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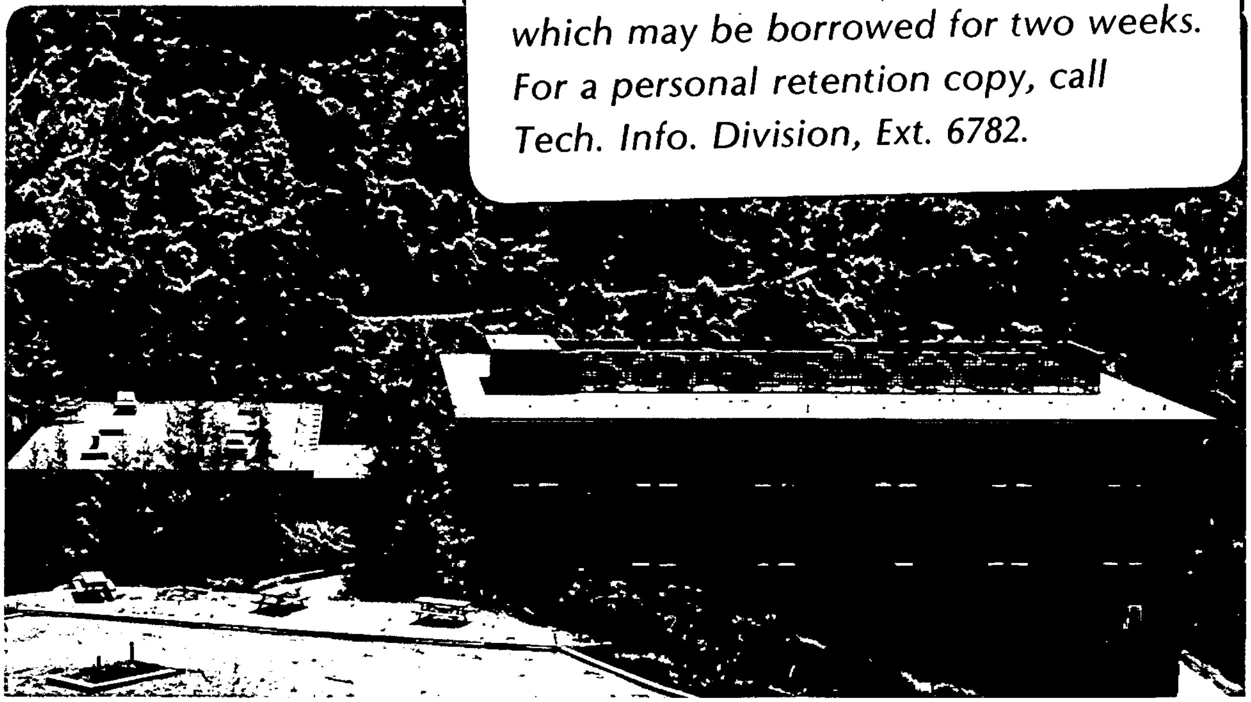

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Interaction of Magnetic Domain Walls with Microstructural Features in Spinel Ferrites

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Abstract

Lorentz microscopy studies of (Mn,Zn)Fe_2O_4 and $LiFe_5O_8$ show that grain boundaries, pores, microcracks, stacking faults and nonmagnetic second phase particles act as pinning sites for moving domain walls. Second phase particles, pores and some grain boundaries also act as favorable sites for reverse domains to be nucleated. In addition, it is observed that domain walls cannot move smoothly as they approach a grain boundary.

I. Introduction

The magnetic properties of ferromagnetic and ferrimagnetic materials depend on their chemical composition, crystal structures and microstructures. The recognition of the effect of microstructure on some of the magnetic properties has led to the development of various processing techniques and thermomagnetic treatments so as to optimise the microstructural variables for the desired combination of properties [1-3]. The actual studies of the effects of grain boundaries, grain sizes, porosity, second phases etc. on coercivity, permeability, maximum energy product and other properties have also received considerable attention [3]. Development of various experimental techniques, such as the Bitter technique and the Kerr effect have made it possible to directly examine the effect of microstructures on the domain configurations [4]. The development of Lorentz microscopy (in transmission electron microscopy) has enabled imaging of magnetic domains and domain walls to be done at higher resolutions and also has made it possible to study the interaction of magnetic domains and microstructure [5]. In the latter technique, if a magnetic field can be applied in situ, then the dynamics of domain wall motion and its interaction with the microstructures can be examined directly. Since domain wall motion is related to many of the magnetic properties, such dynamic studies are of interest in understanding the magnetic behavior of materials. In this paper a study of the interaction of magnetic domain walls with grain boundaries, stacking faults, second phase precipitates, grain boundary segregates, pores and internal microcracks in some spinel ferrites is described. The motion of the domain wall is studied in situ and the possible influence of such motion on the properties is discussed.

