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R. Ramesh and G. Thomas

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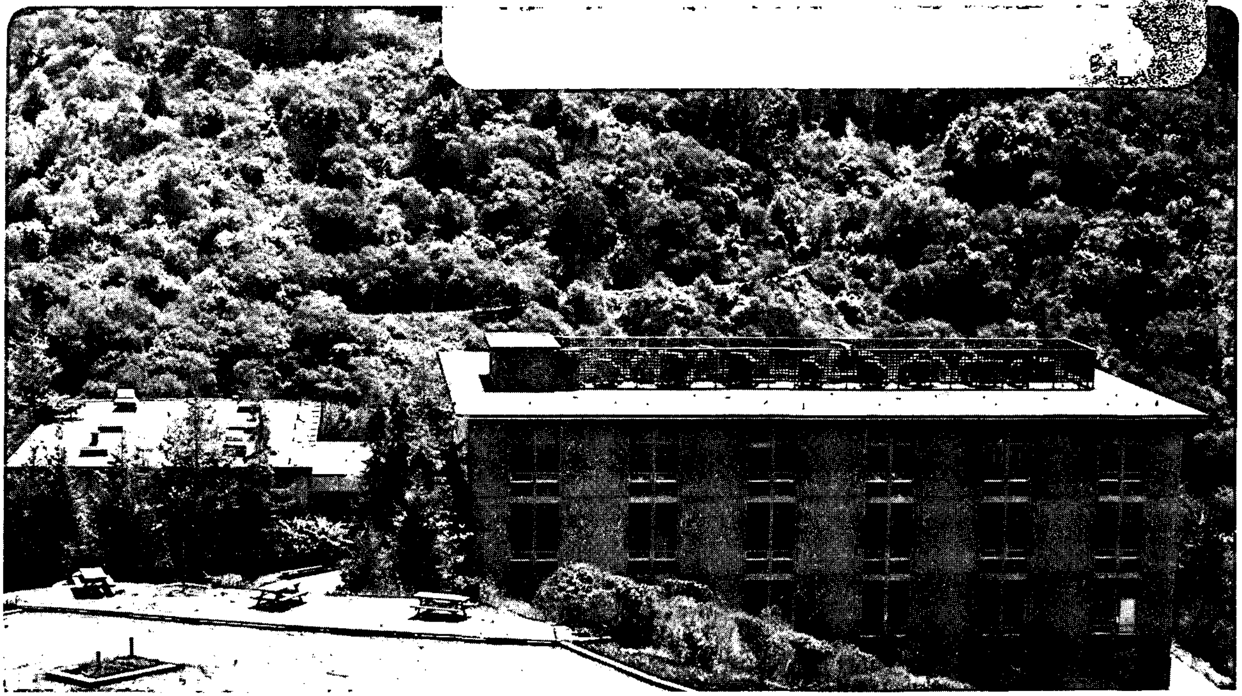

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HIGH VOLTAGE LORENTZ ELECTRON MICROSCOPY OF Fe-Did-B MAGNETS

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ABSTRACT : A high voltage lorentz electron microscopic investigation of the Fe-Did-B magnetic system has been carried out. The magnet consists of a multi-domain structure in the thermally demagnetized condition. Depending upon foil thickness, orientation and objective lens defocus, both stripe and bubble domains were observed. Domain walls were pinned at grain boundaries where a thin layer of a non-magnetic fcc phase existed. In the case where no fcc phase was present, the domain walls were continuous across grain boundaries. It is inferred that a thin layer of the fcc phase at the grain boundaries is essential to achieve high coercivities. The domain wall width was estimated from defocussing experiments to be about 50Å, in accordance with theoretical determinations.

