Observations of ferrite inclusions in magnesium oxide substrates used in nickel ferrite film growth

In a recent study of the oxidation of $NiFe_2$ and $CoFe_2$ thin films [1] some preliminary results were presented on the growth of thin nickel ferrite films produced by the oxidation of vacuum deposited $NiFe_2$ thin films on single crystal magnesium oxide substrates.

To study the microstructure of the oxidized thin films the magnesium oxide crystals were thinned from the uncoated side. Selected-area electron diffraction patterns from these thinned areas were identified as due to single crystal ferrite and small crystallites were observed in the thinned areas. It is important to note that due to the similarity of the spacings of atomic planes in magnesium oxide and ferrite, all of the magnesium oxide reflections effectively coincide with the ferrite reflections in electron diffraction patterns for the camera lengths used in the electron microscope, in normal use. Only a few diffraction spots such as {220} ferrite can be assigned solely to the ferrite diffraction pattern (Fig. 1a).

Recent more detailed investigations, using dark-field electron microscopy, have established that upon oxidation of thin films of $NiFe_2$ at temperatures of the order of 1200°C on MgO single crystal substrates, nickel and iron ions diffuse into the MgO substrate. These ions come out of solution to form ferrite precipitates when the temperature of the substrate is lowered to room temperature. The precipitates are coherent and probably have the composition, magnesium nickel ferrite. The exact proportions of mag-

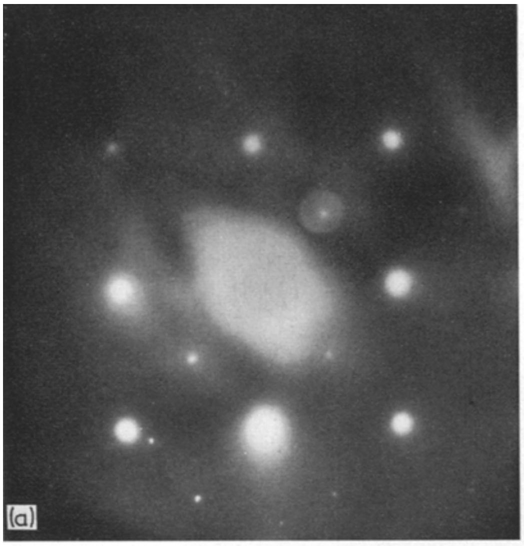
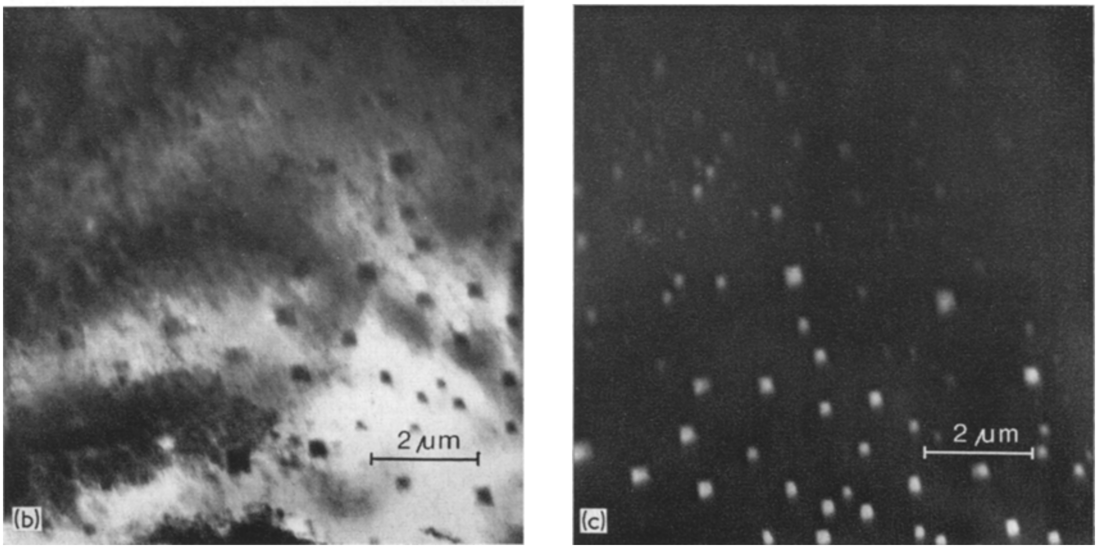


