Photoelasticity of Fe_3O_4 Film isolated from Iron Single Crystal

By Shigeto YAMAGUCHI

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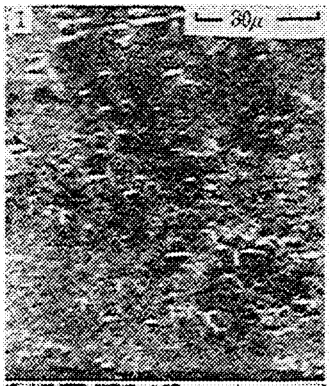
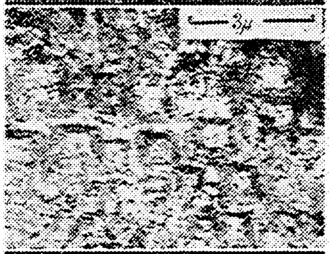
The test piece of iron single crystal was etched with an ethanol-bromine (10: 1 by volume) solution. The Fe_3O_4 film (thickness: about 200 A.) formed on the etched surface was isolated from the underlying substrate.[1] The isolated oxide film was placed upon a glass plate and was examined by means of polarizing microscopy. When the specimen was placed between a polarizer and an analyzer set for extinction, some light passed through it and interference effect was observed, which is shown in Fig. 1. Fig. 1 indicates that an isotropic substance Fe_3O_4 (cubic structure, spinel type) is doubly refracting, i.e., under unsymmetrical strain.

(1) S. Yamaguchi, *J. Appl. Phys.*, **22**, 680 (1951); S. Yamaguchi, T. Nakayama and T. Katsurai, *J. Electrochem. Soc.*, **95**, 21 (1949).





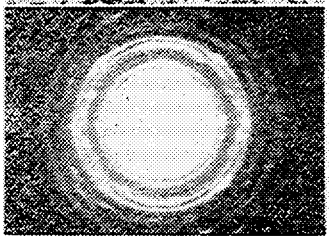


Fig. 1.—The micrograph obtained from the Fe_3O_4 film placed between a polarlizer and an analyzer set for extinction. The oxide film contains the doubly refracting domains, since the film retains unsymmetrical strain forced on it while performing a lattice fit with the underlying substrate.
Fig. 2.—Electron micrograph of the oxide film prepared in the same way as for Fig. 1.
Fig. 3.—Electron diffraction pattern obtained from the Fe_3O_4 film of Fig. 2. This pattern shows that the oxide crystallites are oriented relative to the substrate facets.

The oxide film prepared in the same way as for Fig. 1 was magnified by means of electron microscopy (vid. Fig. 2). In Fig. 2 are found the crystal forms reprinted from the etched surface by oxide replica.[2] The figures characteristic of Fig. 2 are similar to those of Fig. 1.

The oxide film of Fig. 2 gave an electron diffraction pattern of Fig. 3, which verifies the presence of regularly oriented crystallites of Fe_3O_4. This regular orientation of Fe_3O_4 crystallites suggests the lattice fit between the Fe_3O_4 crystal and the substrate crystal of iron.[2] The $Fe^{\cdot\cdot}$-$Fe^{\cdot\cdot}$ distance found in the normal Fe_3O_4 crystal is 8.37 A. (lattice constant of Fe_3O_4), and the doubled value of the distance (4.045 A.) between the nearest two atoms in the (111) planes of iron is $4.045 \times 2 = 8.09$ A. The difference between these two distances is so small (3.5 %), i. e., the strain in the oxide film caused by the underlying substrate is so slight, that the orientation and strain within the oxide film are retained even after the film is divorced from its substrate. As a matter of fact, this isolated film exhibits an anisotropic property, as is shown in the micrograph of Fig. 1, although it is difficult to determine exactly the lattice deformation of Fe_3O_4 film by measuring the diffraction pattern.[3]

The oxide film formed on a mechanically polished surface of iron and isolated from it showed no anisotropic property, distinguishable from that formed on the etched surface of single iron crystal. This oxide film gave a diffraction pattern showing the random orientation of Fe_3O_4 crystallites.

It was here demonstrated, that when a thin film of oxide of cubic structure isolated from a metal surface is doubly refracting, it is composed of oxide crystallites oriented regularly relative to the substrate facets. This photoelastic orientation is given to the oxide film by a slight shift in the lattice fit between the oxide crystal and its substrate.

Scientific Research Institute. Ltd.,
Bunkyo-ku, Tokyo

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(2) S. Yamaguchi, *J. Appl. Phys.*, **22**, 1295 (1951); **23**, 445 (1952); This Bulletin, **24**, 237 (1951); *Naturwiss.*, **39**, 19 (1952); cf. *J. Appl. Phys.*, **23**, 935 (1952).
(3) cf. E. K. Halteman, *J. Appl. Phys.*, **23**, 150 (1952).