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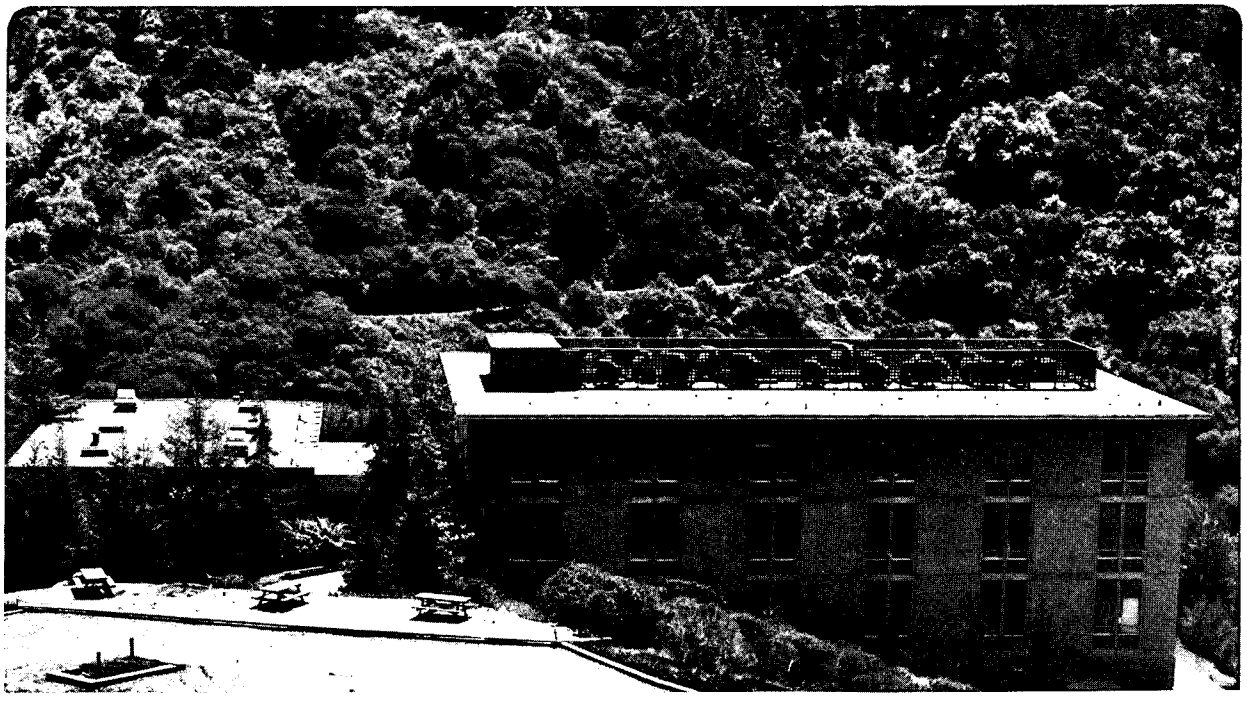

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THE MECHANISM OF θ' PRECIPITATION ON CLIMBING DISLOCATIONS IN Al-Cu

U. Dahmen and K. H. Westmacott

Materials and Molecular Research Division

Lawrence Berkeley Laboratory

University of California

Berkeley, California 94720

The numerous ways in which crystal imperfections can facilitate precipitation of a second phase from supersaturated solid solution are well documented. In appropriate alloys and under appropriate conditions, grain boundaries, stacking faults, dislocations, and vacancies all act as heterogeneous nucleation sites for precipitation. Two different aspects of this phenomenon may be distinguished: static and dynamic. In the static case, the disturbance in the lattice periodicity produced by the defect, particularly a grain boundary or dislocation, can partially accommodate disparities in the size or structure of the precipitating phase, thereby reducing the strain energy factor in the nucleation equation.

