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# HVEM IN CERAMICS RESEARCH

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# HVEM IN CERAMICS RESEARCH

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The particular advantages of high voltage electron microscopy viz., improved penetration, higher resolution and reduced ionisation damage, lend themselves well to problems in ceramics. In previous conferences attention has been paid to these factors (1) and considerable progress is now taking place. Even so, the field of microstructure-properties relationships in ceramic materials lags well behind these in physical metallurgy. In the present paper I shall summarise some of the research programs currently underway in my group viz., magnetic materials e.g. ferrites and refractory ceramics e.g. $Si_3N_4$ and its more complex derivatives the "Sialons".

## Refractory Ceramics - Silicon Nitride and Sialons

(a) Intergranular Phases: The potential advantages of refractory ceramics for high temperature applications such as turbines, liquid metal containers are well recognized (2) since they have very attractive properties (high modulus: density ratios, high melting points, oxidation resistance). However, due to fabrication difficulties the use of hot-pressing additives such as MgO or $Y_2O_3$ are needed and the properties at high temperatures are impaired. It has been proposed that the impairment is due to the formation of an intergranular phase, probably glassy as a result of the formation of silicates or crystalline oxy-nitrides. Attempts to prove this directly failed until high resolution TEM was adopted (3). The problem of resolving intergranular phases and whether they are amorphous or not is not trivial. From an electron microscopy viewpoint therefore, the following features at grain boundaries require characterization. 1) Detecting the intergranular phases and their distribution, 2) Determining whether these phases are crystalline, 3) Determining their chemical compositions.

From a morphological viewpoint it is essential to choose the proper orientation conditions as sketched in Fig. 1. Under conventional imaging conditions the grain boundary should be viewed edge-on with simultaneous strong Bragg excitation in adjacent grains. The contrast from intergranular phases if present and resolved depend upon whether they are amorphous or crystalline. Amorphous phases (most probably silicates) generally appear in light contrast (low atomic number--mass thickness contrast) as shown in Fig. 2a. Dark field imaging of the grains, although difficult, is preferable to enhance grain boundary interphase contrast. However, the failure to detect intergranular phases by this method (good down to about 20Å or so) does not necessarily mean that no intergranular phase is present. One must then resort to high resolution lattice or structure imaging as shown in Fig. 2b. This work must be done currently at 100kV due to the inferior resolution capabilities of our 650kV microscope, but with the addition of a new 1.5MeV instrument capable of lattice imaging to 2Å resolution, solution of these problems will be facilitated greatly.

is a void surrounded by a region of garnet, different in composition from the bulk due to microsegregation. The stress field around this generates some dislocations in the CGG as seen in this micrograph. Further work on the microsegregation problems requires HVEM and STEM to establish the actual nature of the segregating species.

Thus in conclusion, I would like to emphasize that HVEM is having a significant impact on ceramics research in answering some of the existing questions, and the development of HV-HREM and STEM will provide even further insight especially with regard to highly localized defects.

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FIGURE CAPTIONS

Fig. 1 Contrast conditions to detect intergranular phases; grain boundary must be oriented parallel to incident beam with strong Bragg excitation simultaneously in both grains. Lattice images can be obtained of diffraction planes corresponding to $g_1$ and $g_2$.

Fig. 2 a) Bright field image corresponding to Fig. 1 showing intergranular phase and the diffraction conditions. SAD B is from grain B and SAD A is from both the grains at A. Width of the intergranular phase decreases as indicated. 650KV. (T.M. Shaw)

b) High resolution lattice fringe image corresponding to Fig. 1 showing the crystalline grains and the noncrystalline intergranular phase near a triple grain junction. 100KV (D.R. Clarke)

Fig. 3 a) A lattice fringe image showing a third phase between two grains of $\beta$-$Si_3N_4$ and YSiON. Note the discontinuity of lattice fringes at the interphase boundary. 100KV.

b) Microanalysis trace obtained from an oxynitride phase. Note the presence of transition and alkali elements as impurities. (D.R. Clarke)

Fig. 4 a) Lattice fringe image from a polytype of MgSiAlON showing 32Å fringe periodicity. The irregularities in the fringe spacings are due to disorder in the polytype structure. 650KV.

b) Lattice fringe image from $Be_9Si_3N_{10}$ with the periodicity corresponding to the closepacked planes. Note the change in contrast in every fifth fringe in this 15R polytype, and the deviation from ideal structure at A with 6 fringes in one "block". (T.M. Shaw)

Fig. 5 a) Dark field ($g$-$\bar{1}10$) image of wedge shaped $LiFe_5O_8$ crystal near [332] symmetrical orientation. The domains labeled 1 and 2 have $P4_332$ and $P4_132$ structures respectively, and are separated by a boundary. (650KV)

b) Simulated micrograph of a). The boundary has not been simulated $\xi_0/\xi_0'=.07$, $\xi g/\xi g'=.05$ for all g. (O. Van der Biest) (Fig. 5 below)

Fig. 6 a) Darkfield image showing a stacking fault in $LiFe_5O_8$. The fault lies on [111] plane with $R=\frac{1}{6}$ [111] and is an anion fault. 650KV. (R.K. Mishra)

b) Screw dislocations in $Zn_2Ba_2Fe_{12}O_{22}$ with Burgers vectors [1210] on (0001). 650KV. (R.K. Mishra)

