Magnetic parameters of superparamagnetic inclusions of hematite in α-alumina

Recent investigations of the $\alpha Al_2O_3-Fe_2O_3$ system [1–3] suggest the presence of superparamagnetic inclusions of αFe_2O_3 at low concentrations of Fe_2O_3. Since the Mössbauer and EPR studies were made using polycrystalline specimens, the magnetic parameters g and K of the inclusions could not be determined.

In an effort to determine the nature and size of the precipitates from their magnetic parameters, iron-doped alumina single crystals have been investigated employing ferrimagnetic resonance techniques. The crystals were doped by diffusion at 1773 K. Measurements were made in a standard X-band spectrometer at a frequency of 9.25 GHz. The resonance field, H_r, was measured as a function of the angle between the external magnetic field and the c-axis of the alumina matrix.

The resonance conditions for ferrimagnetic samples possessing magnetocrystalline anisotropy are well known (see for example [4]). If the magnetocrystalline anisotropy is taken to have axial symmetry and the shape anisotropy of the inclusions is small, then the resonance condition, provided $\omega/\gamma \gg K_1/M, K_2/M$, is:

$$H_r = \omega/\gamma - (K_1/M)(3\cos^2\theta - 1) - (2K_2/M)(\sin^2 2\theta - \sin^4\theta) \qquad (1)$$

where θ is the angle between the applied field and the axis of axial symmetry.

Typical results at room temperature are shown in Fig. 1 along with the theoretical curve calculated from Equation 1. The data of Fig. 1 and similar data obtained from other specimens are unambiguous evidence for the axial magnetocrystalline anisotropy of the iron-rich precipitate and the coincidence of its lattice with that of the aluminium-rich matrix. The room-temperature constants calculated from the orientation dependence of the resonance field are $K_1/M = -39.18$ G and $K_2/M = 6.54$ G. The splitting factor, g, is found to be 2.010 ± 0.004. The solid curve in Fig. 1 is drawn for these parameters.

The anisotropy field of bulk αFe_2O_3 at room temperature has been measured by Anderson [5] who obtained $H_A = K_1/M = -15\,000$ G, a value almost 400 times larger than the one obtained in the present experiment. This can be explained by assuming that the inclusions are very small; in that case, thermal fluctuations of the direction of magnetization can drastically decrease the measured anisotropy field [6]. This, in fact, provides us with a means for estimating the particle size. It has been shown [7] that, in the case of axial symmetry, the measured anisotropy of a coherent assembly of small magnetic particles is given by:

$$H_A^{SP} = H_A(1 - 3x^{-1}\coth x + 3x^{-2})/(\coth x - x^{-1}) \qquad (2)$$

where H_A is the bulk anisotropy field, $x = I_s VH/kT$, I_s is the intrinsic magnetization of the particles, V is the particle volume and H is the applied field.

In the limit $x \ll 1$, Equation 2 reduces to

$$H_A^{SP} = xH_A/5 \qquad (3)$$

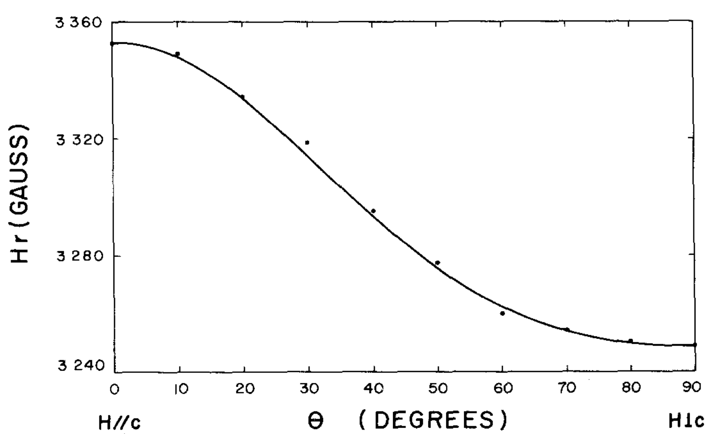

Figure 1 Dependence of the resonance field H_r on the angle θ between the applied field and the crystal c-axis. The solid curve is calculated for $K_1/M = -39.18$ G, $K_2/M = 6.54$ G and $g = 2.010$.

and the particle volume is given by

$$V = (5kT/I_sH)(H_A/H_A^{SP}) \qquad (4)$$

where $H_A = -15\,000$ G and H_A^{SP} is the experimental value, -39.18 G. We have also $T = 300$ K, $I_s = 3.1$ erg cm^{-3} G^{-1} [8], and $H = 3300$ G. Substituting these values into Equation 4, we get $V = 2.56 \times 10^{-20}$ cm^3 or $a = V^{1/3} = 37.5$ Å. This is somewhat smaller than the size estimated by Kalyamin *et al.* [2] using the Mössbauer effect (50 to 100 Å).

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Phase decomposition in liquid-quenched eutectic Au–Ge alloy

Liquid-quenched (LQ) Au–Ge alloys have been observed to contain a number of metastable phases which depend on quenching condition and rate, as well as alloy content [1–4]. Scott [4] recently investigated the formation and stability of the metastable phases in LQ eutectic Au–27% Ge (all compositions are given in atomic percent) by X-ray diffraction and thermal analysis techniques. He found that LQ from the temperature range 500 to 1300° C yields a three-phase microstructure: Au-rich f c c phase, α; metastable h c p-phase, β; and a second metastable phase, γ, with a b c t structure. Samples quenched at a slower rate from 500° C contained only the α and the γ phases. Furthermore, on the basis of the DTA results during isochronal annealing, Scott [4] concluded that the LQ eutectic Au–Ge decomposes in two single stages: (i) decomposition of the β-phase into equilibrium α and Ge in the temperature range 70 to 100° C; (ii) decomposition of the b c t-γ into α and Ge at about 125° C.

X-ray diffraction was used mainly for identifying the metastable phases in Scott's study. Because of the coexistence of a number of constituent phases with widely different volume fractions, and in view of the fact that the mass absorption coefficient of Au is very high as compared to that of Ge, any Ge-rich phase which may be present as a minor constituent can easily go undetected.

In the present work the Au–27% Ge eutectic alloy was prepared by inert-gas induction melting of appropriate amounts of high-purity materials. The homogeneity of the alloy was ensured by repeated melting and quenching into water. Small samples of this alloy were then LQ from 800° C using the gun-technique, yielding typical cooling rates in excess of 10^6° C sec^{-1}. Samples from the bulk of the gun-quenched foils (~60 to 70 μm thick) were used for isochronal and isothermal resistivity measurements. The resistivity measurements were made employing a standard four-probe potentiometric technique. Ageing of the resistivity samples was carried out in appropriate baths controlled to ±0.5° C. The resistivity measurements were made in acetone at 21° C. X-ray diffraction measurements on the central bulk samples were carried out on a diffractrometer. A Debye–Scherrer camera was used for obtaining powder patterns from the LQ thin edge flakes. It was ensured that the bulk samples and the flakes used in X-ray analysis contained the same phases in the as-LQ condition. Small flakes from the edges of the gun-quenched samples were also used for TEM. Ageing of the TEM specimens was done *in situ* in the microscope hot stage.