The dynamic case occurs when the product phase forms on a moving grain boundary or dislocation and particularly interesting precipitate configurations can arise under these conditions. In a sense, these events represent combinations of lattice defect involvement since the point defects that induce the boundary or dislocation mobility (climb) are also indirectly involved in the precipitation process. Diffusion-induced-grain boundary-migration (DIGM) or the alternative description as chemically-induced grain boundary-migration (CIGM) is a subject of considerable current interest (see e.g. 1,2,3), but the phenomenon is still not fully understood.

Many examples of repeated precipitation on climbing dislocations have been reported since the original observations of NbC formation on climbing Frank partials in stainless steel (4). Striking precipitate arrays were observed in Cu-Ag (5), Si-Cu (6) and Al-Cu (7,8) but again the precise formation mechanisms have not been identified.

The configurations of θ' in Al-4w/o Cu (see Fig. 1) first described by Guyot and Wintenberger (7) and studied in detail by Headley and Hren (8,9) can be produced by a direct quench from the solution treatment temperature to a high

final aging temperature ($\gtrsim$ 160°C) circumventing the precursory GP zone and θ" formation. While the overall nature of the precipitate arrays is now understood, the basic processes giving rise to the various, often complex, precipitate structures is not. Guyot and Wintenberger (7) proposed that repeated nucleation of θ' occurred on {100} plane segments of the climbing edge dislocation, and described the slip and climb processes required to produce the observed configurations. They believed, as did Nes from studies on Si-Cu (6,10), that climb of the dislocation was at least partially driven by a point defect flux generated by the growing precipitate. Headley and Hren (8) showed that, at least in Al-Cu, the climbing dislocation loops originated mainly from Bardeen-Herring sources operating on {101} planes by the quenched-in vacancy supersaturation. The long laths of θ' observed lying along <010> directions (examples are seen in Figs. 1-4) were also attributed to nucleation of {100} plane dislocation segments produced by slip and climb, and Headley and Hren developed a model to explain their spacing. They proposed that rotation of the source climb plane off {101} would result in many dislocation kinks which could coalesce into large 'super-kinks' lying in the required 100 planes with a characteristic equilibrium separation. Although not explicitly emphasized, Guyot and Wintenberger described the process occurring in the [$\bar{1}$01] direction of the (101) plane while Headley and Hren considered the [010] direction. In common with earlier workers the mechanism of θ' nucleation was thought to be simply the lowering of the free energy barrier by the dislocation stress field. The present contribution reports a TEM contrast analysis of the dislocation bounding the precipitate colony which reveals that the main role of the dislocation in the precipitation process is a structural one.

Fig. 2 shows the leading edge of a (101) precipitate colony which is growing in the [010] direction. Attention is focussed on this part of the colony because for

geometric reasons the associated (100) and (001) θ' laths are not formed in the orthogonal $[\bar{1}01]$ direction (see Fig. 1b). In Fig. 2c the colony has been tilted edge-on and the dislocation segments on (100) and (001) brought about by the slip and climb process are evident. Furthermore, it is particularly apparent from the weak beam dark field image of Fig. 2b that the contrast exhibited by the (100) dislocation segments is distinctly different from the other parts. The fainter contrast seen for example at A is consistent with that expected from a unit dislocation that has dissociated into two partials according to a reaction of the type $\frac{1}{2}[101] \longrightarrow \frac{1}{2}[100] + \frac{1}{2}[001]$. Evidence for this dissociation may be seen at every point along the line where the {100} segments join the rest of the dislocation. One of the partial dislocations has kept up with the climbing dislocation while the other has trailed behind in a wide dipole bounding the latest in the series of θ' laths to form. The $\frac{1}{2}$<100> dislocation components resulting from the dissociation have pure shear and pure edge character. The question arises as to whether the leading edge of the forming θ' lath is bounded by the shear or edge component. In principle, either case is possible but in fact contrast analysis of many different dissociated segments showed only the latter; the leading partial was always the edge. A simple proof of this is seen in Fig. 3 where the dissociation of the unit $\frac{1}{2}[101]$ dislocation bounding the (001) lath at A is clearly evident. With g = 200 (Fig. 3b) the trailing segment is in, and the leading segment out of, contrast. In 3a where g = $0\bar{2}0$, g.b = 0 for both segments but g.b x u residual contrast is observed for the leading segment only, consistent with Burgers vectors of $\frac{1}{2}[001]$ (edge) and $\frac{1}{2}[100]$ (shear) for the leading and trailing dislocations respectively.