INTRODUCTION : Considerable interest has been focussed on the Fe-Nd-B system since their discovery by Sagawa et.al$^{(1)}$ and Croat et.al$^{(2)}$ as a potential permanent magnet system. The magnet derives it excellent properties from the large magnetocrystalline anisotropy and saturation magnetization of the tetragonal phase, $Nd_2Fe_{14}B$. This paper focusses on the domain structure and microstructure-domain wall interaction in sintered Fe-Rare Earth-B magnets. In the sintered magnets, the high coercivity is attributed to the difficulty in nucleating reverse domains$^{(3-5)}$.As in the case of Sm-Co alloys, pinning also plays a significant role in the enhancement of coercivity. Domain walls can be examined at high resolution by Lorentz Electron Microscopy (LEM). The advantages of high voltage LEM are also well established$^{(7,8)}$. In this paper we report results of High Voltage LEM studies on a derivative system of Fe-Nd-B, Fe-Didymium-B, where didymium refers to Nd-Ce-Pr.

EXPERIMENTAL : The composition of the alloys used in this study was Fe-32.5 wt.% RE-1Wt.%B, wherein the rare earth composition was 80%Nd-15%Pr-5%Ce. The samples were examined in the sintered and slow cooled condition, whence the magnetic properties were optimized. The samples for electron microscopy were prepared by first mechanically thinning down foils to about 75 micrometers and then Argon ion milling 3 mm discs to electron transparency. Lorentz microscopy was carried out using a Hitachi HU 650 microscope operated at 500kV in the Fresnel mode. Domain wall widths were measured on divergent wall images and were done using an optical comparator. Samples were examined with (a) the c-axis normal to the foil ; (b) c-axis in the plane of the foil.

RESULTS AND DISCUSSION : In foils with the c-axis normal to the foil, stripe and bubble domains were generally observed. Since the foils were observed in the presence of the magnetic field of the objective lens (about 15kOe at 500kV)$^{(9)}$, there was a favourable orientation of the magnetization leading to domains of unequal width. Bubble domains were observed for certain values of foil thickness and objective lens defocus. Fig.1 depicts all of these observations. Note that bubble domains form from the stripe domains in the thinner regions of the foil through a Rayleigh instability(10). Of greater interest, however, is the domain configuration close to the grain boundaries and the interaction of domain walls with microstructural features such as grain boundaries, second phase at grain boundaries, inclusions, etc. Since it had already been shown earlier that a non-magnetic Nd-rich fcc phase exists at all triple grain junctions and frequently extends into two grain boundaries(11,12), the effect of this phase on domain walls was examined. As a comparison, grain boundaries where no fcc phase existed, were also examined.

In the case where no fcc phase isolated the grains of $Nd_2Fe_{14}B$, domains and domain walls were continuous across grain boundaries. This was observed in foils of both orientations. Fig.2 is a Lorentz micrograph of a sample with the c-axis normal to the foil showing domain walls which are continuous across the grain boundaries, indicated by the arrow. Fig.3(a) and (b) show a similar case for the c-axis in-plane sample. Fig.3(b) is the in-focus image of the lorentz image shown in Fig.3(a). In both the cases, no fcc phase was detected by diffraction. Note, in Fig.2, that a thin domain is pinned to the grain boundary. This pinning appeared to be strong since the walls could not be unpinned by tilting the foil through large angles. On the other hand, the domain wall in Fig.3 could be easily moved by tilting the foil, which changed the in-plane component of the objective lens field. The main feature of the two images, however, is that in both cases there is no fcc phase at the grain boundaries and in both cases the domain wall is continuous across the grain boundary. This indicates magnetic flux continuity, even across a disordered region such as a grain boundary and supports the idea that the domain structure in a polycrystalline magnet is considerably influenced by interactions across grain boundaries(13). In the case of easy axis aligned grains (as in this case), the interaction may be even stronger. In contrast, when a non-magnetic phase separates two grains, the domain walls terminate at the interface and there is no correspondence of domains in the two grains. One such example is shown in Fig.4(a&b). Fig.4(a) is the bright field image showing the fcc phase at the interface, while Fig.4(b) is the corresponding Lorentz image showing domain walls in one grain but not continuous with those in the other grain.