Fig. 7 BF image showing a void (A) in Gadolinium Gallium Garnet (GGG). The Region B surrounding the void has garnet structure and the contrast is due to the composition difference. Some dislocations (C) are present around B. 650 KV. (R.K. Mishra)

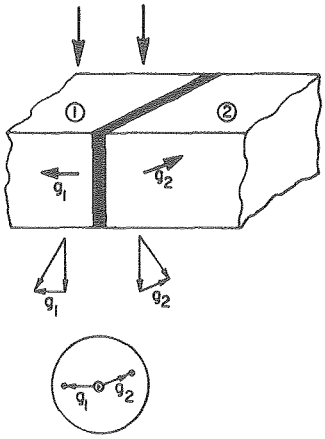


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Fig. 1

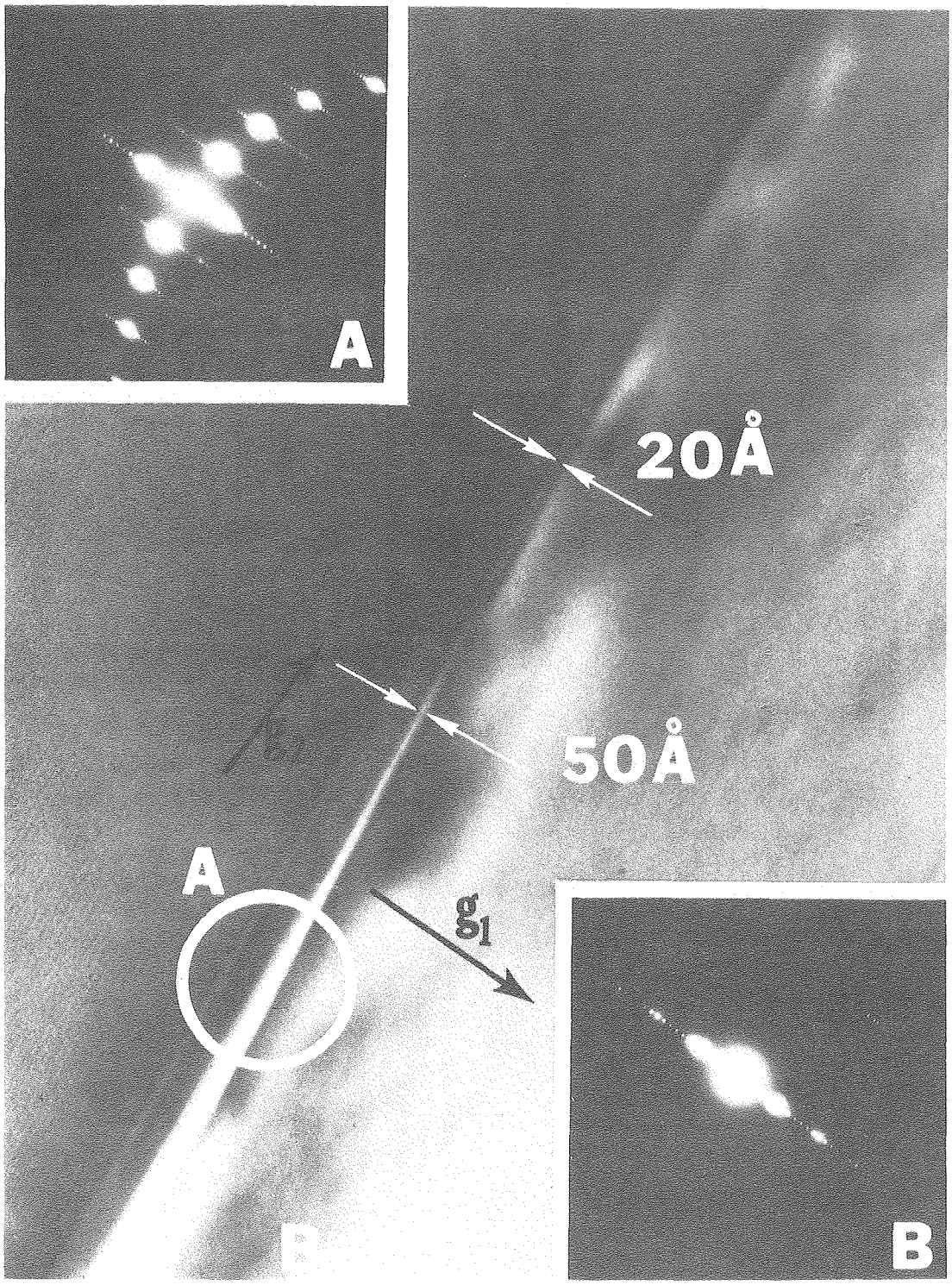


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Fig. 2a

