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Publication Date

1966-05-01

UCRL-16852

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For 6th International Congress for Electron
Microscopy - Kyoto, Japan - Aug. 28-Sept. 4,
1966. To be published in Proceedings

UCRL-16852

UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory
Berkeley, California

AEC Contract No. W-7405-eng-48

SOME INVESTIGATIONS OF PRECIPITATION

G. Thomas

May 1966

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1) Precipitation in npn Silicon (with E. Levine)

A detailed investigation has been carried out of the defects produced in silicon as a result of doping treatments with phosphorus and boron, (1). Due to the change in lattice parameter accompanying diffusion of the solute, a solute contraction stress is set up which results in the formation of dislocation networks, (1) (2). In the case of a npn transistor formed by double diffusion of P and B, we have also observed precipitates but only near the emitter surface (in the n-type layer). These precipitates form as plates on $\{111\}$, and it appears that these are produced concomitantly with the dislocations in such a way as to minimize the solute contraction stress, i.e., they are produced during the diffusion and not after subsequent cooling down. This result is deduced from the micrograph of Fig. 1(a). Here only one set of precipitates have formed on $(11\bar{1})$ and only one set of dislocations are seen, these lying normal to the precipitates along $[11\bar{1}]$. The Burgers vector of these dislocations is $a/2 [1\bar{1}0]$ and they are pure edge. Contrast experiments on the precipitates showed that they are extrinsic in sense and with displacements in $\langle 111 \rangle$ normal to the habit plane. Thus in $[112]$ one set of edge dislocations and a set of precipitates normal to the dislocations can minimize the solute contraction stress. Thus precipitates always form in $\{111\}$ planes most nearly normal to the diffusion front. In this respect precipitates and dislocations are equivalent and equivalent patterns of dislocations and precipitates are formed for other crystal orientations. The extrinsic character of the precipitates is confirmed from their diffraction patterns e.g., Fig. 1b. The precipitate patterns index as base centered orthorhombic (e.g., Fig. 1c) with $a=3.8\text{\AA}$, $b=6.6\text{\AA}$, $c=6.75\text{\AA}$. The c axis is parallel to the $[111]$ Si, hence one unit cell of the precipitate in the matrix can be regarded as an extrinsic Frank defect of displacement vector slightly larger ($\sim 7\%$) than $1/3 [111]$. Thus the "strength" of the precipitate is less than that of a dislocation ($b=a/2\langle 110 \rangle$) and so for equivalent stress relief, the precipitate spacing is less than that of the dislocation spacing (Fig. 1a).

Refs: 1) E. Levine, J. Washburn and G. Thomas, J. Appl. Physics, 1966 (in press).

2) H. J. Queisser, G. Thomas and J. Washburn, J. Appl. Physics 35, 1909 (1964).

2) Relplane Diffraction from Needle-Shaped Precipitates (with W. L. Bell)

Diffraction patterns from small needle shaped precipitates consist of sheets of intensity due to the effect of the shape factor in relaxing two laue conditions for each needle. We have investigated such effects in Al-Mg₂ Si (1). A typical micrograph of a $[110]$ foil in dark-field at $s < 0$ (2) is shown in Fig. 2(a). The needles lie along the $\langle 100 \rangle$ axes. Fig. 2(b) is a selected area diffraction pattern of 2(a). This pattern can be interpreted in terms of the sketch shown in Fig. 2(c). Each needle gives rise to a sheet of intensity normal to $\langle 100 \rangle$. The sheets consist largely of incoherent radiation. The intersection of the reflecting sphere with the intensity sheets for the inclined needles gives rise to curved streaks about $[002]$ whereas straight streaks are formed in projection when the intensity sheet is parallel to the beam. The modulations in intensity arise from the superposition of diffraction patterns from each precipitate and have maxima and minima related to the structure factor and shape factors for the needles, and strain effects. Contrast experiments in dark-field in two beam orientations indicate that the strain field is of interstitial type (See Fig. 1(a) (where the dark side of most images is in the direction of \bar{g}), as expected from earlier considerations. (1).