As seen in Fig. 4 the contrast associated with the precipitate laths changes with increasing distance from the front. This (101) plane colony is inclined to the electron beam allowing its bounding dislocation to be seen in contrast. One set of

precipitate laths lies on edge (A) and the other (B,C) lies at 45° to the beam. It is clear that though the laths at B and C are the same variant, the trailing dislocation segments exhibit different behavior. An explanation for the differences based on contrast analyses is as follows.

The configuration at B corresponds to the formation of a single unit cell of θ' on the $\frac{1}{2}[100]$ dipole trailed behind the climbing dislocation. It thus exhibits g.b = 1 behavior in a 200 reflection. At C there is a region immediately behind the leading (edge) dislocation where the precipitate exhibits no contrast. This is consistent with the formation of a second unit cell of θ' by a shear process (11) that shifts the lattice in the opposite sense to the original dislocation dipole. The resulting double unit cell of θ' distorts the matrix by only a small amount (0.052nm) in the direction normal to the plate and it is thus virtually invisible by strain contrast. Further away from the growth front at x contrast (residual for this g vector) reappears and analysis shows this to be consistent with the formation of a further unit cell of θ' via the alternative vacancy condensation mechanism (11). For example, in the g = ± 111 weak beam pair shown in Fig. 3c,d, the contrast bounding the laths trailed behind the front is typical of g.b = ± 2/3 with s ≫ 0, i.e. weak outside contrast (A in 3c, B in 3d) and strong inside contrast (A in 3d, B in 3c). Since the displacement of the matrix in the c direction produced by 3 unit cells of θ', one of which formed by vacancy collapse, is 0.052nm + 0.228nm = 0.69a, this is precisely the expected contrast. The continued variation in the precipitate contrast toward the center of the colonies is consistent with further thickening by the shear and/or the collapse (vacancy) mechanisms. Additional support for this interpretation is found in the A laths imaged edge-on in Fig. 4, Moving away from the leading dislocation the strain contrast sequence is medium, weak, strong, in

good agreement with that expected from a 1 unit cell (shear), 2 unit cell (shear + antishear), 3 unit cell (shear + antishear + vacancy loop) growth sequence.

These results provide proof for the structural role of dislocations in the precipitation event as opposed to a purely dilatational one, in excellent agreement with a detailed model presented elsewhere. In essence, it was proposed (11) that two criteria govern the θ' growth thickness sequence; minimization of shape change and minimization of volume change. Shape changes are eliminated by forming growth steps from units with pairs of $\frac{1}{2}$<100> shears of opposite sign. Similarly, the volume change is minimized with a growth sequence which assembles different units with partially cancelling interstitial- and vacancy-type strains normal to the habit plane. One consequence is that the formation of single unit cells of θ' is normally inhibited because it would require a single shear. This prediction has been confirmed experimentally (11,12).

For the present purpose it is sufficient to note that a $\frac{1}{2}$<110> dislocation lying on {100} dissociated in the manner observed can readily initiate θ' formation. Since the shear segment is trailed behind, the area enclosed by the dipole is sheared relative to its surroundings and formation of a single unit cell of θ' can occur without introducing an additional shear. Thus, the role of the dislocation is not to provide precipitate misfit accommodation in the conventional sense (i.e. dilatation) but rather to facilitate formation of a region with the structural atomic arrangement close to that of the precipitate. The stacking fault bounded by the $\frac{1}{2}$<100> partial dislocation changes the AB stacking of {100} Al planes to the AA stacking required in the θ' structure. The formation of the resulting single unit cell of θ' can then occur with a lower activation energy than the double unit cell found in matrix nucleation of θ' (12).

In summary, the observed configurations associated with the heterogeneous nucleation of θ' on climbing dislocations are consistent with a structural role of matrix dislocations in the precipitation event, i.e. they facilitate plate formation with minimal volume and shape change.