This type of interaction of the domain wall with the fcc phase may be very significant in explaining the reversal mechanism in this magnet. The presence of a contiguous film of the non-magnetic phase along the grain boundaries may mean that each grain would require a separate nucleation event to reverse its magnetization. On the other hand, in the absence of this phase, adjacent grains may reverse through a single nucleation event on any one of them. Thus, the group of grains behave collectively, as a larger particle. This aspect is currently being investigated in detail.

DOMAIN WALL WIDTHS : Domain wall width measurements were carried out by the linear extrapolation technique(14,15). Divergent wall images were recorded for various known defocus values of the objective lens, for fixed settings of the condenser lens. Thus, at a defocus value "z", the divergent wall image width is given by : $W_z = 2.W_O + 2z\emptyset.(B_y/B_s)$, where B_y is the in-plane component of the magnetic field and $\emptyset = e.t.B_s/m.v$, the maximum Lorentz deflection angle (e=electron charge; m=electron mass; t=foil thickness; v=electron velocity and B_s=the saturation magnetization). Thus, the wall width can be determined by extrapolating the divergent wall width at defocus "z" to z=0. Fig.5 shows a plot of divergent wall image width as a function of the objective lens defocus. Both underfocussed as well as over focussed conditions were used. The linear extrapolation =led to a wall width of about 55Å. This value agrees well with those obtained from theoretical calculations. For a 180° wall, the width is given by $2W_o = \pi(A/K)^{1/2}$, where A is the Exchange constant and K, the anisotropy energy constant. Using published values of A and K, the wall width can be calculated to be 52Å(16). The wall width estimated by this experiment is in good agreement with those determined by Suzuki et. al(17).

CONCLUSIONS : It has been shown that domain walls in this hard magnetic system can not only be imaged by High Voltage Lorentz microscopy but also moved by using the goniometer tilt stage and the field of the objective lens. By this method it has been shown that domain walls in general are not affected by the presence of grain boundaries. Thus, they have been observed to easily cross grain boundaries. On the other hand, when a thin contiguous layer of the non-magnetic Nd-rich phase is present along the grain boundaries, the walls are pinned at the interface of the $Nd_2Fe_{14}B$ grain and the grain boundary and are unable to move from one grain to another. This observation may be of importance in explaining the reversal mechanism in this magnet. Domain wall widths have been estimated using a linear extrapolation technique from defocussed divergent wall images. The wall width obtained agrees well with theoretical calculations and with recent experimental findings.

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REFERENCES

(1) M.Sagawa, S.Fujimura, M.Togawa, H.Yamamoto and Y.Matsuura, J.of Appl. Phys., Vol.55(1983), 2083.

(2) J.F.Herbst, J.J.Croat, F.E.Pinkerton and W.B.Yelon, Phys. Rev.B 29(1984), 4176.

(3) M.Sagawa, S.Fujimura, H.Yamamoto and Y.Matsuura, IEEE Trans. on Magn., MAG-20(1984), 1584.

(4) J.D.Livingston, in Eighth Int. Workshop on Rare Earth Permanent Magnets, Ed. K.J.Strnat, University of Dayton, Ohio, (1985), 423.

(5) G.C.Hadjipanayis, K.R.Lawless and R.C.Dickerson, J. of Appl.Phys., Vol.57(1985), 4097.

(6) P.Hirsch, A.Howie, R.B.Nicholson, D.W.Pashley and M.J.Whelan, Electron Microscopy of Thin Crystals, R.E.Krieger Publishing Co., 1977.

(7) T.Suzuki and M.Wilkens, Phys., Stat., Sol., Vol.8(a)(1971), 167.

(8) T.Suzuki and A.Hubert, Phys.Stat. Sol., Vol.38, K5(1970),K5-K8.

(9) P.J.Grundy, D.C.Hothersall, G.A.Jones, B.K.Middleton and R.S,Tebble, Phys. Stat. Sol., Vol.9(1972), 79.

(10) Lord Rayleigh, Proc. of London Math. Soc., Vol.10(1879), 88.

(11) R.Ramesh, K.M.Krishnan, E.Goo, G.Thomas, M.Okada and M.Homma, J. of Mag. and Magn. Matls., Vols. 54-57(1986), 566.