The sketch in Fig. 2(c) has been derived from examination of many diffraction patterns. We observe cancellation of intensity near forbidden matrix relpoints (100, 110 etc.) and enhancement near allowed points where all three relplanes intersect. This effect is illustrated in Fig. 2(d) taken near the $[103]$ orientation. Cancellation at intersecting streaks is observed near the 110 and equivalent forbidden positions. At these positions two or three laue conditions are satisfied due to contributions from all three orientations of needles, and so the structure factor can be weakly operating and largely removes the intensity at such positions by coherent interference. The sketch in (c) shows how the intensity appears at allowed and non-allowed relpoints.

Refs: 1) G. Thomas, J. Inst. Metals, 90, 57 (1961).

2) W. L. Bell and G. Thomas, Phys. Stat. Sol., 12, 843 (1965).

Figure Captions

Fig. 1(a) foils prepared of emitter surface of npn transistor (bright-field). Precipitates on $(11\bar{1})$ dislocations along $[11\bar{1}]$. (b) S.A.D. of 1(a) showing precipitate streaks and spots. Satellite spots are due to double diffraction. (c) explanation of pattern in (b), satellite spots indicated with crosses.

Fig. 2(a) foils of Al-Mg₂Si quenched and aged 5 hr. 220°C; dark-field at $s < 0$, only precipitates near top surface are in good contrast. The needles along $[100]$ and $[010]$ inclined at 45° exhibit extinction contrast and strain contrast. The strain contrast indicates interstitial strain fields. (b) S.A.D. pattern dark-field of (a) notice parabolic streaks spread about $[002]$ due to the $[100]$ and $[010]$ needles, and straight streaks along $[220]$ due to the $[001]$ needles. (c) Schematic representation of the intensity sheet distribution about reelpoints. (d) diffraction pattern $\sim [103]$ of needles showing cancellation of intensity at nonallowed reflecting positions (cf-c).