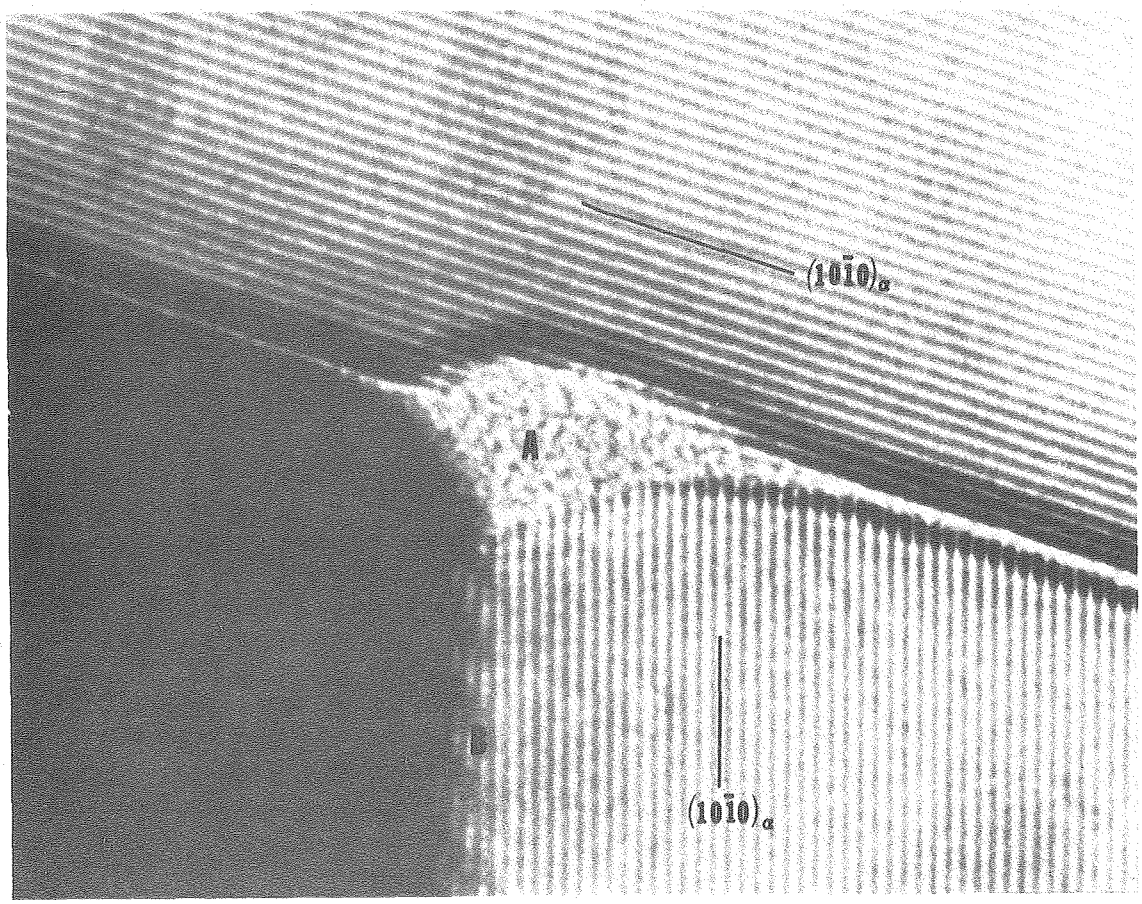


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Fig. 2b

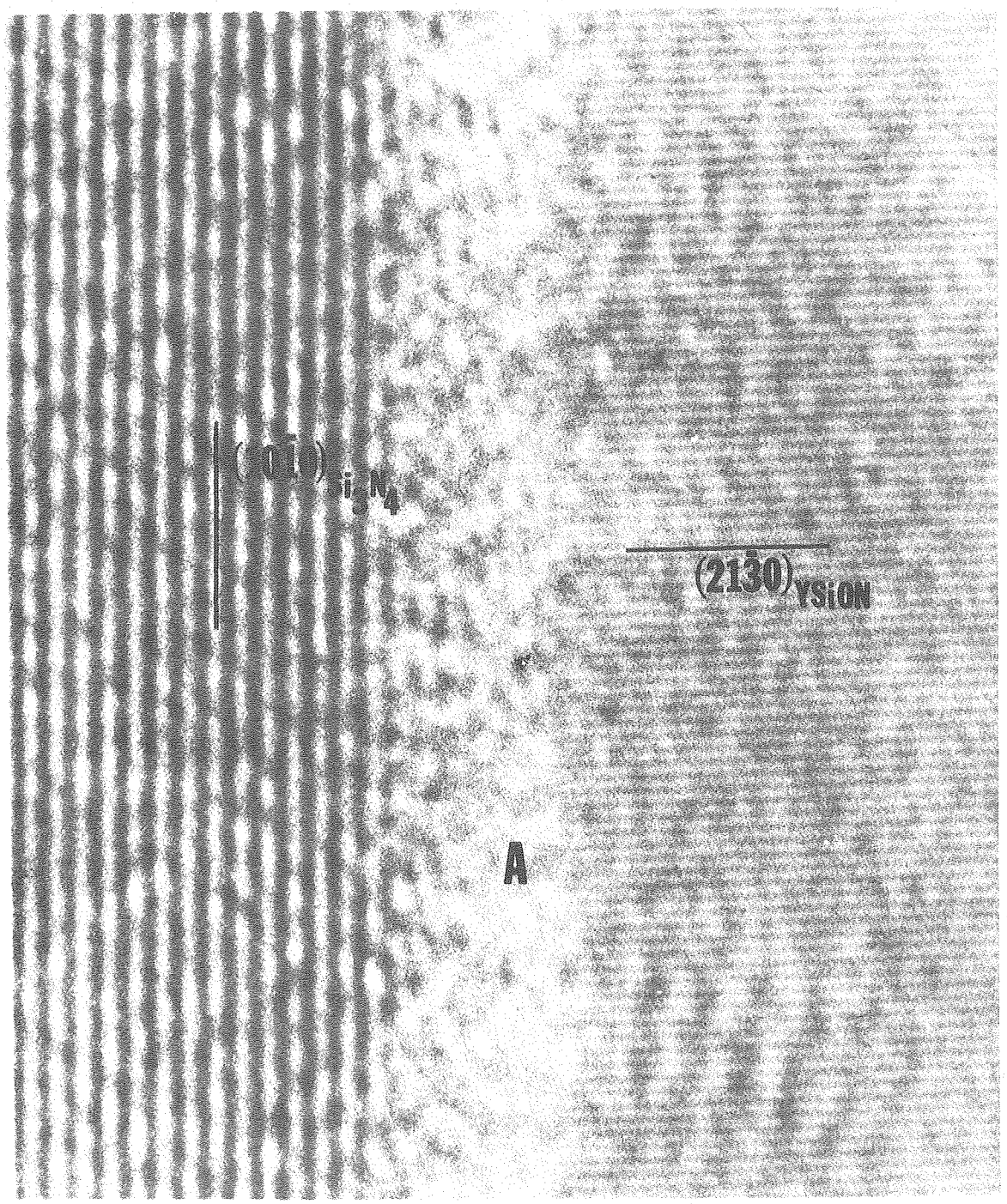


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Fig. 3a