II. Experimental

Sintered (Mn,Zn) Fe_2O_4 with and without CaO impurities and flux grown single crystals of $LiFe_5O_8$ were used for the present study. The detailed composition and processing history of (Mn,Zn)Fe_2O_4 can be found elsewhere [6]. The single crystal $LiFe_5O_8$ was heat treated in air at 1200°C for 1 hr. to obtain a two-phase material [7]. Thin electron transparent specimens, suitable for transmission electron microscopy, were prepared from the bulk material by ion thinning [8]. The Lorentz microscopy was done on a Philips EM301 microscope operating at 100kV. Using the four lens system of the EM301, the Lorentz image could be focussed with the diffraction lens.

A magnetic field was applied to the specimen by exciting the objective lens of the microscope with a very low current [9]. The strength and direction of the magnetic field in the plane of the specimen could be controlled by appropriately tilting and rotating the specimen (using a tilt-rotation holder) so that it is at an angle with the magnetic flux. A strong field normal to the specimen surface was thus always present and could not be avoided in this study. The microstructure was examined under normal operating conditions of the microscope.

III. Results and Interpretation

Under the application of a magnetic field, some of the domains will grow and some will shrink as a result of the rotation of the magnetisation vectors in the domains. The motion of the domain walls and their interaction with the microstructure can thus be examined. In the following figures, the direction of the applied field is from top to bottom of the figure (the direction is reversed in figures labelled with a -ve number). A pair of Lorentz images in both underfocussed (u) and overfocussed (o) conditions in each stage of magnetisation are shown in Figs. 1-5 along with a sketch of the domain wall configuration in each case.

1) Effect of grain boundaries and grain boundary segregates

The interaction of a domain wall with a grain boundary depends, among other things, on the angle between the grain boundary and the domain wall. As shown in Fig. 1, when the moving wall is parallel to the grain boundary, the boundary acts as a pinning site for the wall. On the other hand, if the wall makes an angle with the grain boundary, the motion of the wall is retarded, there being least retardation for walls normal to the grain boundary (Fig. 1). When the grain boundary is inclined to the specimen surface (whereas normally, due to magnetostatic effects, the magnetisation lies in the plane of the sample and the domain wall is normal to the surface), the effect is similar. In addition to the pinning effects, the grain boundary region acts as a site for the nucleation of new domains.

In the case of materials containing an intergranular phase [6], as the domain wall approaches the boundary, its motion is retarded and the wall stops at a distance from the boundary. As the applied field is increased, the wall suddenly "jumps" as a result of magnetisation reversal by rotation in the region between the grain boundary and the impeded wall. Domains are also nucleated at these grain boundaries and junctions containing the second phase. All these are shown in Fig.

2. It can also be seen in the figure that if the applied magnetic field is reduced, the domain wall motion is not reversed. For example, domain wall 1 - 1 in Fig. 2c does not retreat; instead, it splits and a new closure domain E is nucleated. The domain wall 4 - 4 in Fig. 3 cannot move at all until a new domain nucleates.

2) Effect of the second phase

The size and nature of the second phase determine the effect they may have on the motion of the domain wall [2]. As can be seen in Fig. 3, the nonmagnetic $LiFeO_2$ precipitate in $LiFe_5O_8$ [7] can act as a barrier to domain wall motion or as a site for nucleation of a reverse domain. It is observed that if the precipitate is small (< 300nm), it acts only as a barrier to the wall motion and if the precipitates are distributed closely, domain walls show very little mobility. For larger precipitates, the semicoherent interfaces act as sites for reverse domains to be nucleated. As to their influence on the properties, the small precipitates will enhance the coercivity while the larger ones will reduce coercivity and may increase permeability [10].

3) Effect of stacking faults

The most common stacking faults found in spinel ferrites are the cation faults [11] and several of these faults can be seen in Fig. 4. The Lorentz images of an area containing these faults show that the magnetic domain walls are pinned to the faults on either side and the faulted area is a thin 180° domain. The domain walls pinned to the fault do not move easily. Also it can be seen in Fig. 6 that when a field is applied, new domains such as (a) and (b) are created at the stacking faults and the domain wall away from the fault sweeps through the unfaulted matrix easily.

4) Effect of pores and cracks

Pores and microcracks are regions where the material has a small enclosed free surface. As shown in Fig. 5a, pores act as pinning sites for domain walls in

$MnZnFe_2O_4$. Similarly, Fig. 5b shows that a crack can stop a moving domain wall. It is observed that, unlike the case of grain boundaries, a domain wall moves continuously towards the crack until it is finally pinned. Since small pores can be found in many ceramic ferrites not only at the grain boundaries, but also inside the grains, the effective distance between domain wall barriers in such ferrites will be reduced [12].