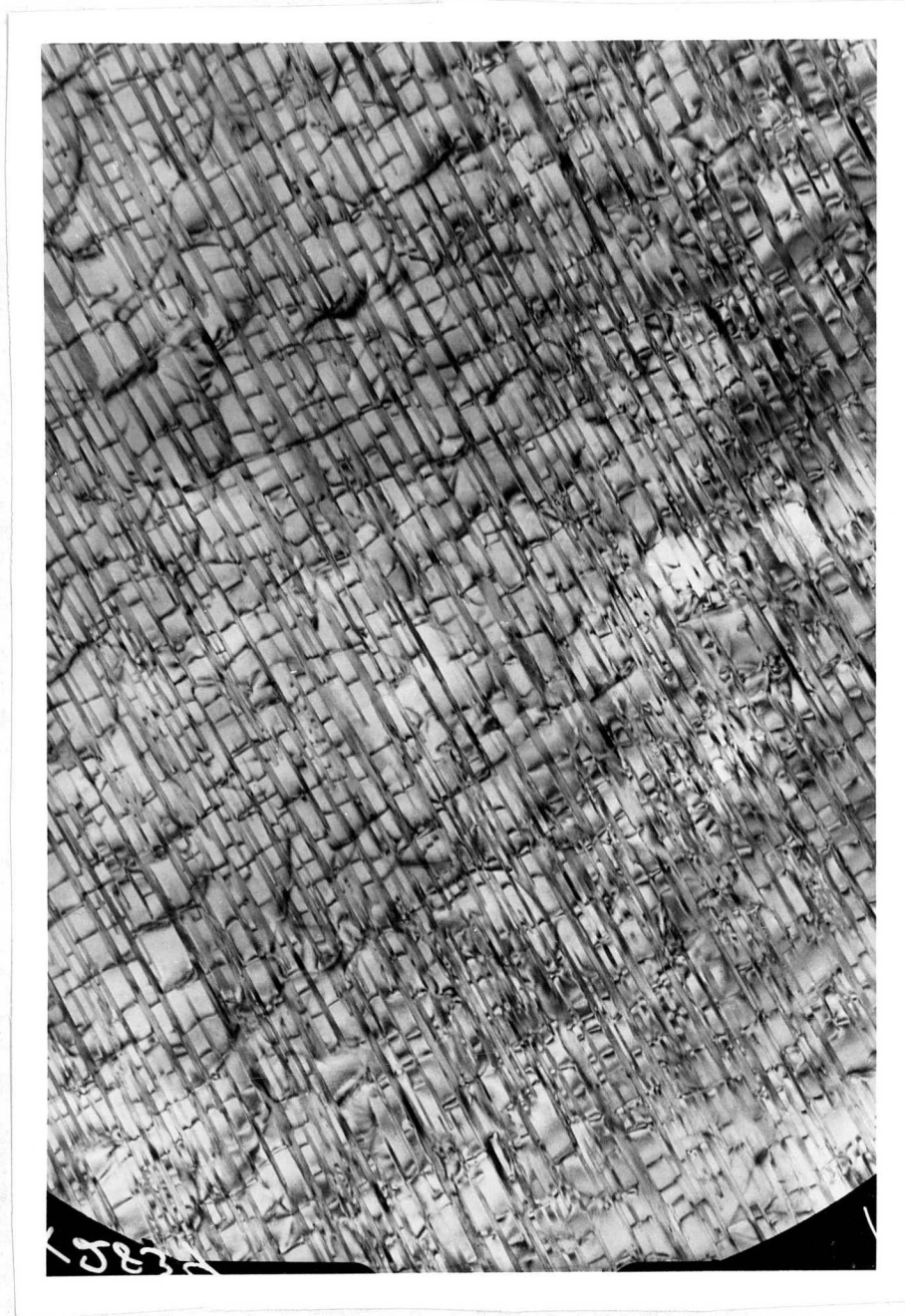


Fig. 1 (a)