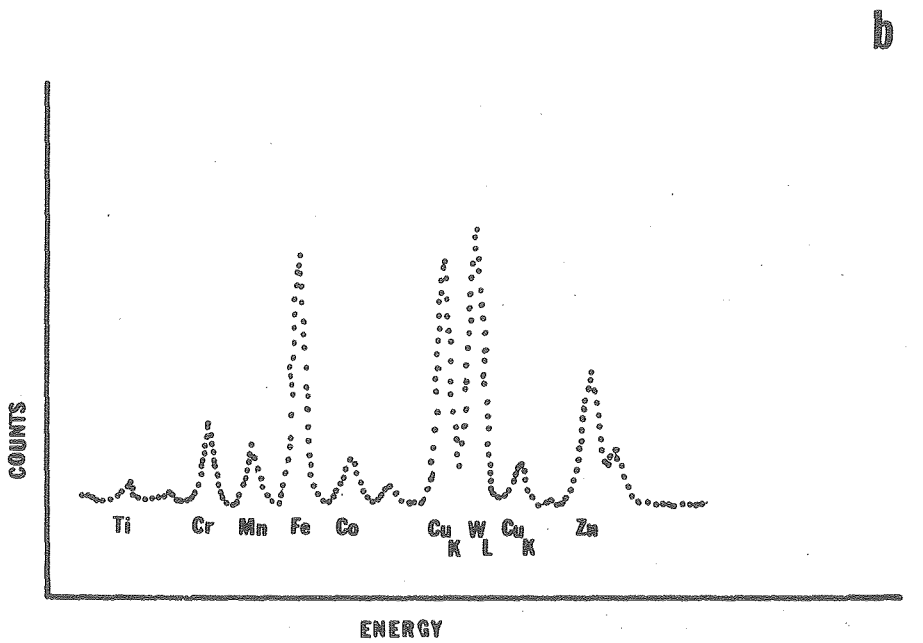


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Fig. 3b

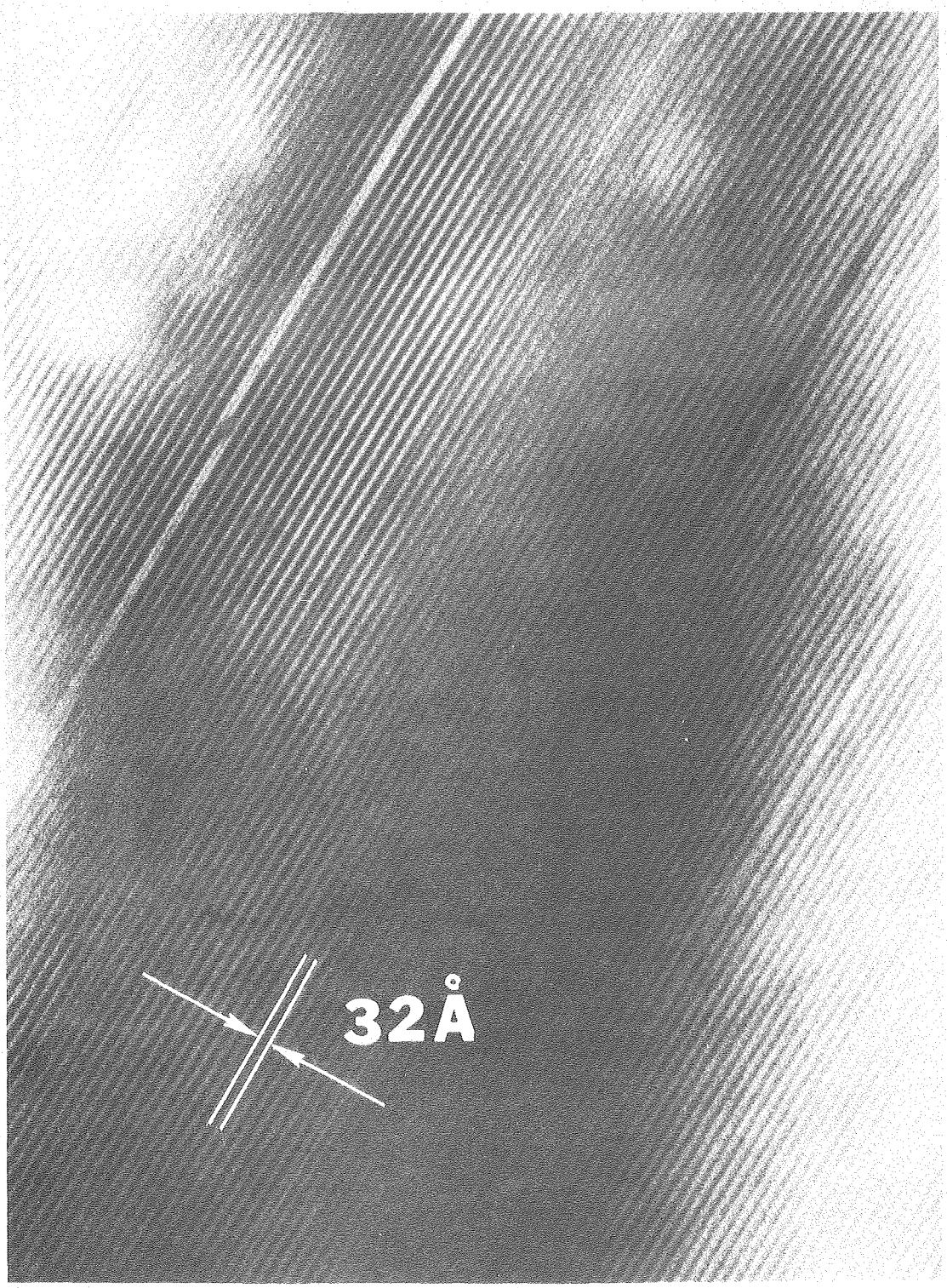


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Fig. 4a