IV. Discussion

The typical microstructure of a ceramic ferrite contains grain boundaries, cracks, pores, etc. It has been recognized that grain boundaries act as barriers to domain wall motion [1]. As the above study shows, the pinning behaviour of different grain boundaries is different depending on whether the boundaries have any segregates or not. In the case of $(Mn,Zn)Fe_2O_4$ sintered with some CaO, it has been shown that the Ca rich intergranular phase strains the spinel lattice in the vicinity of the boundary [6]. The present observation that the domain wall in such materials moves to the boundary by a sudden "jump" in the vicinity of the boundary can be understood by assuming that the stress in the spinel lattice leads to a local change in the anisotropy and the magnetisation vector rotates only when a sufficiently large field is applied. Such a material inevitably will have a lower permeability compared to materials with no intergranular phases or strain centers near the grain boundaries [6].

The formation of reverse domains at pores, second phases and grain boundaries/junctions are due to the demagnetising fields at these inhomogeneities. When the demagnetising field is large enough, a region of the material near the inhomogeneity is magnetized in the reverse direction. Materials with many such sites will typically have a lower coercive force.

Since many ceramic materials, due to their processing schemes, contain small pores, microcracks, etc., and since these features act as barriers to the domain wall motion, they must be taken into account while studying the behavior of magnetisation of the ceramic ferrites. For example, if pores etc. are present inside the grains and not only at the grain boundaries, the interface distance and not the grain size defines the distance over which a wall can travel without any obstacle.

The formation of a non-magnetic second phase can be utilised in some ceramic systems, particularly the hard ceramic magnets, to enhance their coercivity. Since precipitates of varying sizes and distributions can be formed by manipulating the composition and thermal treatments (from knowledge of phase and kinetic diagrams), it should be possible to manipulate the microstructure to control the properties.

Finally, it must be emphasized that the above observations are qualitative. In order to make a quantitative study of domain wall motion vs. applied magnetic field, a special specimen stage with proper field compensation [13] must be designed and then one can measure magnetising field, etc. directly by observing the Lorentz images. Nevertheless, the qualitative observations described in the present paper shed some light onto the interaction of microstructural features with the domain wall. Caution must be used in extrapolating the results of these observations on thin foils to the case of bulk magnets.

Acknowledgements

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Figure Captions

Fig. 1. Interaction of domain wall with an edge-on grain boundary (dotted line g-g) in a $(Mn,Zn)Fe_2O_4$ sample. Domain walls parallel to grain boundary (1-1,2-2) are stopped completely while the domain wall normal to the grain boundary (2-3) can move up and down. (a) is the focussed image of a grain boundary. Lorentz image pairs u_1, o_1, and u_2, o_2 are taken with different applied fields.

Fig. 2. Underfocussed (u) and overfocussed (o) Lorentz images taken from the same area of a $(Mn,Zn)Fe_2O_4$ sample show how the domain walls move and get pinned at the grain boundary containing CaO segregates (dotted line). The domain wall 1-1 is stopped at a distance from the grain boundary in stages 1 and 2 and jumps to the boundary in stage 3 as the field is increased. When the field is reduced (stage 4), a new closure domain E is created. Domain C is nucleated in stage 2 at the grain boundary.

Fig. 3. BF image showing $LiFeO_2$ precipitates (d,e,g) in $LiFe_5O_8$ and the Lorentz micrographs 1 and 2 showing the domain wall pinning by the precipitates. A reverse domain (B) is nucleated as the applied field is increased.

Fig. 4. BF image shows the cation stacking faults in $LiFe_5O_8$. Domain walls (solid lines) are pinned at the stacking faults in stage 1 when they lie parallel to the fault plane, but the wall 1-1, normal to the faults, can move. Stacking faults act as a site for nucleation of reverse domains (a) and (b) in stages 2 and 3.

Fig. 5. (a) domain wall 1-1 moves to the left or right as the field is increased or decreased (stages 2 and 3 in the Lorentz images), but is pinned by pore P; (b) domain wall 1-1 moves continuously towards crack C as the field is increased. Finally walls 1-1 and 2-2 annihilate each other and the material is unformly magnetised across the crack.