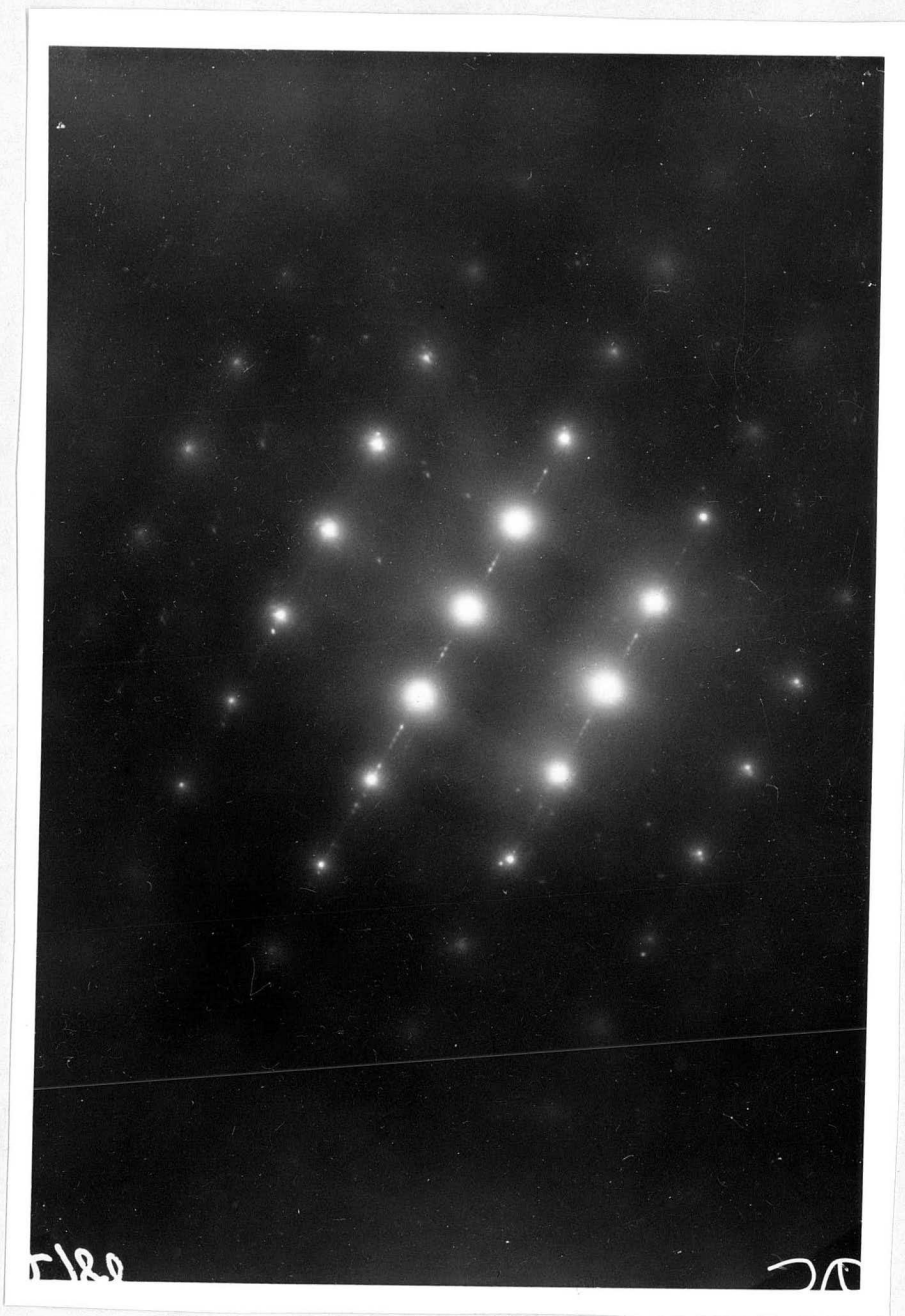
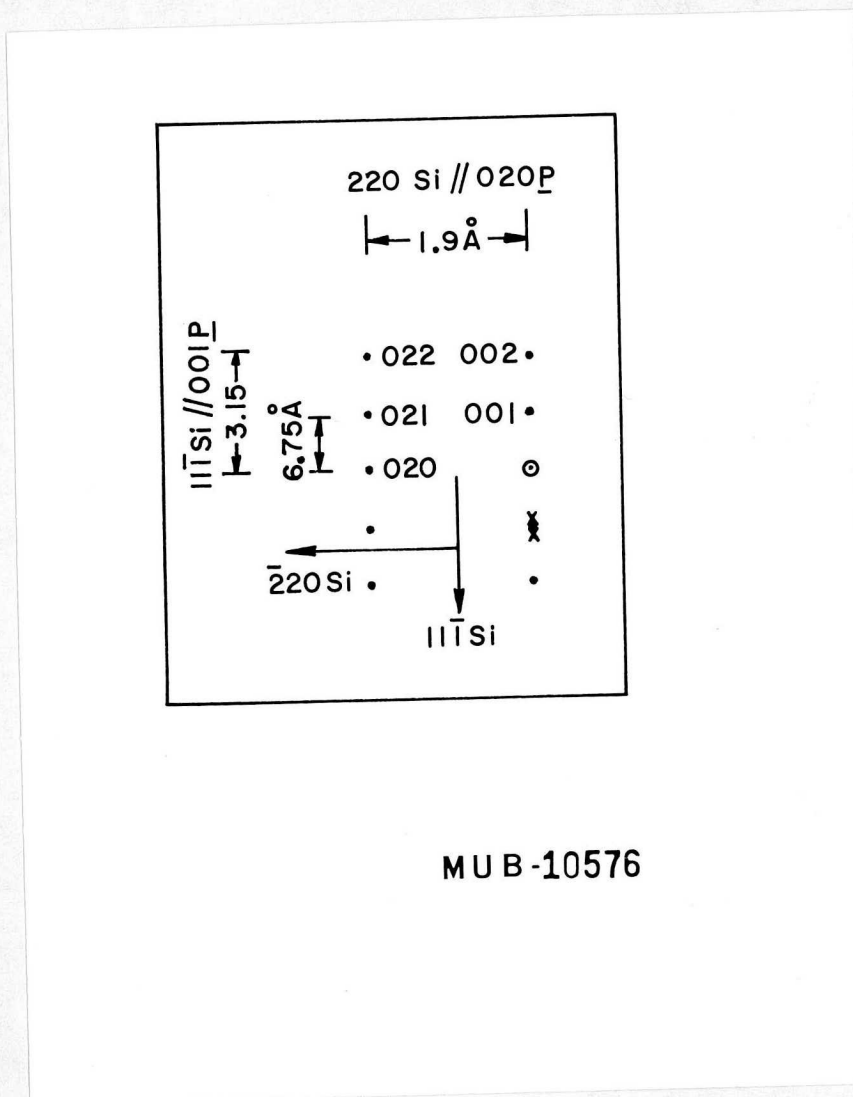


Fig. 1(b)



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Fig. 1(c)