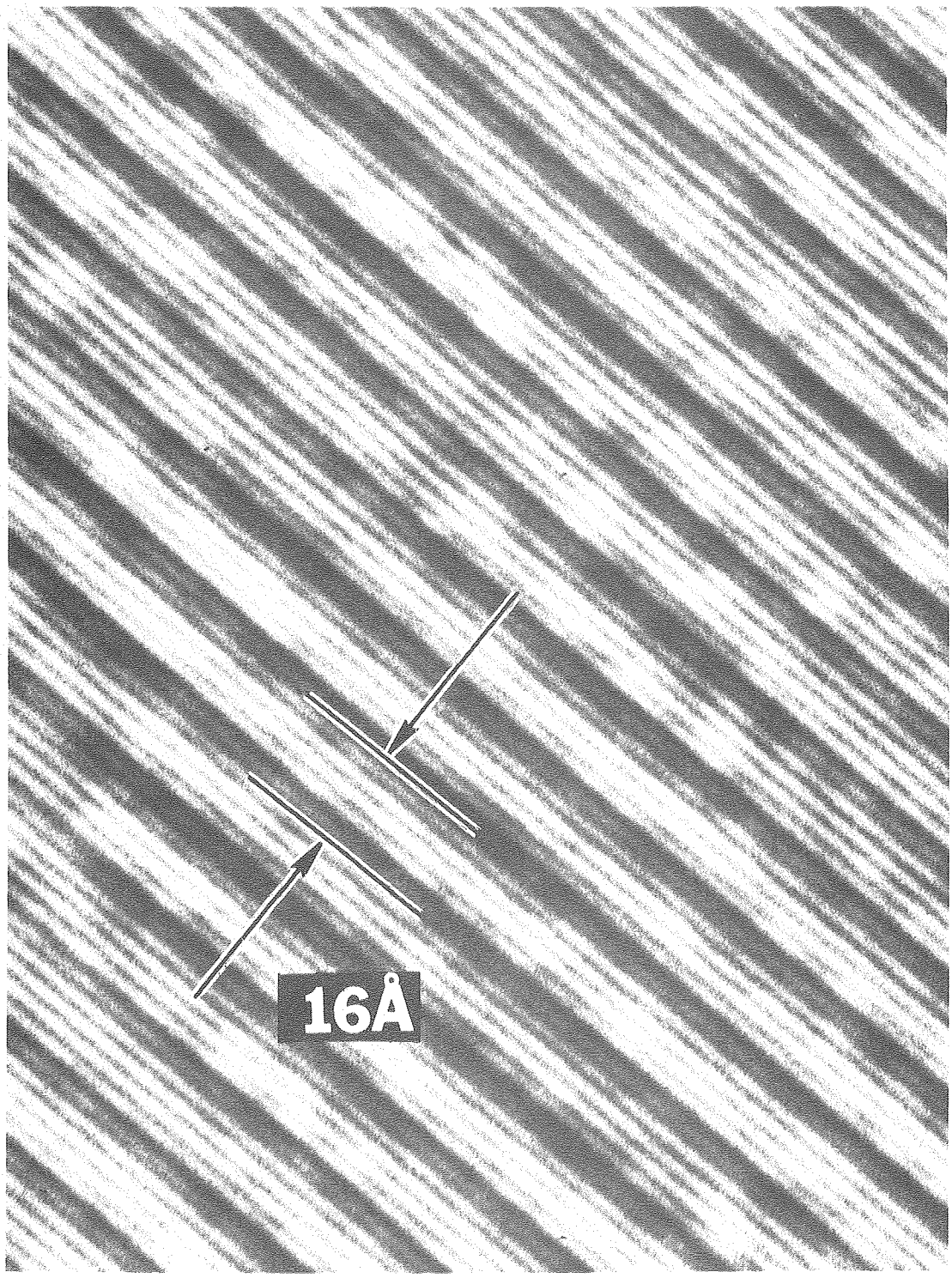


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Fig. 4b

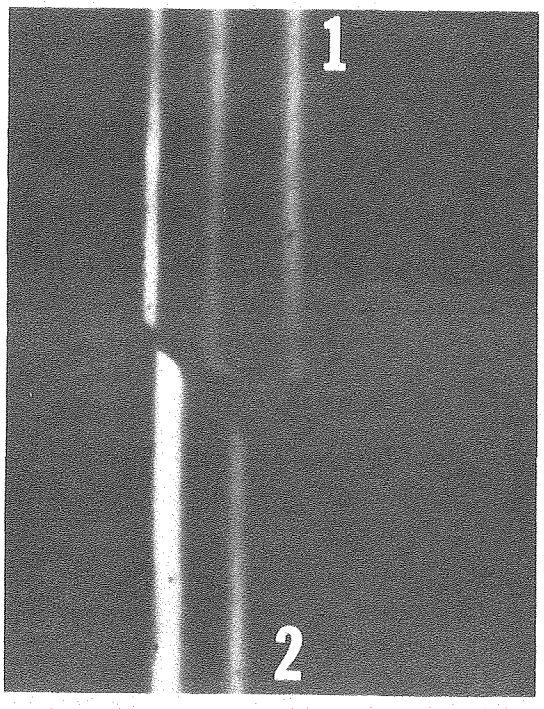


Fig. 5a

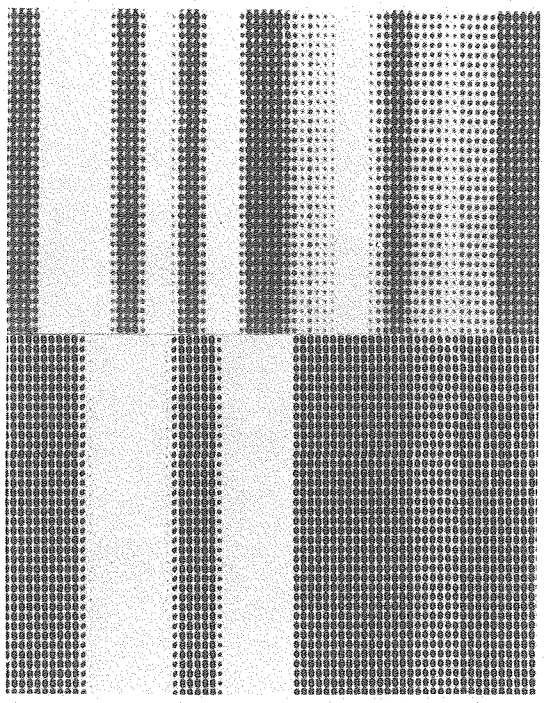