INTERACTION OF GRAIN BOUNDARY
IN MnZn-FERRITE

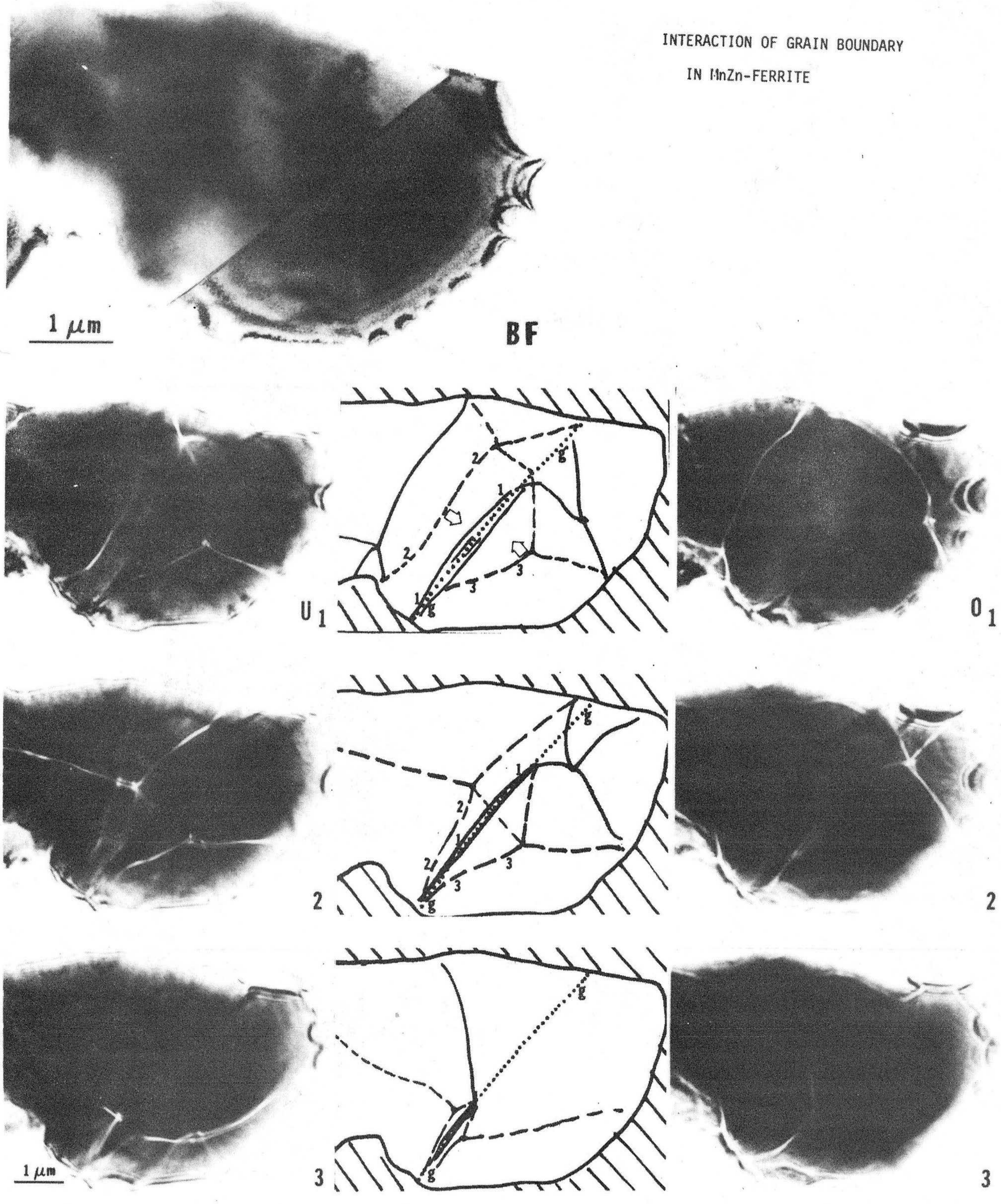

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Fig. 1

