\nu)} \frac{3Z}{(S_p^2 + Z^2)^{5/2}} \left(3 - \frac{30Z^2}{S_p^2 + Z^2} + \frac{35Z^4}{(S_p^2 + Z^2)^2}\right) . \tag{3}$$

Assuming glide motion as non-thermally activated process we can find the lower limit of Peierls stress as follows: Let ΔE_{int} be the change in interaction energy when the loop moves from Z_1 to Z_2

$$\Delta E_{int} = \frac{3\mu b_1 b_2 A_1 A_2}{4\pi(1-\nu)} \left[-2(S_p^2+Z^2)^{-3/2} + 8S_p^2(S_p^2+Z^2)^{-5/2} - 5S_p^2(S_p^2+Z^2)^{-7/2}\right]_{Z=Z_1}^{Z=Z_2} . \tag{4}$$

Also

$$\Delta E_{int} = 2\pi r\, \tau_p\, b(Z_2 - Z_1) \tag{5}$$

where τ_p is Peierls stress, r is the radius of the gliding loop and b is its Burgers vector.

As mentioned earlier Foreman and Eshelby's analysis is good only for infinitesimal or widely spaced loops. For closely spaced and finite sized loops Foreman and Eshelby's analysis becomes a poor approximation. For closely spaced and finite sized loops, Foreman's[14] following analysis is used.

Let the center of one loop be at the origin of an orthogonal coordinate system (x,y,z) with z axis perpendicular to the plane of the loop and the center of other loops at $(x_1 y_1 z_1)$. If the vectors $d\ell_1$ on one loop and $d\ell_2$ on the other loop represent two small segments of their perimeters with coordinates $(x_1 y_1 z_1)$ and $(x_2 y_2 z_2)$ respectively, the interaction energy dE_{int} between them according to Foreman is:

$$dE_{int} = \frac{\mu b^2}{4\pi(1 - \nu)} \left\{\frac{1}{R} + \frac{(z_1 - z)^2}{R^3}\right\} d\ell_1 \cdot d\ell_2 \qquad (6)$$

where

$$R^2 = (x_1 - x_2)^2 + (y_1 - y_2)^2 + (z_1 - z_2)^2 \quad .$$

The total loop-loop interaction is obtained by taking the double line integral of Eq. (5) around both loops. This integral may not be evaluated anayltically. For calculations of ΔE_{int}, Foreman's results of numerical integration have been used. For the pair of dislocation loops at 1 in Fig. 2, the measured displacement along the glide cylinder is 80 Å. Using the diameter of the loop 300 Å, from Foreman and Eshelby's analysis, τ_p is equal to 1.1×10^{10} dynes/cm^2 and Foreman's analysis gives τ_p as 7.0×10^8 dynes/cm^2. Probably the latter value is more reliable as the loops are not widely spaced or infinitesimal. Foreman and Eshelby's analysis is good for loops spaced more than the diameter of the bigger loop in a pair. In fact two analyses give almost identical results at or after such spacing.

In the case where a small loop is gliding because of interaction with a very big loop having parallel glide axis and the separation of axes of the two loops is equal to the sum of their radii (see Fig. 7). The energy of interaction can be approximated by the interaction energy of a loop with a straight dislocation. Following Kroupa and Price,[15] interaction energy can be written as:

$$E_{int} = \frac{\mu b b_1}{1-\nu} RZ \left\{1-\left\{(2Y^2+Z^2-1)+2\sqrt{[(1+Y^2+Z^2)^2-4Y^2]}\right\}^{-1/2}\right.$$

$$\left.\cdot[(Y^2+Z^2-Y)((Y-1)^2+Z^2)^{-1/2}+(Y^2+Z^2+Y)((Y+1)^2+Z^2)^{-1/2}]\right\} \quad , \quad (7)$$

where b and b_1 are Burgers vectors of loop and dislocations respectively and $Y = y_1/r$ and $Z = z_1/r$ (Fig. 7).