The wide and complex range of contrast effects observed within the precipitate colonies indicates that both the vacancy and solute atom supersaturations are eliminated entirely by heterogeneous processes. The vacancies initially by condensation on Bardeen-Herring climb sources, and later on the heterogeneously nucleated θ' plates; and the solute atoms by θ' nucleation on the dissociated climb source dislocations.

Acknowledgement

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References

1. M. Hillert, Scripta Met. 17, 237 (1983).
2. J. W. Cahn, J. D. Pan, and R. W. Balluffi, Scripta Met. 13, 503 (1979).
3. R. W. Balluffi and J. W. Cahn, Acta Met. 29, 493 (1981).
4. J. M. Silcock and W. J. Tunstall, Phil. Mag. 10, 361 (1964).
5. H. M. Miekk-Oja and R. Räty, Phil. Mag. 24, 1197 (1971).
6. E. Nes and J. Washburn, J. Appl. Phys. 42, 3559 (1971).
7. P. Guyot and M. Wintenberger, J. Mat. Sci. 9, 614 (1974).
8. T. J. Headley and J. J. Hren, Phil. Mag. 34, 101 (1976).
9. T. J. Headley and J. J. Hren, J. Mat. Sci. 11, 1867 (1976).
10. E. Nes, Acta Met. 22, 81 (1974).
11. U. Dahmen and K. H. Westmacott, to be published.
12. W. M. Stobbs and G. R. Purdy, Acta Met. 26, 1069 (1978).

Fig. 1. Typical colonies of θ' precipitates heterogeneously nucleated on climbing dislocation loops in Al-4Cu directly quenched from 550°C to 220°C, and aged 5 min at this temperature and quenched in water. Colonies lying on (101) plane contain only the (100) and (001) variants of θ'. The precipitates are drawn out into long laths along the [010] growth direction of a colony, and form discrete plates in the [$\bar{1}$01] growth direction (micrographs by M. Wall).

Fig. 2. Leading edge of (101) colony growing in [010] direction with θ' laths trailing behind the dislocation. The contrast changes where precipitates contact the dislocation such as at A. The edge-on view (c) shows that at the precipitates the dislocation line has kinked onto the (100) and (001) planes.

Fig. 3. Contrast analysis of (001) precipitate at A in a (101) colony showing g.b x u contrast only at the leading edge (a) and the g.b = 1 dislocation contrast only at the trailing partial (b). Figs. 3c and d show a weak-beam dark-field g/3g = $\pm$ $\bar{1}$11 pair of the entire precipitate colony. The laths (A) exhibit weak outside contrast in (c) and strong inside contrast in (d) characteristic of g.b = $\pm$ 2/3, 4/3 and have vacancy-type strain fields.

Fig. 4. (101) colony in [011] projection showing the progressive change in contrast of precipitates corresponding to increasing thickness behind the growth front.