(12) R.Ramesh, J.K.Chen and G.Thomas, Submitted to J. of Appl. Phys.

(13) D.J.Craik and R.S.Tebble, Ferromagnetism and Ferromagnetic Domains, North Holland Publishing Co., 1965.

(14) L.Reimer and H.Kappert, Z.Ang. Phys., Vol.26(1966), 366.

(15) H.Kappert and P.Schmiesing, Phys. Stat. Sol., Vol.4(1971), 737.

(16) M.Sagawa, S.Fujimura, H.Yamamoto, Y.Matsuura, S.Hirosawa and K.Hiraga, in Eighth Int. Workshop on Rare Earth Permanent Magnets, Ed. K.Strnat, University of Dayton, Ohio, 1985, 587.

(17) T.Suzuki, K.Hiraga and M.Sagawa, Jap. Jl. of Appl. Phys., Vol.23(1984), L421.

FIGURE CAPTIONS

Figure 1 : Lorentz image of a foil with the c-axis normal to the foil plane depicting bubble domains as well as stripe domains.

Figure 2 : Lorentz micrograph of a sample with the c-axis normal to the foil plane showing domains at grain boundaries where no fcc phase exists. Note that the domain walls are continuous across the grain boundaries.

Figure 3 : (a) In-focus bright field image of a sample with the c-axis in-plane. (b) Defocussed mode lorentz image corresponding to (a) showing domain walls which are continuous across the grain boundaries(indicated by arrows).

Figure 4 : (a) Bright field image of a region close to the grain boundary depicting the second phase. (b) Lorentz image corresponding to (a) showing domain walls pinned at the second phase.

Figure 5 : Domain wall width as a function of objective lens defocus, both underfocus as well as overfocus.