We have studied cases where Y = 1, so Eq. (7) looks like

$$E_{int} = \frac{\mu b b_1}{1-\nu} \left\{1-[2Z^2+2Z\sqrt{(Z^2+4)}]^{-1/2} \cdot [Z+(2+Z^2)(4+Z^2)^{-1/2}]\right\} \quad . \quad (8)$$

Neglecting the thermal activation, the Peierls stress τ_p is

$$\Delta E_{int} = \int_{Z_1}^{Z_2} dE_{int} = 2\pi r \tau_p \, b_o (Z_2-Z_1) \quad , \quad (9)$$

where Δz is the displacement of the moving loop along the glide cylinder and ΔE_{int} is the change in interaction energy. Applying this for loops 1 and 2 in Fig. 3, τ_p is 3.3×10^8 dynes/cm^2. Diameter of the loop is 4 1/2 Å and displacement along the glide cylinder of the loop 1 was 180 Å. These values of τ_p are in good agreement with the available values from creep data.[16]

For more accurate values of Peierls stress, the problem should be treated as thermally activated process. Using double-kink model the

velocity of a dislocation loop[17] along ⟨110⟩ can be written as

$$\nu = \frac{dZ}{dt} = \frac{\nu_D b^2 L}{2W^2} \exp - \left[\frac{4b^3 G^{1/2} \tau_p^{1/2}}{\pi^{3/2} kT} \left(1 - \frac{\tau}{\tau_p}\right)^2 \right] \tag{10}$$

where ν_D = Debye frequency, L = average spacing of the kinks, W = width of the kink, G = shear modulus, τ = stress on the loop, T = temperature and k = Boltzmann's constant.

Experimentally Z as a function of t is known at a given temperature. Substituting for τ from Foreman and Esbelby or from Foreman's analysis in Eq. (10), we can find τ_p. Currently efforts are being made for more accurate values of Z vs. t and to integrate numerically Eq. (10) to obtain more reliable values of τ_p.

ACKNOWLEDGMENT

The author is grateful to Prof. J. Washburn and the late Prof. J. E. Dorn for stimulating discussions.

This work was done under the auspices of U. S. Atomic Energy Commission through IMRD of Lawrence Berkeley Laboratory.

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FIGURE CAPTIONS

Fig. 1 Prismatic slip of dislocation loops 6 and 7, and loop at 5. Notice the coalescence in Figs. 1-D and 1-C for loops 6 and 7, and loops at 5 respectively.

Fig. 2 Prismatic slip or glide of loops at 1. They coalesce to form a single loop (see Fig. 2-D).

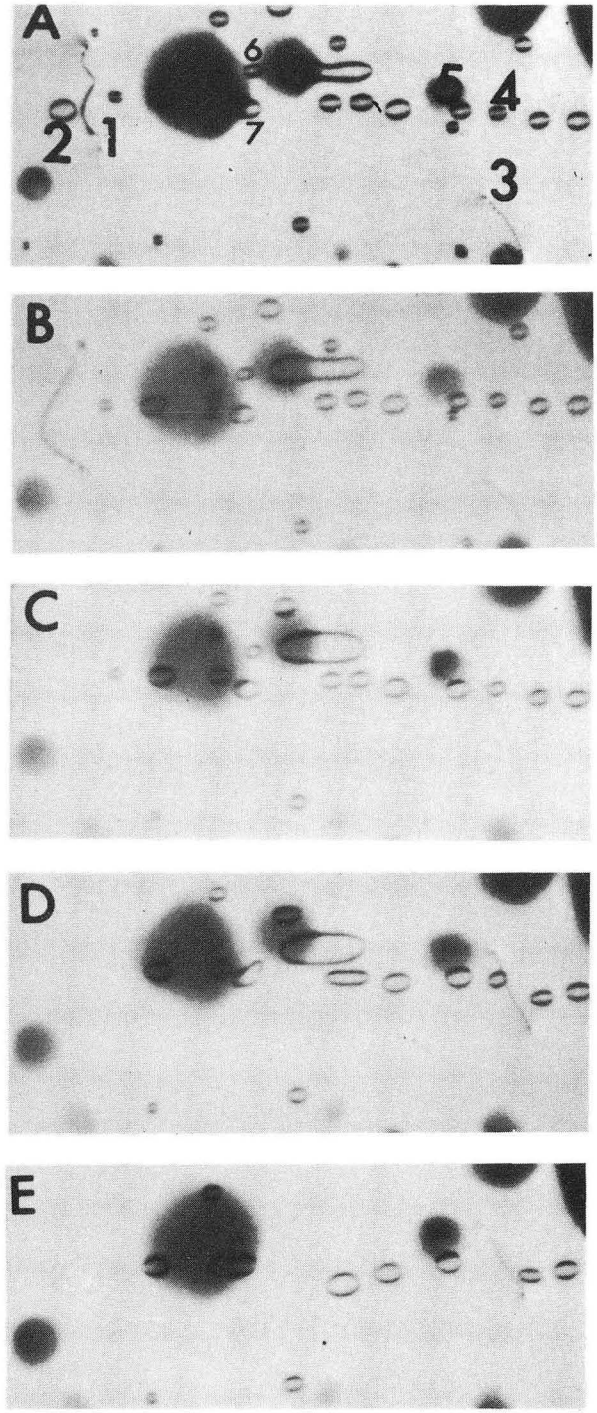
Fig. 3 Prismatic slip of loops 1 and 3, due to interaction with very big loops.