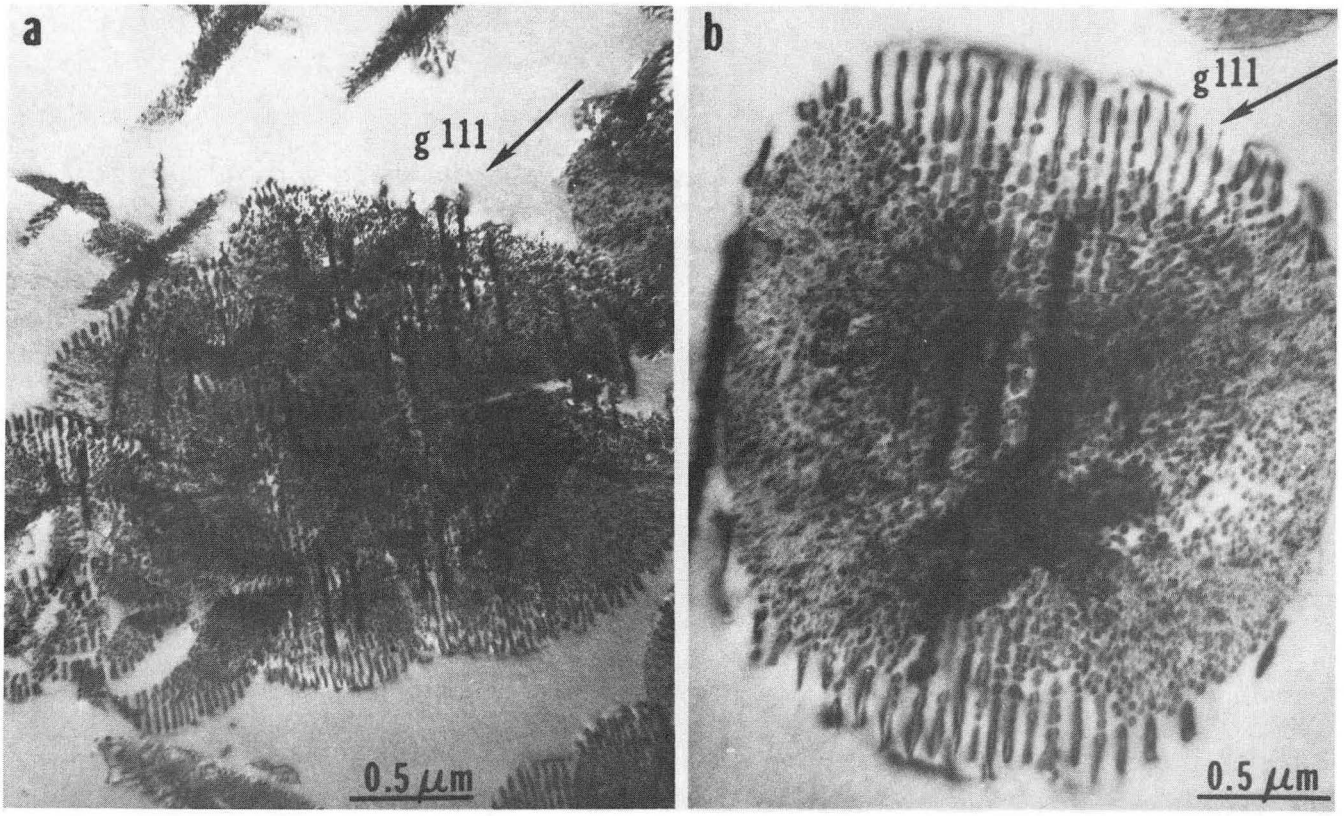

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Fig. 1

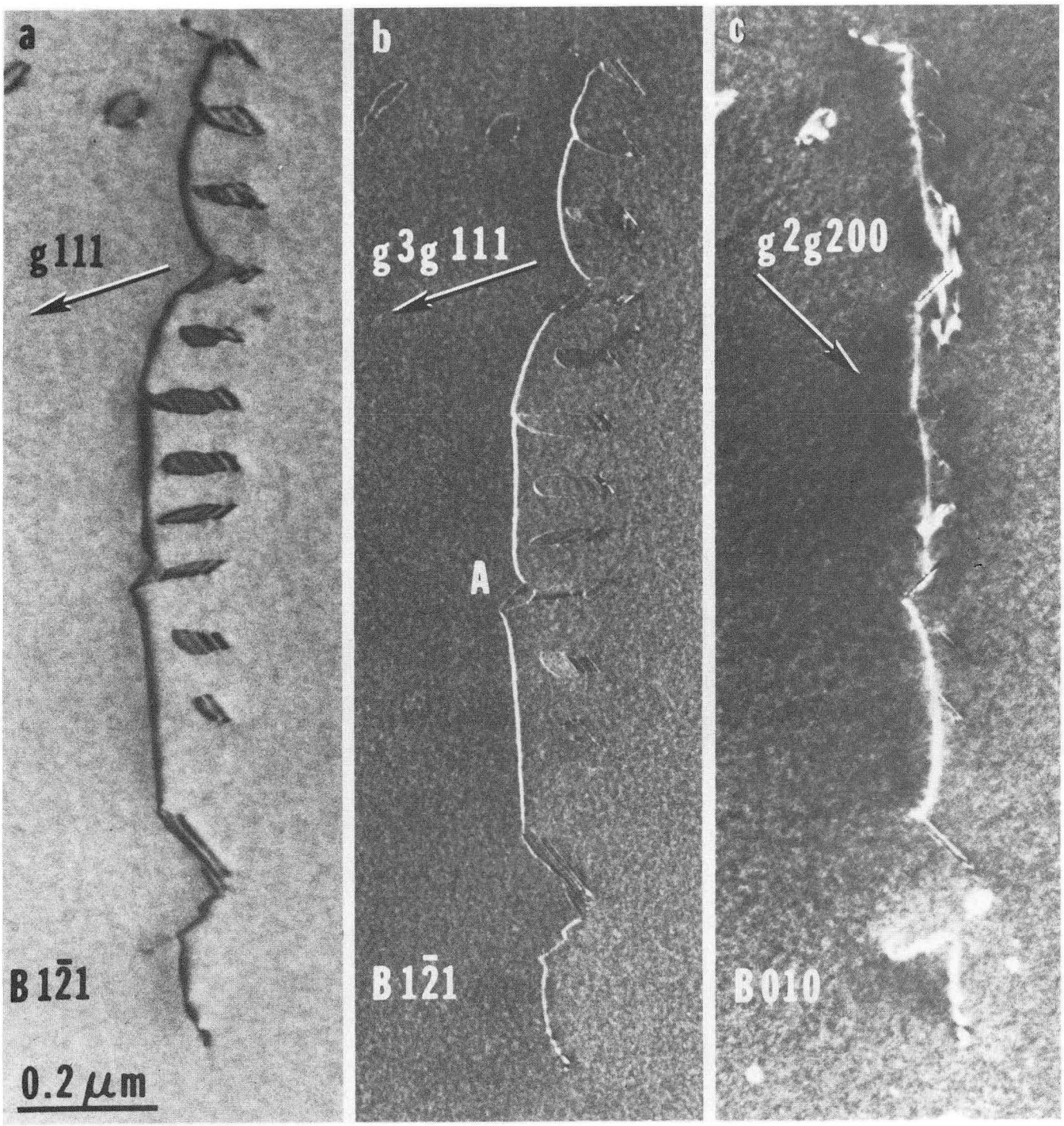