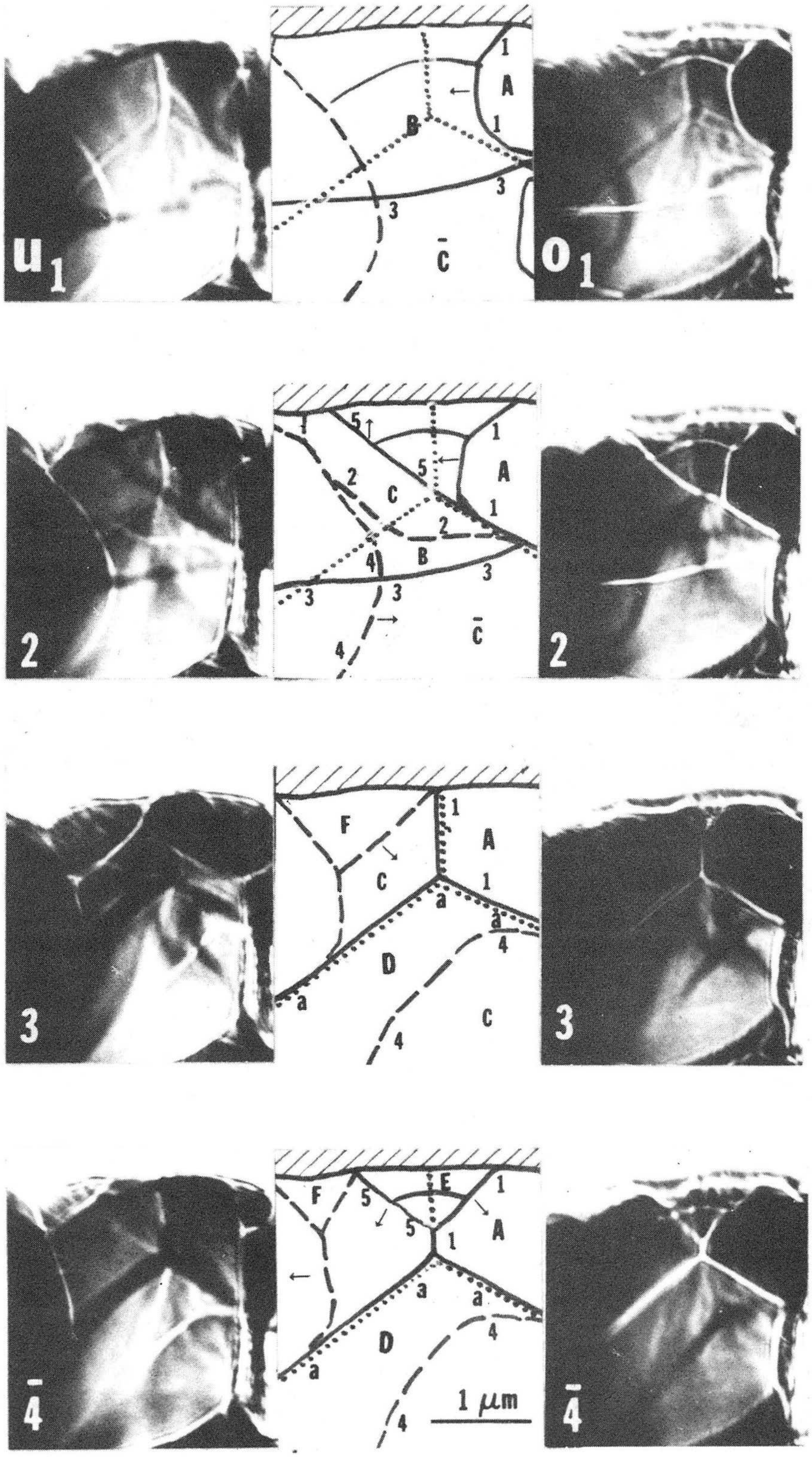

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Fig. 2