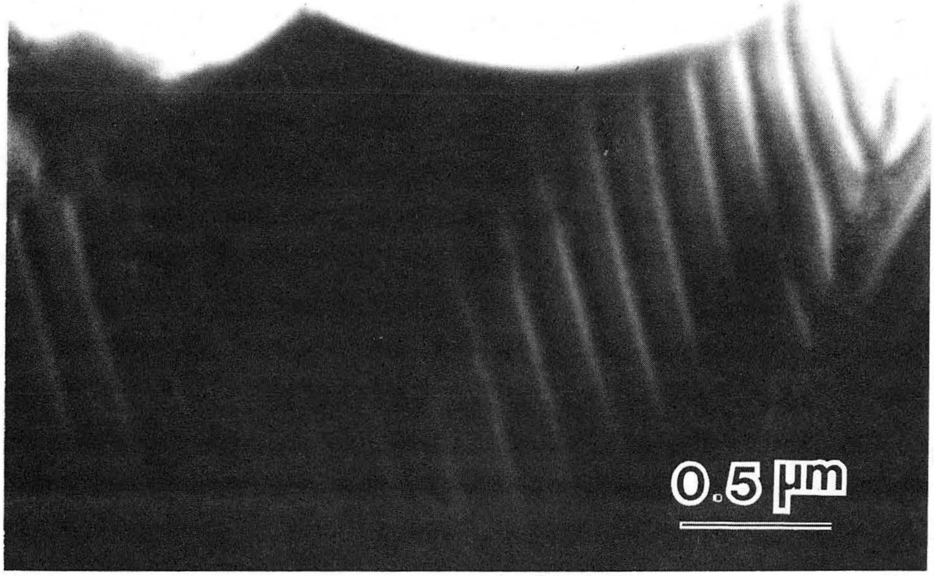

FIG 1 XBB 857-5409

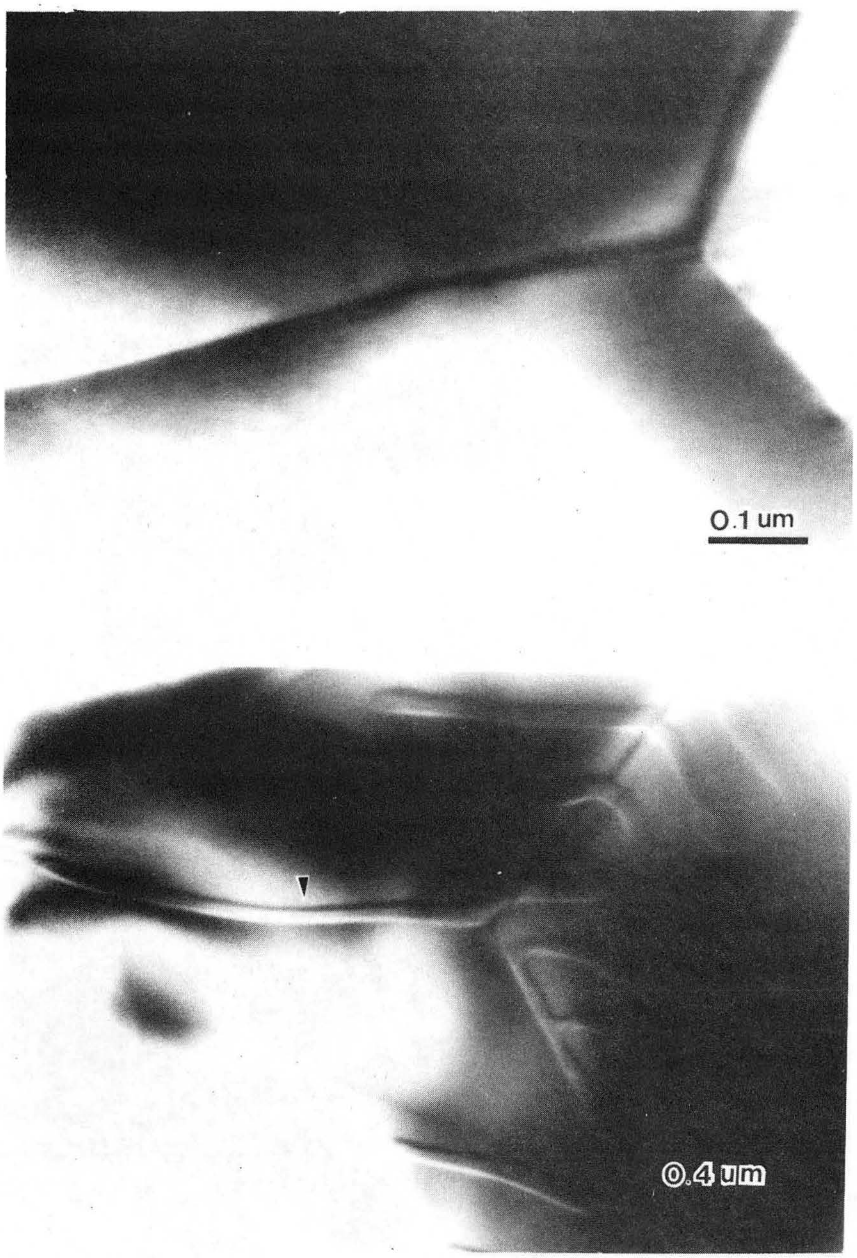

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FIG 2