Fig. 4 Glide of an interstitial loop because of interaction with a vacancy loop.

Fig. 5 Dislocation loops at 1 and 3 intersecting the surface are slipping out of the foil due to interaction with the surface. Part of a dipole at 2 has also slipped out because of surface forces.

Fig. 6 Slipping out of dislocation loops at 2, 3, 4 and 5 which intersect the surface.

Fig. 7 Interaction of a circular edge dislocation loop with an infinite straight edge dislocation (shown schematically).



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0.5 μ

Fig. 1.

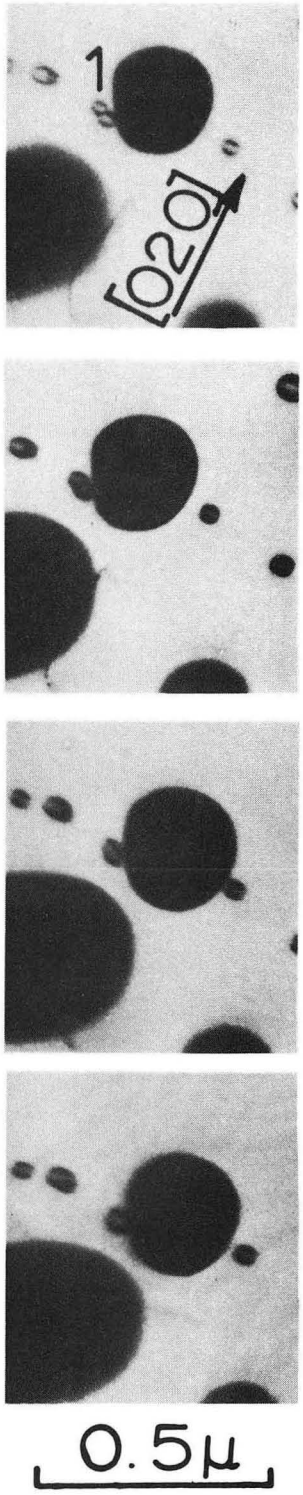

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Fig. 2.