Figure 1 (a) Selected-area electron diffraction pattern from a $NiFe_2$ film deposited on single crystal magnesium oxide and oxidized at 1200°C. The ferrite (220) diffraction spot used in dark field (Fig. 1c) is delineated by the objective aperture. The electron beam is parallel to ⟨001⟩. (b) Bright-field electron micrograph showing ferrite precipitates in the MgO matrix. (c) Dark-field electron micrograph of the area in (b) showing strong contrast from the ferrite precipitates. The reflection operating is the ferrite (220) reflection.



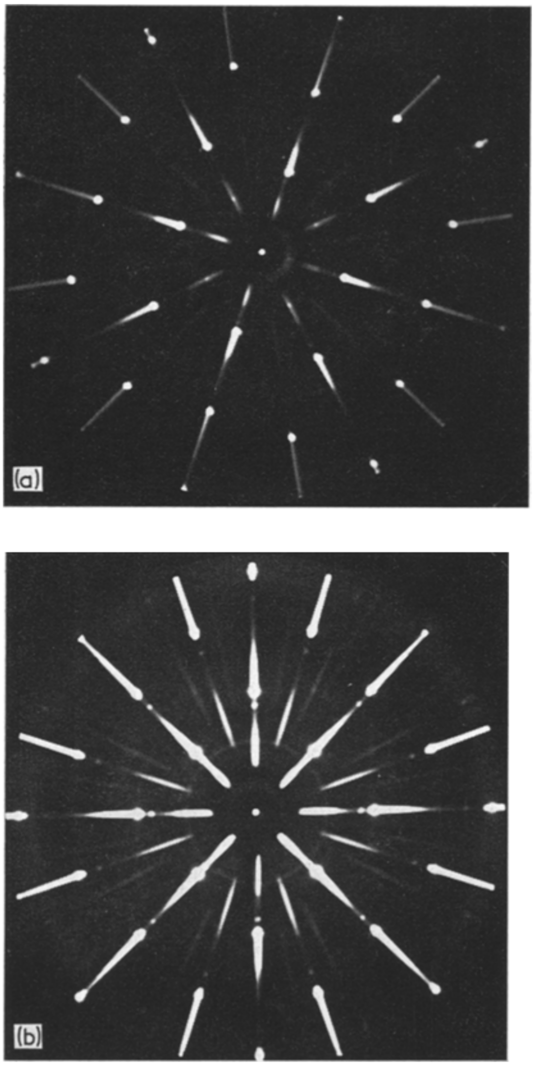


Figure 2 (a) Zero level precession X-ray diffraction pattern from a thin sputtered nickel ferrite film annealed at 1200°C, (001) orientation. (b) Zero level precession X-ray diffraction pattern from a thick sputtered nickel ferrite film annealed at 1150°C, (001) orientation.

nesium, nickel and iron are as yet undetermined. Similar effects have also been observed in a previous investigation of oxidized iron films on MgO substrates [2] where magnesioferrite precipitates were formed.

Fig. 1b is a bright-field electron micrograph showing the ferrite precipitates in the thinned region. Fig. 1c is a dark-field electron micrograph using a reflection which belongs to the ferrite structure only. Only the precipitates exhibit contrast in this condition verifying the conclusions given above, that oxidized thin $NiFe_2$ films on MgO substrates give rise to coherent ferrite precipitates in the magnesium oxide substrate.

Similar precipitates have also been observed after high temperature anneals of thin films sputtered onto magnesium oxide substrates from a nickel ferrite target.

Precession X-ray diffraction patterns (Fig. 2a) from oxidized $NiFe_2$ thin films and sputtered nickel ferrite thin films annealed on MgO substrates give rise to single crystal ferrite diffraction patterns. It is clear that neither X-ray diffraction or electron diffraction observations are capable of resolving the structural composition of specimens prepared by these two techniques. This is due to the coincidence of the majority of ferrite and MgO diffraction spots in the situation where an epitaxial ferrite film is formed, or in the situation resolved here, where coherent ferrite precipitates have only been identified by resort to dark-field electron microscopy.