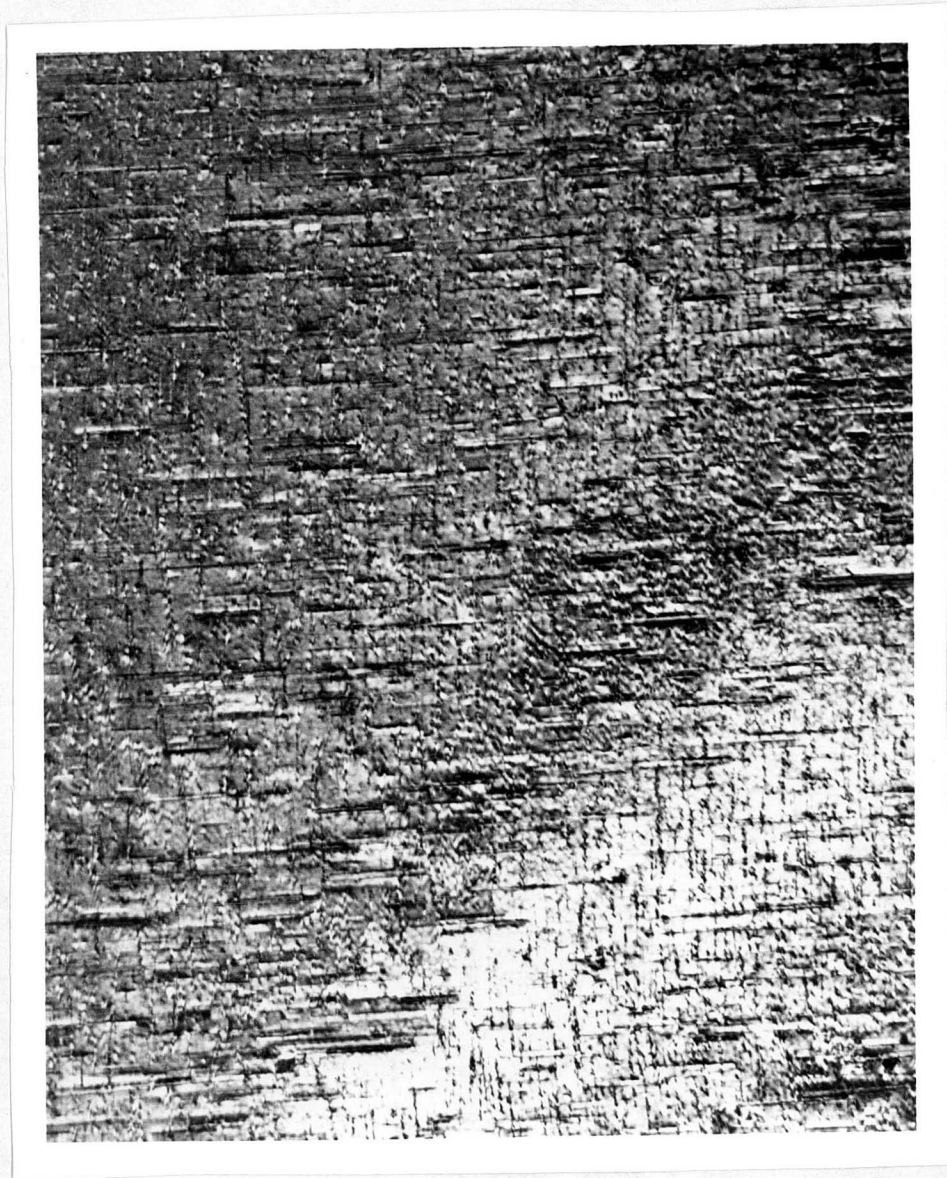


Fig. 2(a)