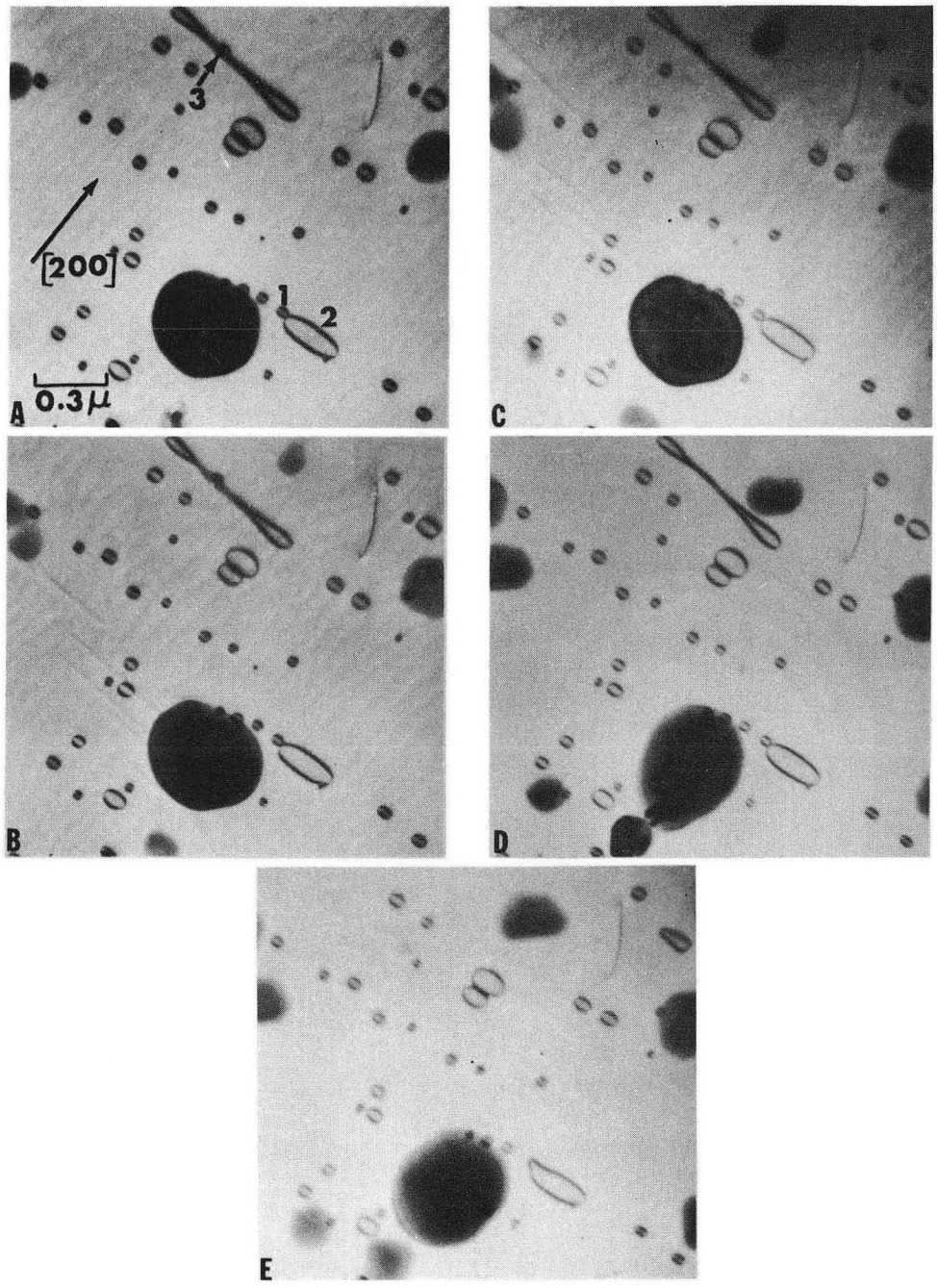

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Fig. 3.