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Fig. 2

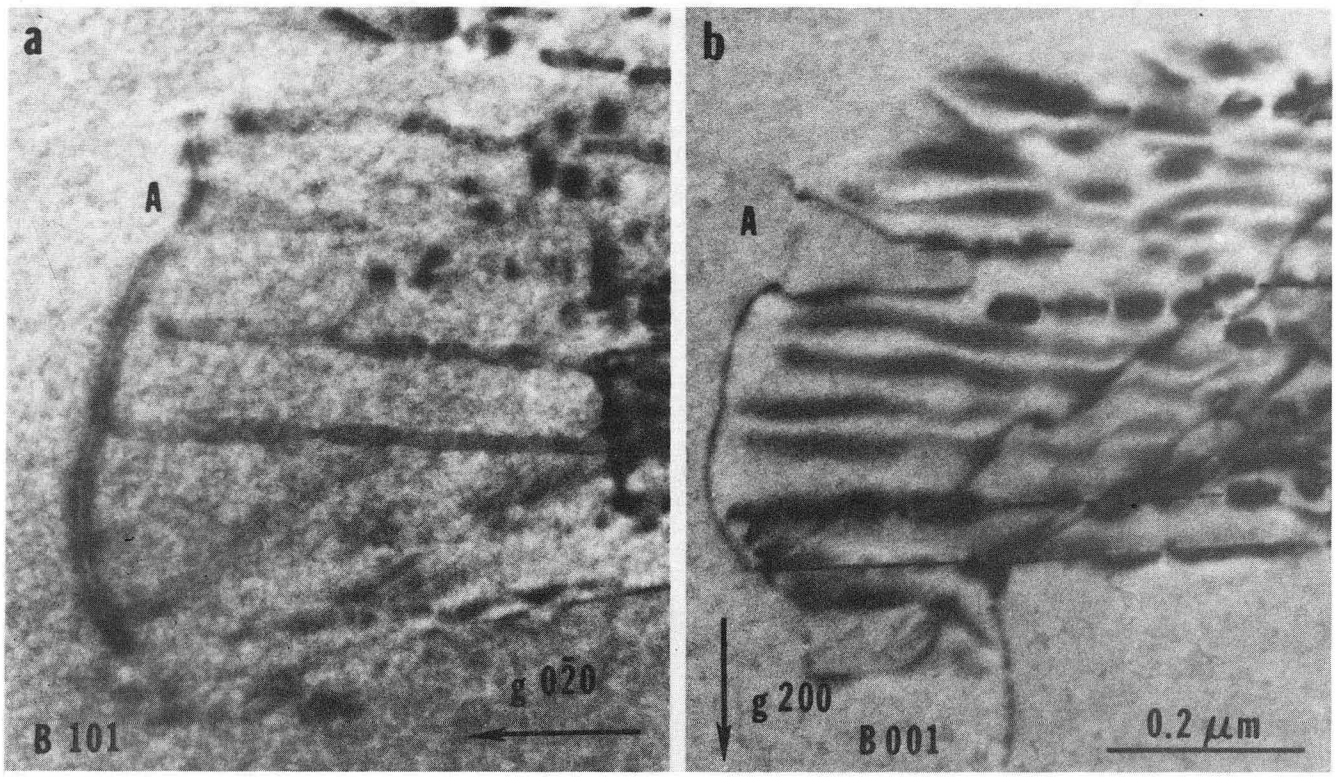

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Fig. 