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Fig. 5b

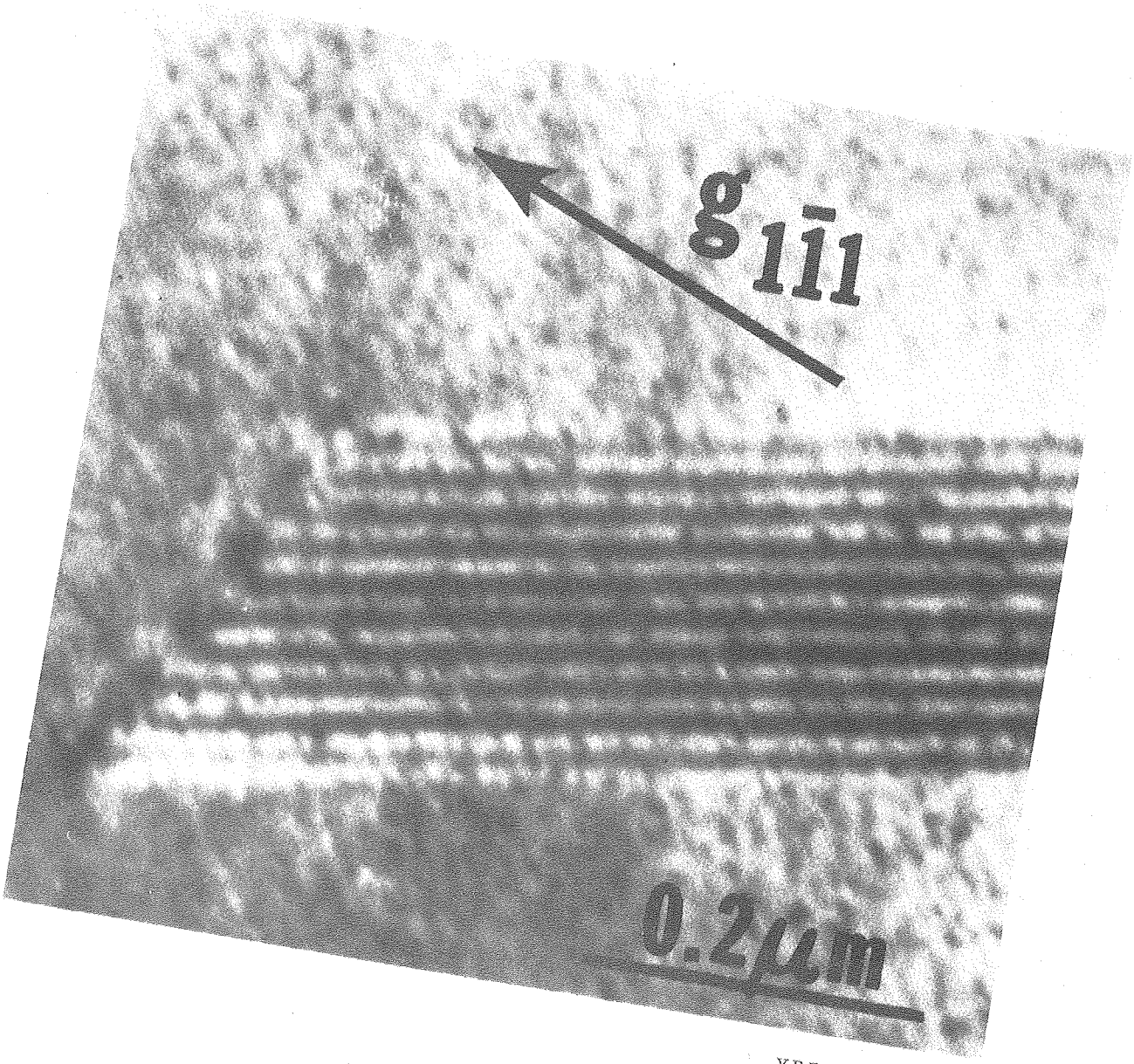


Fig. 6a

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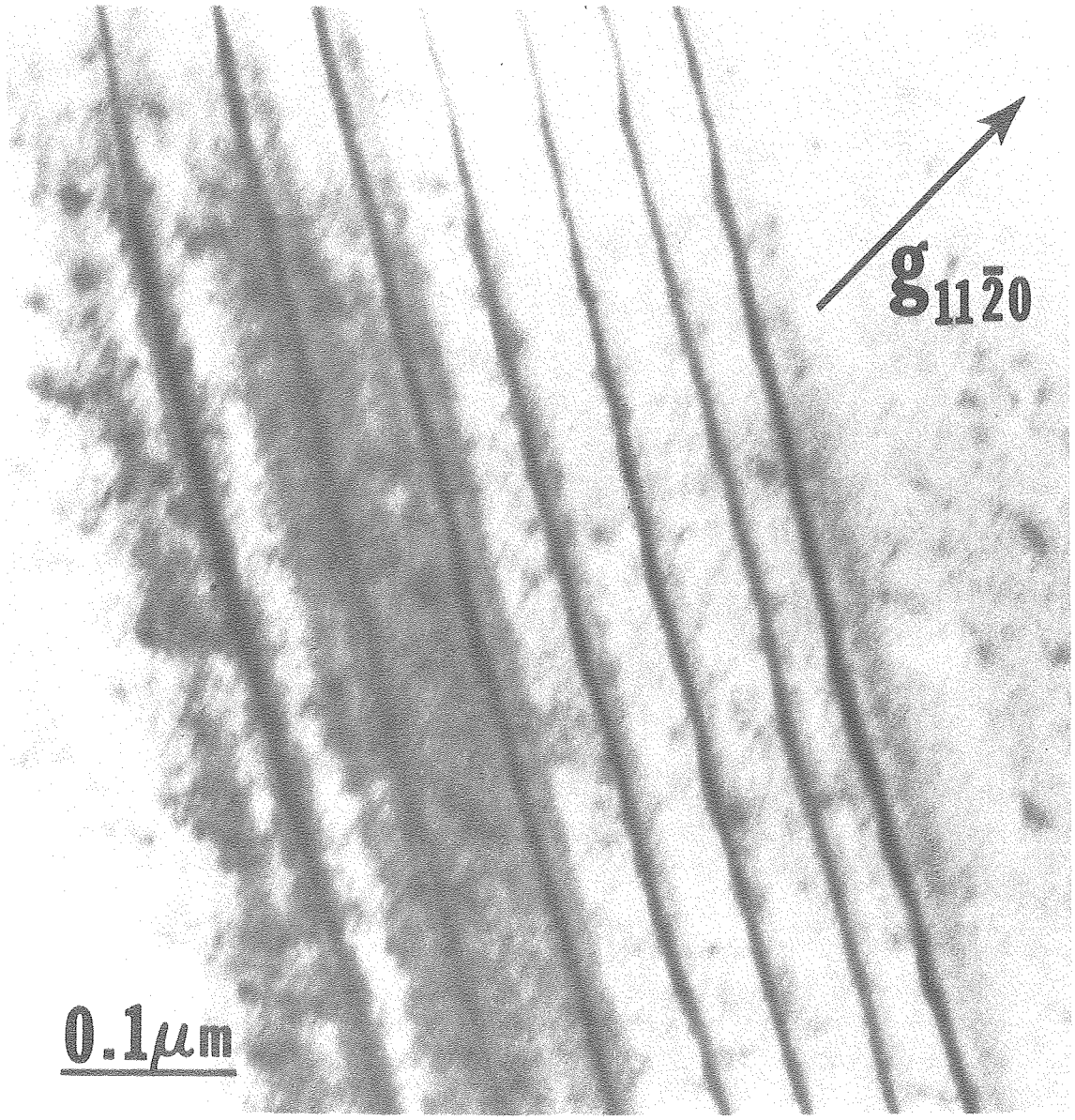