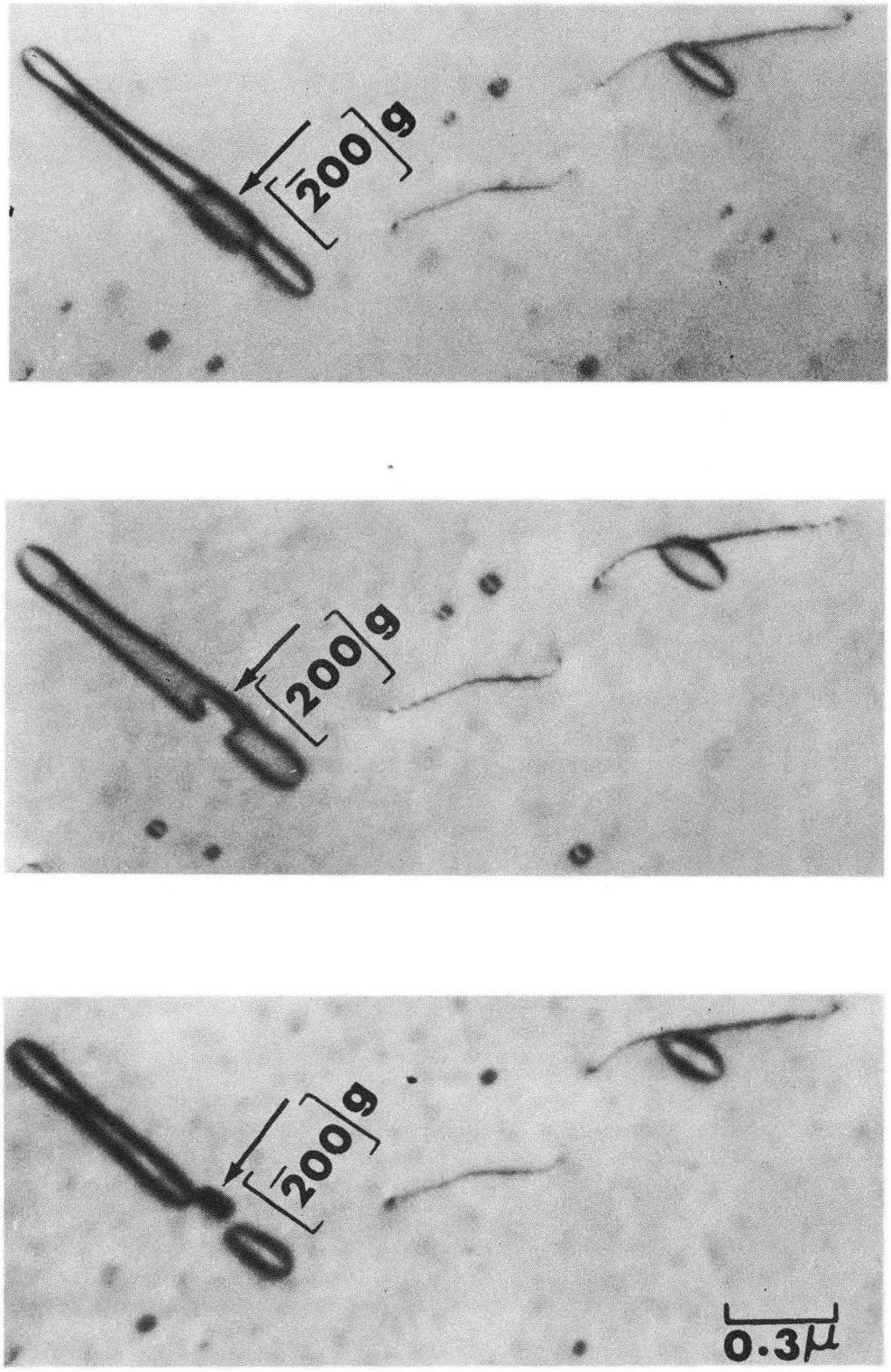

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Fig. 4.