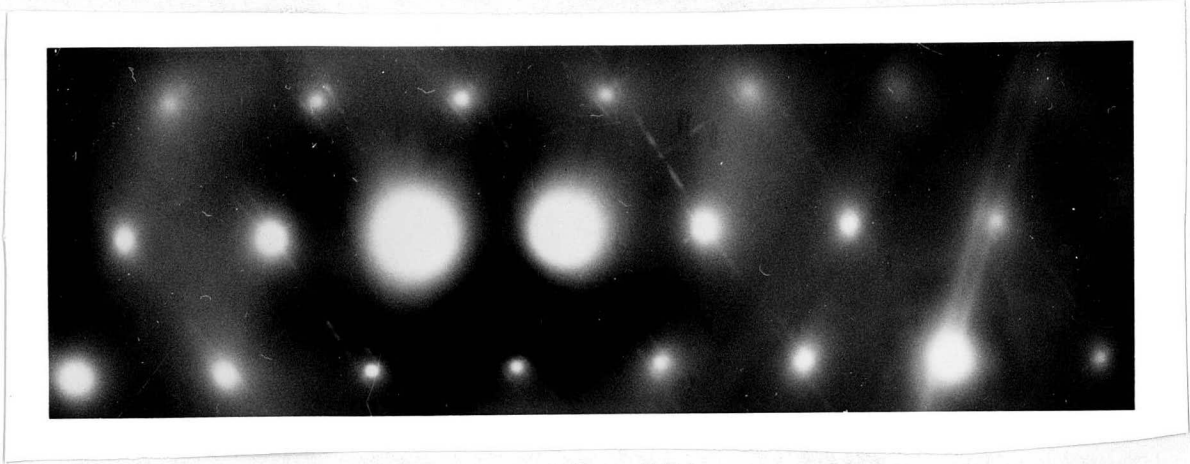


Fig. 2(b)

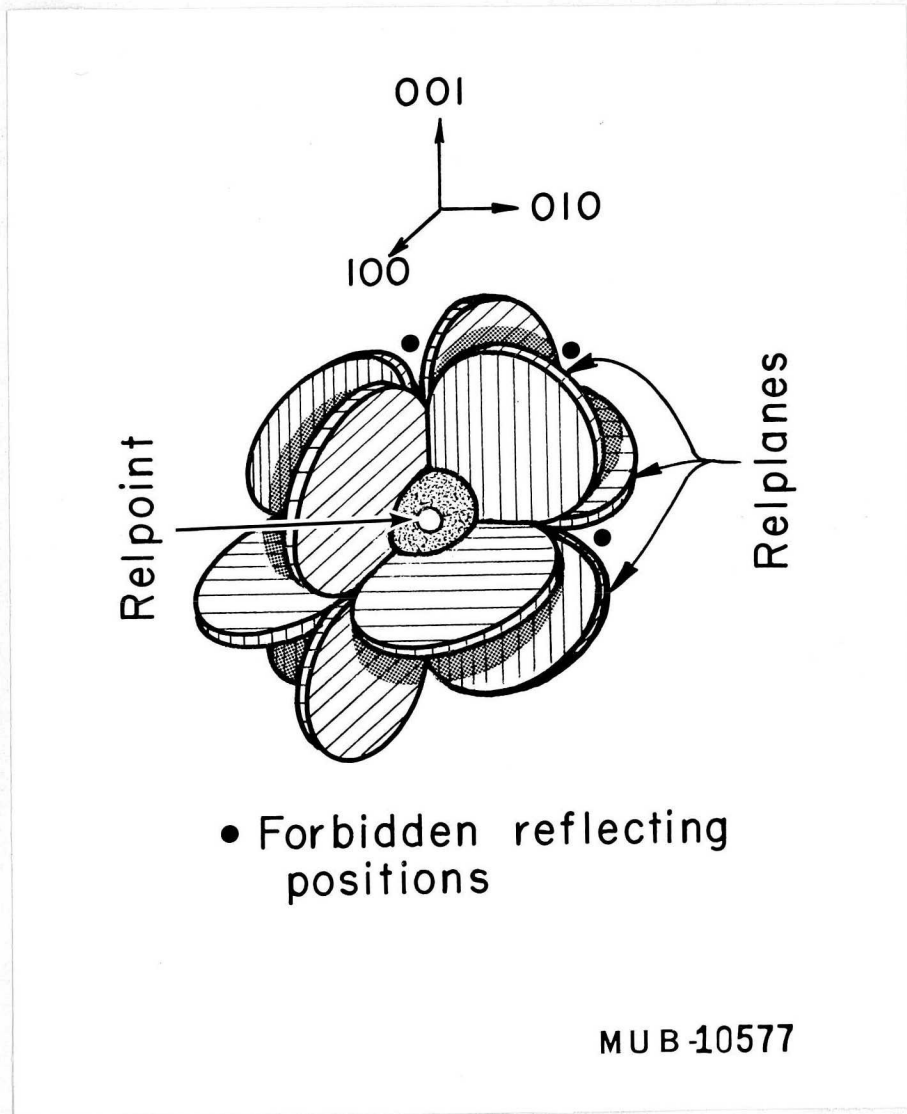


Fig. 2(c)