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Fig. 6b

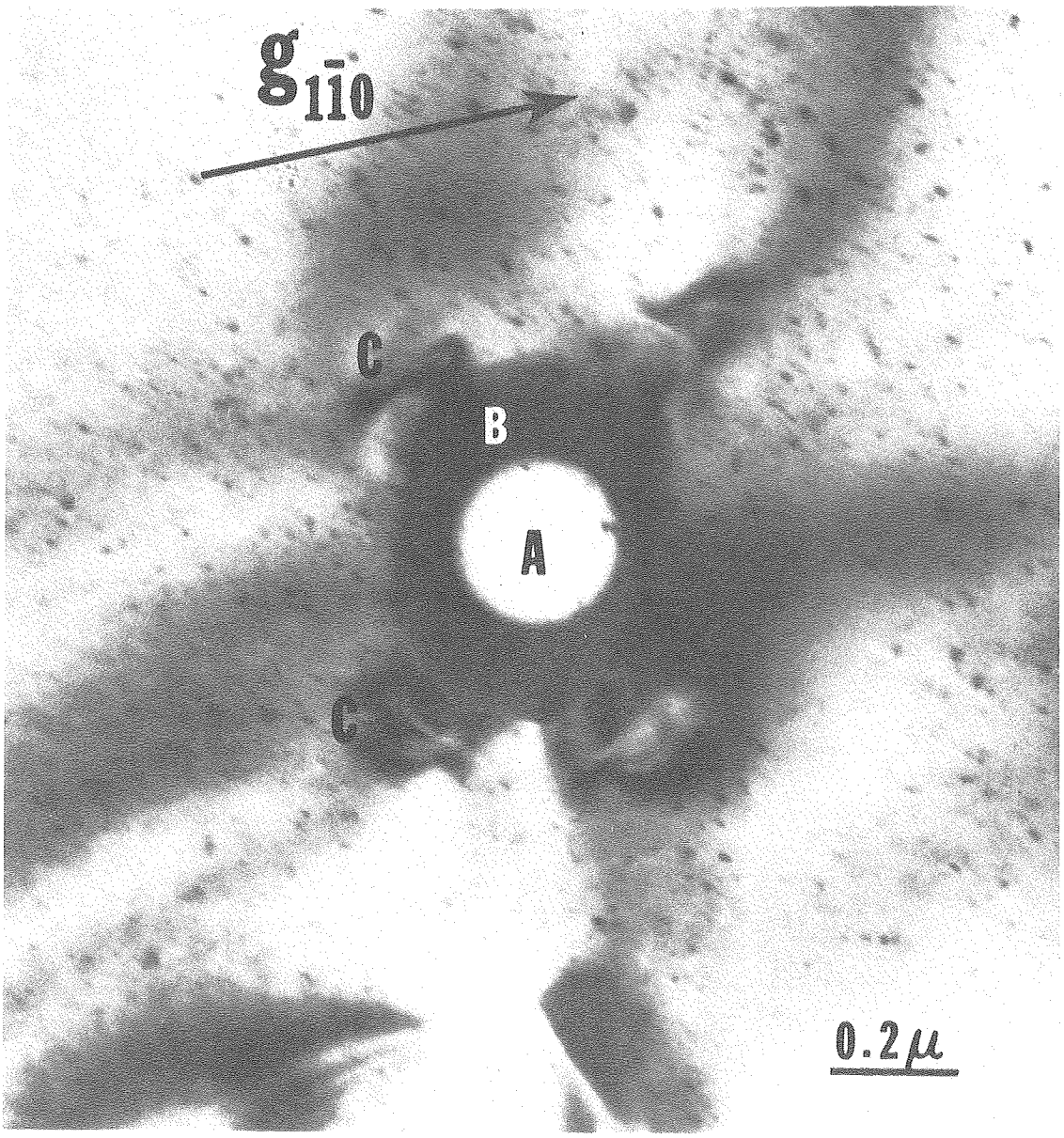


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Fig. 7

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