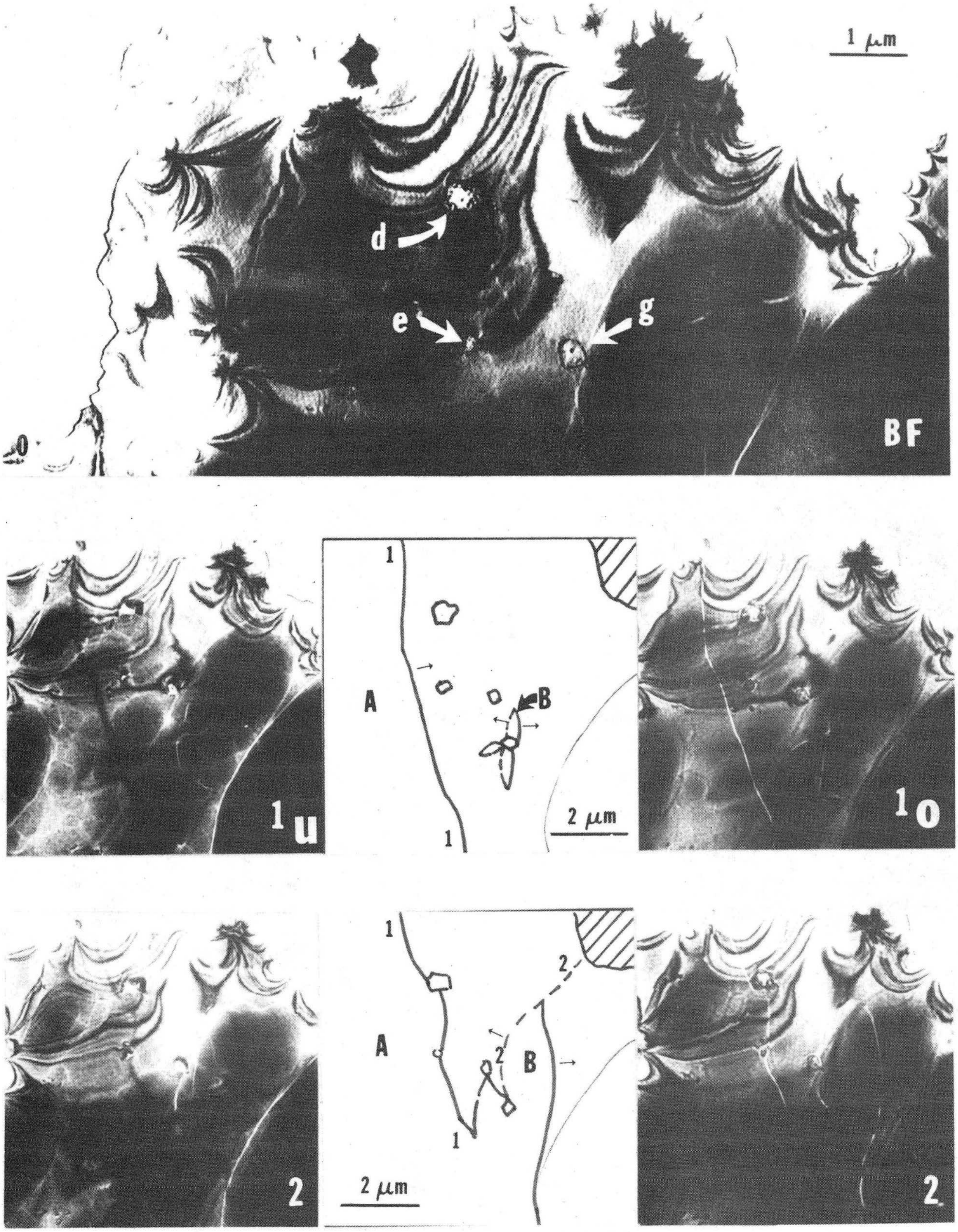

Fig. 3

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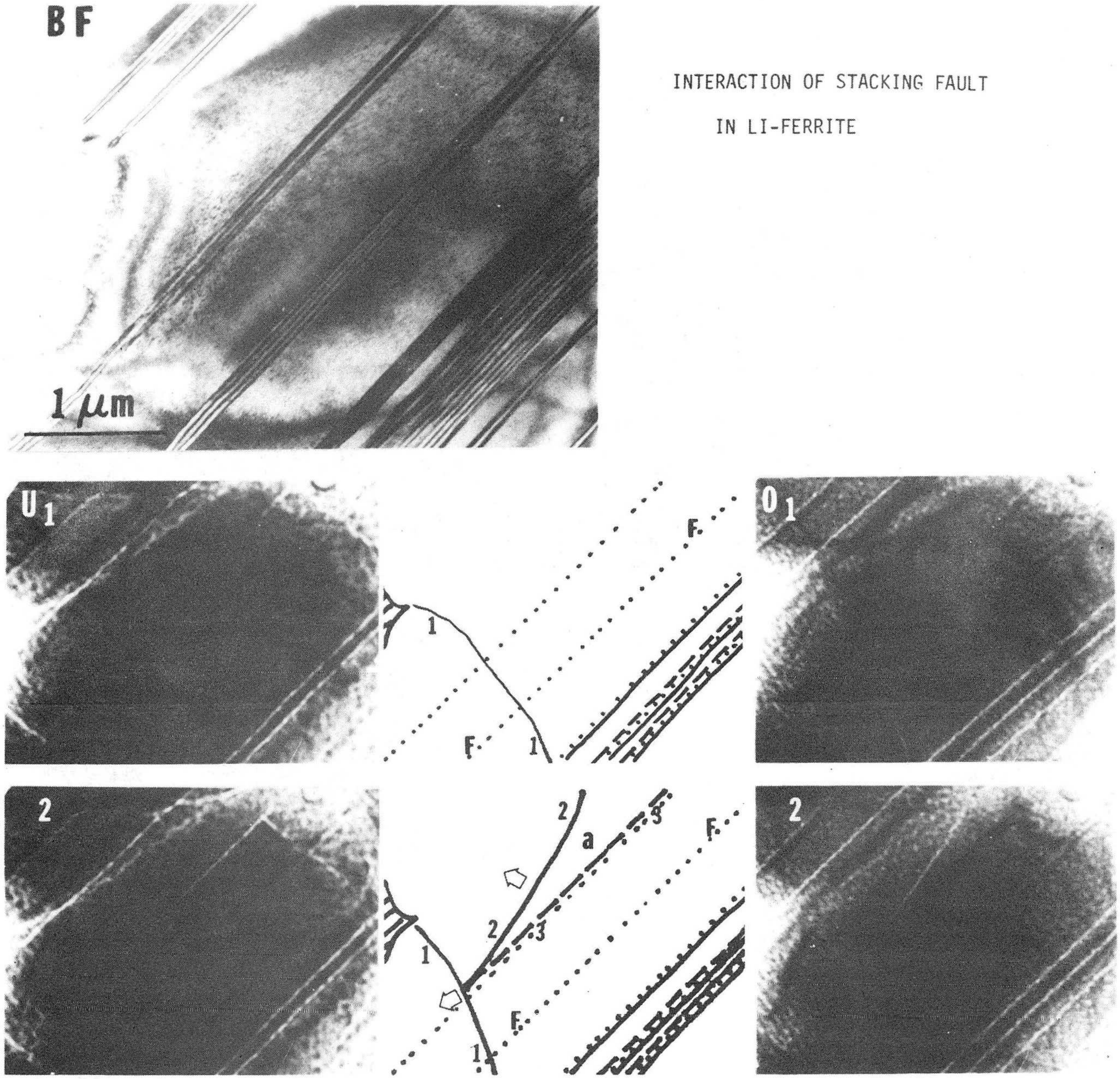