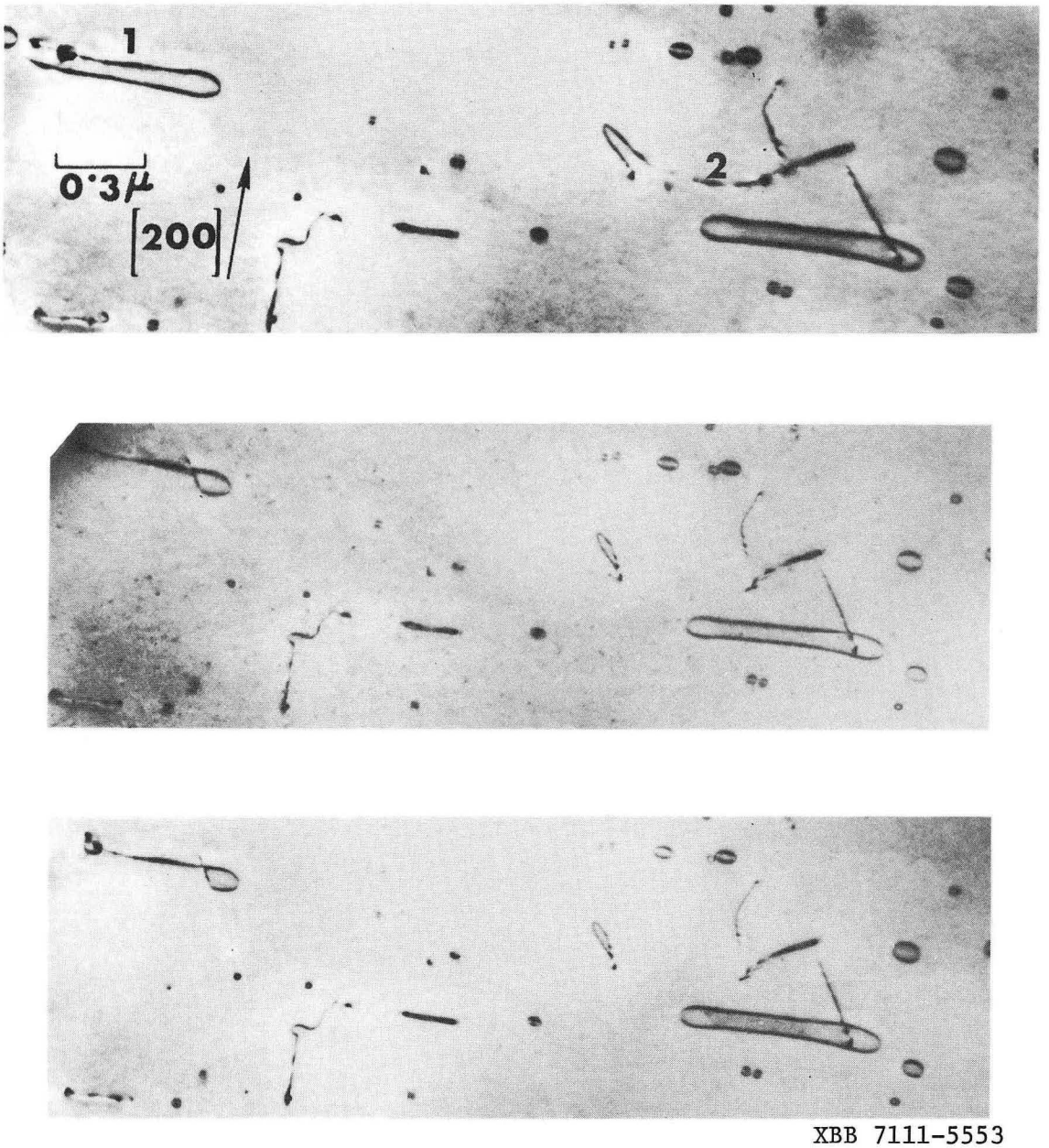

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Fig. 5.

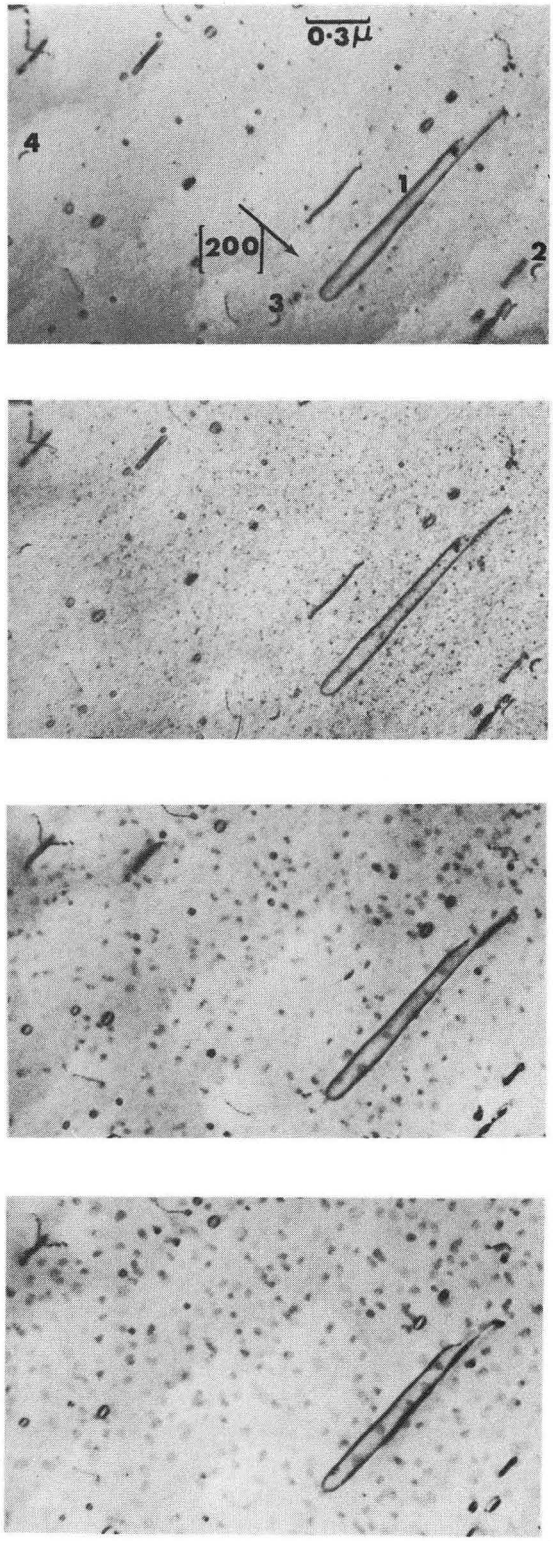

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Fig. 6.