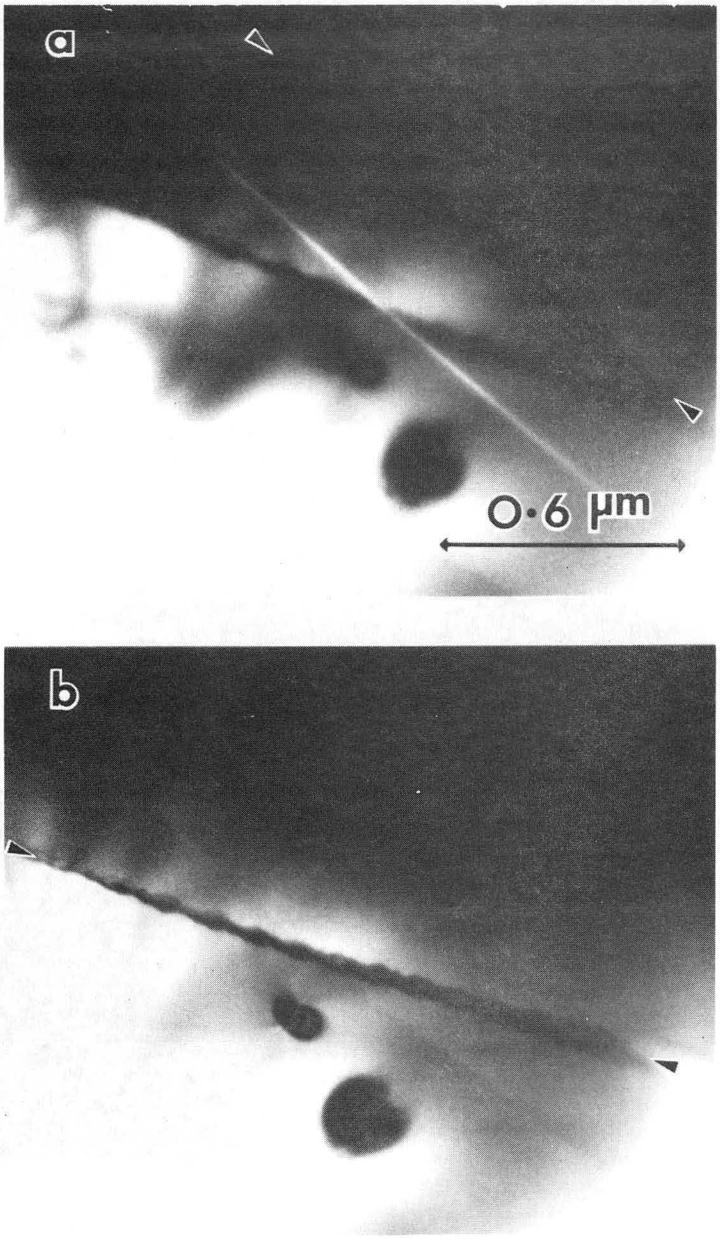

FIG 3

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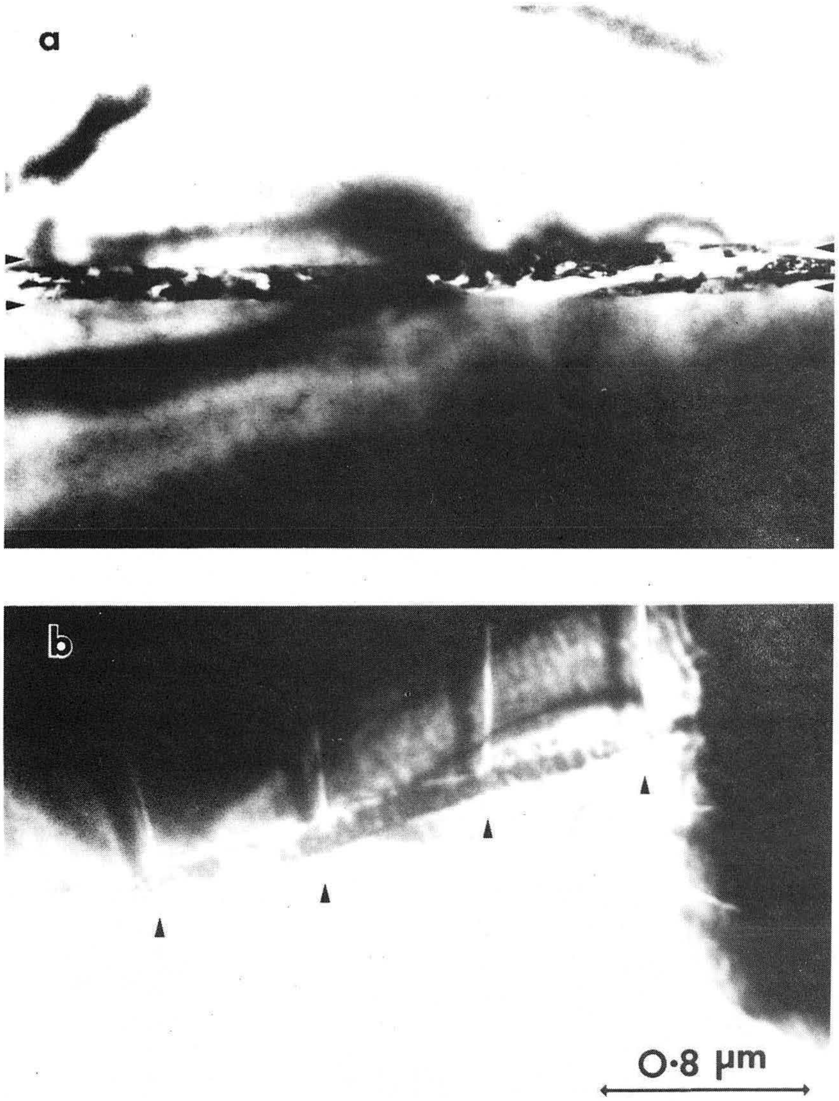