INTERACTION OF STACKING FAULT
IN LI-FERRITE

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Fig. 4

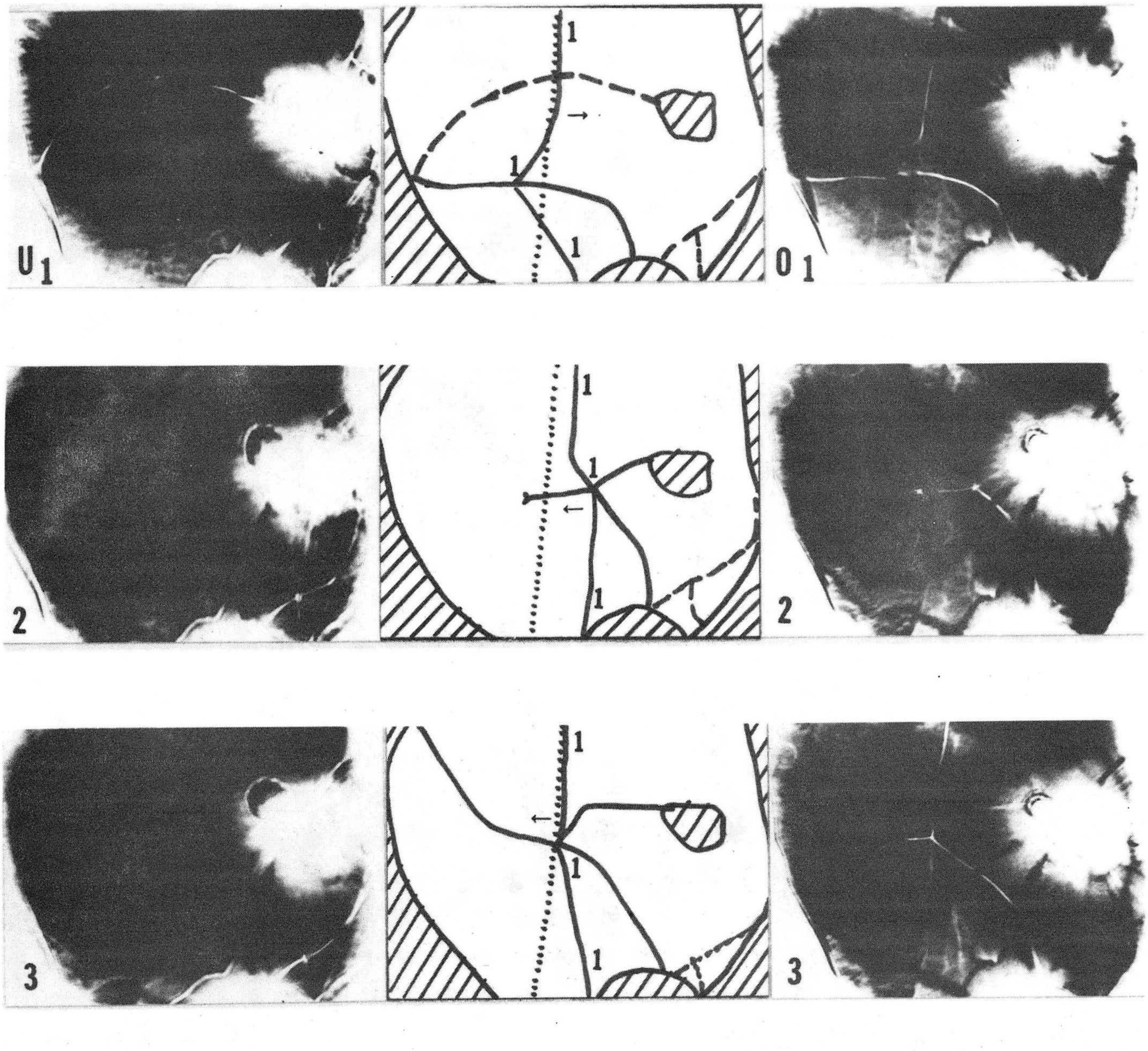

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Fig. 5a