These observations have led to a study of the structure resulting from the deposition of thicker films. It is found for example, for sputtered films of thickness greater than approximately 3 μm, that polycrystalline films eventually form (Fig. 2b). Single crystal ferrite spots, however, are still observed in the precession X-ray diffraction patterns along with polycrystalline ferrite rings. Diffusion therefore takes place in the substrate-film interface and ferrite inclusions form in the MgO substrate.

It can be concluded from these observations that ferrite inclusions are formed in the MgO substrate close to the MgO-film interface when nickel ferrite films are produced by oxidation of $NiFe_2$ films or by sputtering from a nickel ferrite target.

A more detailed investigation of the composition of the ferrite inclusions will be published elsewhere.

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The mode of deformation of oriented polypropylene in compression at low temperatures

When an oriented polymer is stretched or compressed a deformation band is sometimes obtained, its formation being dependent on the temperature, rate of testing and the direction of the tensile or compressive axis. The deformation bands have been observed in various oriented polymers: nylon rods [1], polyethylene sheets [2-5], polyethylene terephthalate [6] and polypropylene [7].

In a previous note [8], we showed, quantitatively, the effects of the orientation on the yield behaviour of oriented polypropylene and the results of tests at room temperature suggest clearly that the modes of deformation in tension and compression are different. However, in this communication a new mode of deformation of oriented polypropylene is proposed based on observations of deformation bands formed at yield in compression tests at low temperatures.

All tests were conducted using a conventional Instron machine provided with a cooling chamber. Compression tests were made using a compression cage to produce a compressive load on the specimen with the machine in its tensile mode. Tests were done at $-50°$C, (achieved by blowing cold carbon dioxide gas around the specimen) at a strain-rate of 1.67×10^{-3} sec^{-1}. The compression specimens were machined from an extruded rod of deformation ratio 5.5:1 and birefringence of order $\sim 2.4 \times 10^{-2}$ to produce blocks of dimensions $6 \times 5 \times 10$ mm^3. Specimens were cut at different angles, θ, to the orientation direction.

At $-50°$C deformation bands were observed at all orientations. The geometry of the bands may throw some light on the stress system which causes the deformation. Fig. 1 shows photographs of deformation bands formed at different angles to the orientation direction in oriented polypropylene. The level of strain at which the band formed varied in magnitude with respect to θ. At $\theta = 90°$, the overall strain as the band formed was about 6%, while at $\theta = 0°$ or 45°, the strain was at least 20 to 25%. The approximately constant value of the band angle (angle between the band direction and the compressive direction) of $45° \pm 5°$ implies that deformation occurs on planes of maximum shear stress which make 45° to the compressive axis. The geometry of deformation bands, as shown in Figs. 1 and 2, varies in a complex manner according to the value of the angle θ. At small values of θ (approximately $0° < \theta < 20°$) the maximum shear strain occurs on planes whose normals lie in the compressive axis/orientation direction plane similar to the deformation bands formed in oriented polymer sheets. The shear stress on these planes causes slip of one side of the specimen relative to other, making the fibrils kink, i.e. a kinky band appears. This shear stress is nearly parallel to a diagonal direction of the face containing the compressive axis and the orientation direction, and we will call it for brevity the "diagonal" shear stress.

When $\theta = 90°$ the deformation process is clearly defined, on planes of maximum shear stress again making an angle of about 45° with the compressive axis but containing the orientation direction (or molecular direction). The band locates on a specimen face which is perpendicular to the orientation direction. The shear stress in this case is normal to the orientation direction and we call it the transverse shear stress. The mode of deformation can be understood by a lateral slip of fibrils upon each other in a shear plane under the effect of the transverse shear stress.

Thus the geometry of deformation band can clearly throw some light on the deformation mechanisms taking place over a range of θ from 0° to 90°. At $\theta = 0°$ the diagonal shear stress process is operative, at $\theta = 90°$ the transverse shear process is operative. The mode of deformation at intermediate angles, $0° < \theta < 90°$ is