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FIG 4

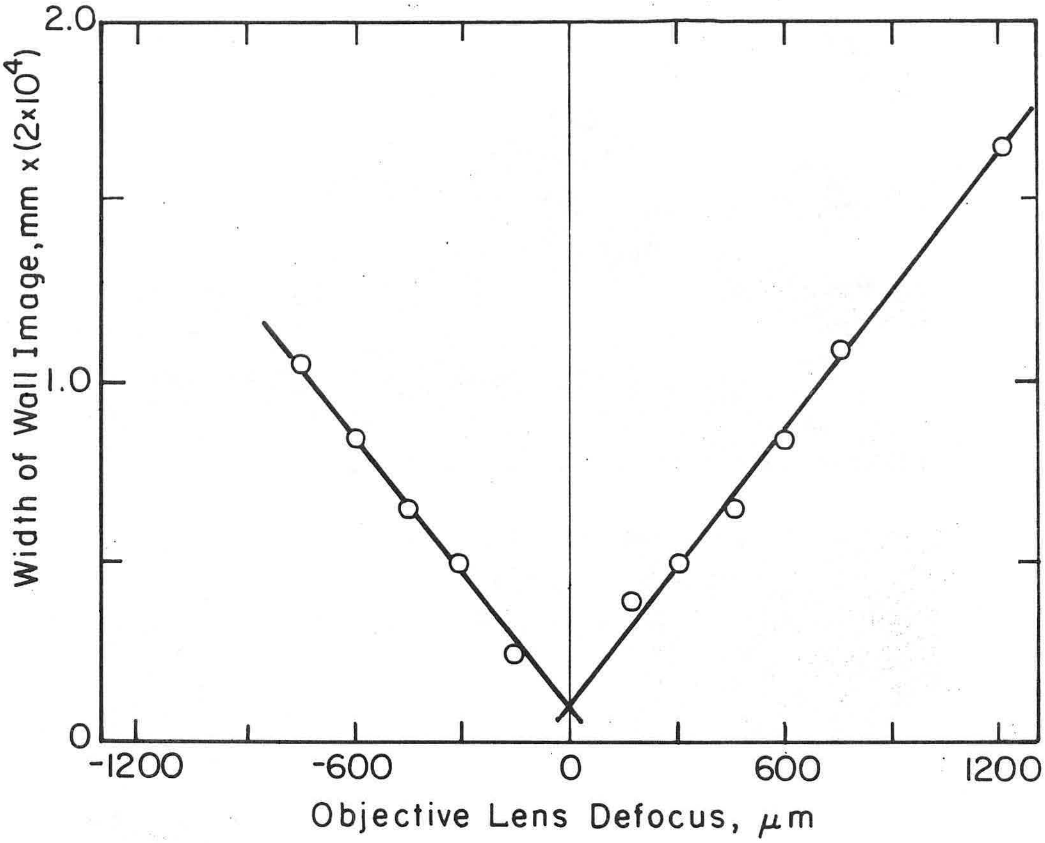

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FIG 5

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