3a,b

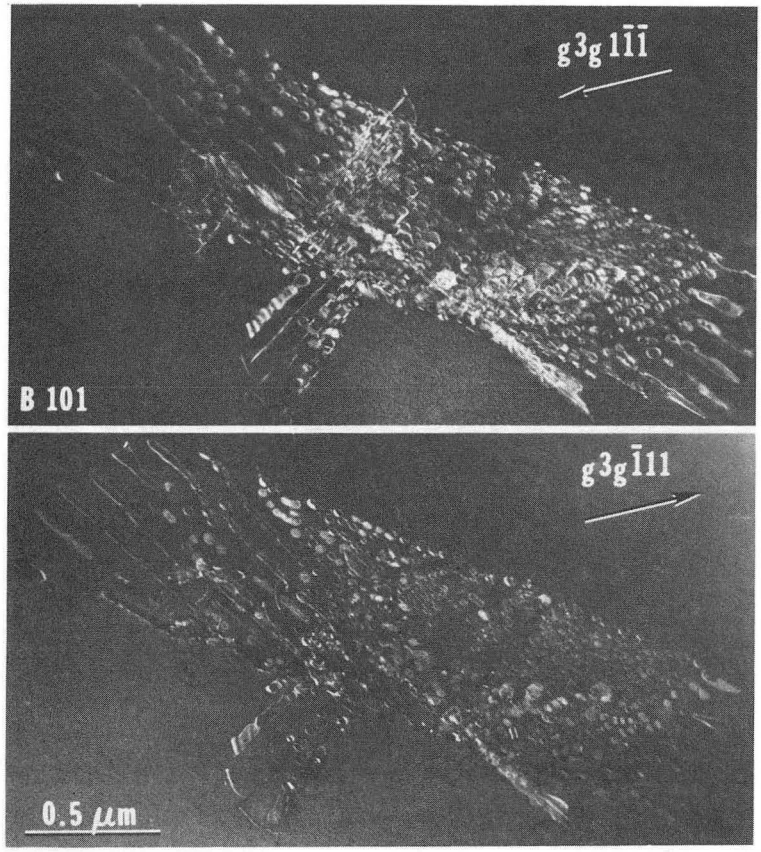

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Fig. 3c,d