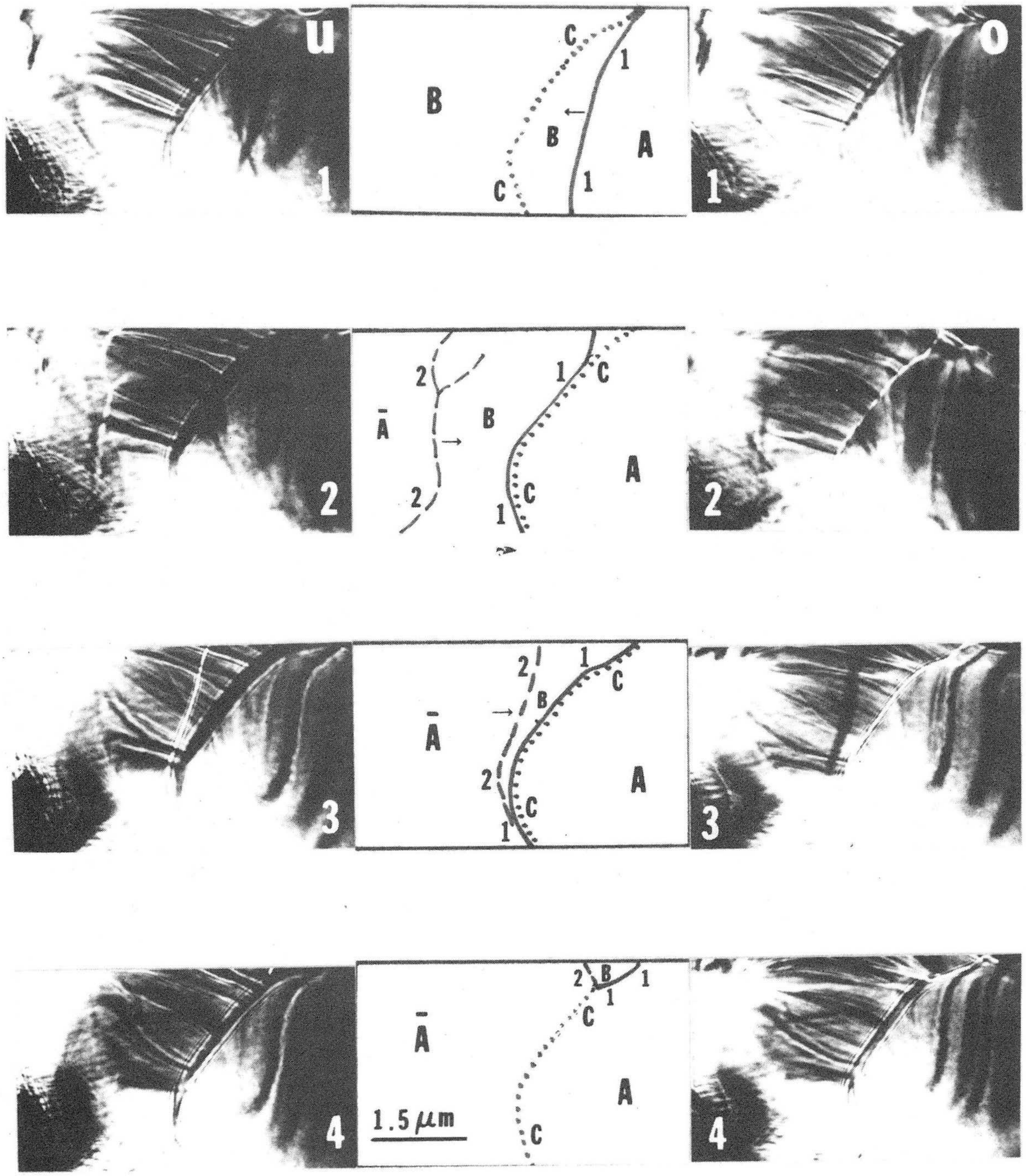

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Fig. 5b

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