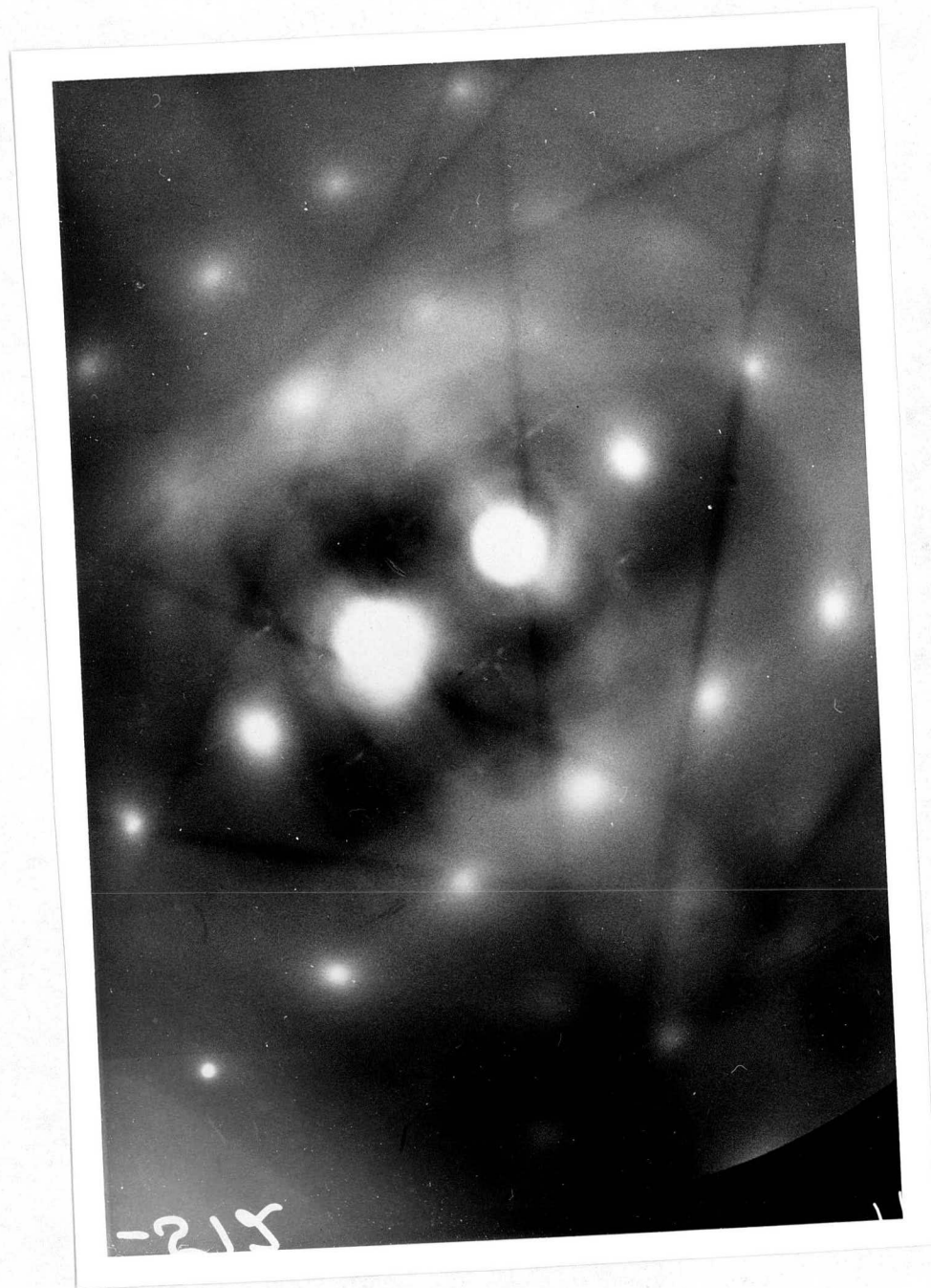


Fig. 2(d)