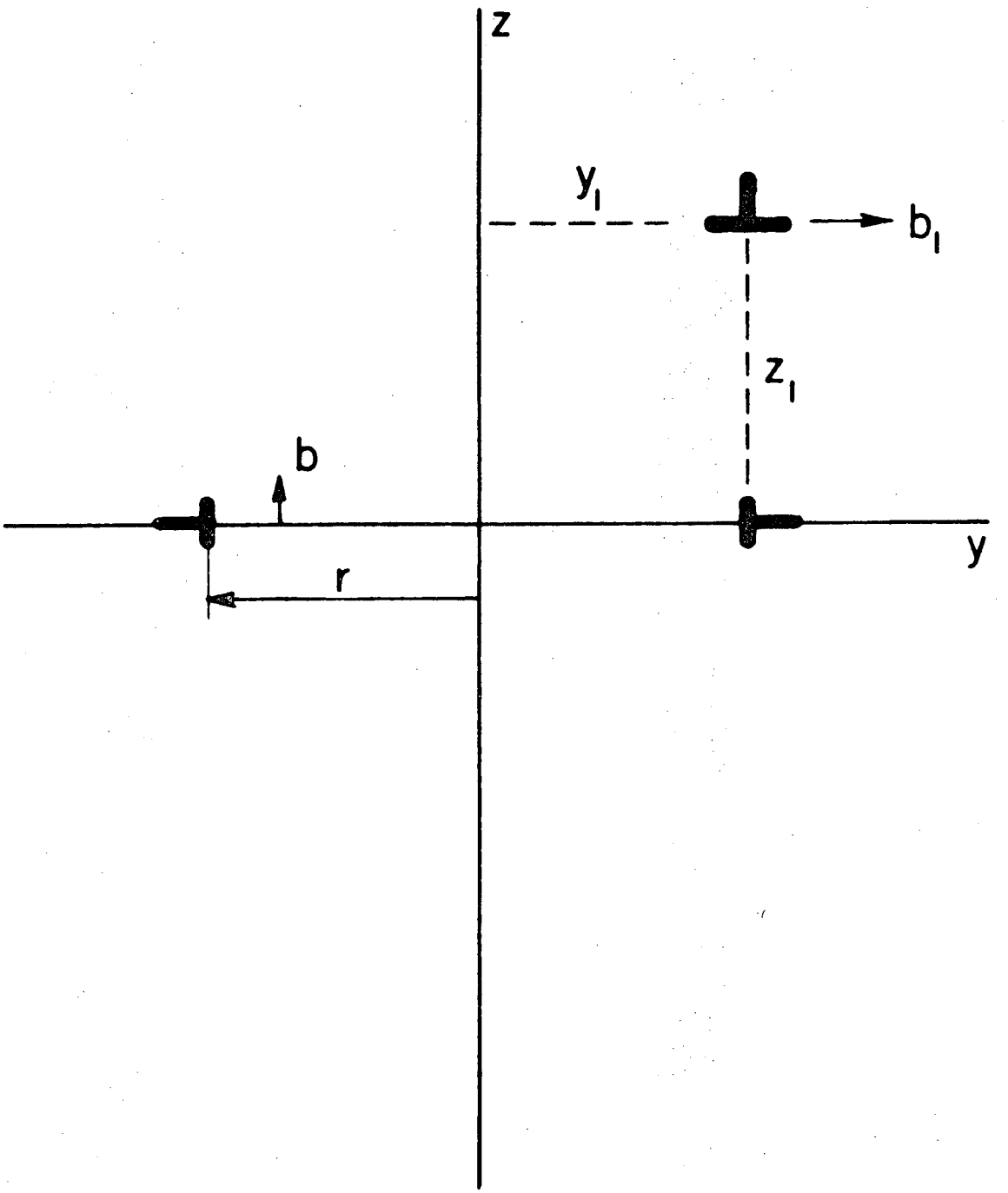

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Fig. 7.

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