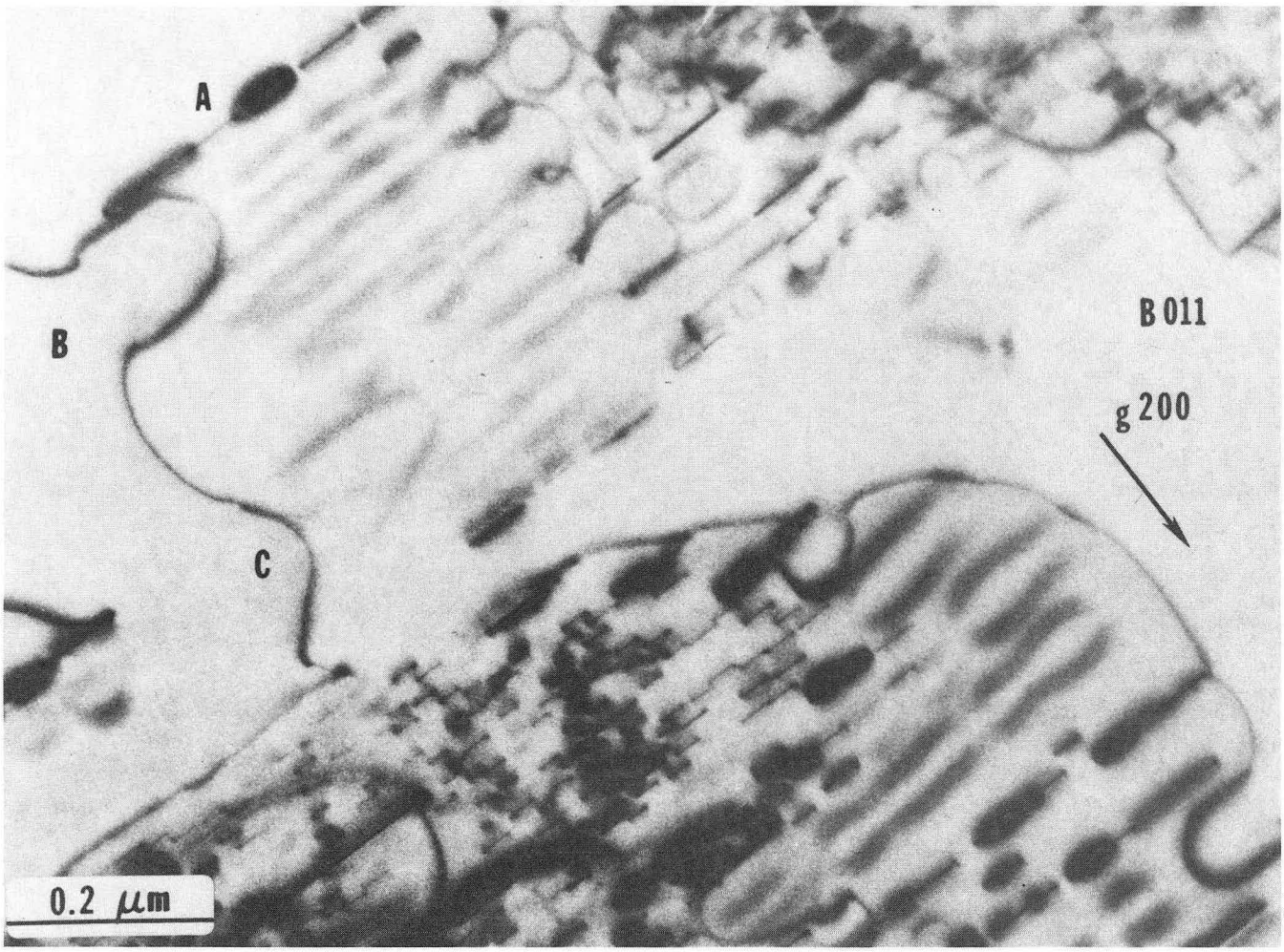

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Fig. 4

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