The Curie Point of Hematite Crystals

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The Curie points of a large number of Hematite crystals from various localities were measured using the electron beam technique. The Curie points were found to be between 640°C and 688°C, and the depression in Curie point could be correlated to some extent with the titanium content as determined by spectrochemical analysis. The fact that a large number of crystals with high titanium content (and from different localities) all had a Curie point of about 640°C suggests that these are associated with the maximum amount of titanium which can be accommodated in the hematite lattice at room temperature, and this maximum value would be $x=0.05$, where the constitution of the crystal is written as $x\,FeTiO_3\,(1-x)\,Fe_2O_3$.

The effect on a 40 kV electron beam of the magnetic field due to ferromagnetic domains have been used to study the behaviour of hematite at elevated temperatures. For this purpose a furnace was constructed which could heat the hematite crystals to above 700°C in the electron diffraction camera; shadow patterns of the crystal were taken as the crystals were heated to above the Curie temperature or cooled from this temperature (see Fig. 1).

In these experiments it was found useful to study the variation with temperature of the sizes (denoted by W) of prominent features in the observed shadow patterns. The results, some of which are indicated in Fig. 2, indicate that the features are almost constant with temperature up to about 600°C.

From 600°C onwards there is a rapid decrease in the size, and by using such curves one can obtain a fair measurement of the Curie point, defining this as the limiting temperature at which the size of the features become negligibly small. This will occur at a higher temperature than would be obtained from the point of inflexion of a magnetisation curve. For Elba hematite, the latter temperature would be 675°C, whereas the magnetic shadow method gives 688°C, with an accuracy of two or three degrees.

A number of hematite crystals from different localities were examined; two crystals were always mounted side by side in the apparatus, one being from Elba and used as a standard. In this way the difference in Curie points (due to impurities such as titanium) could be established reason-

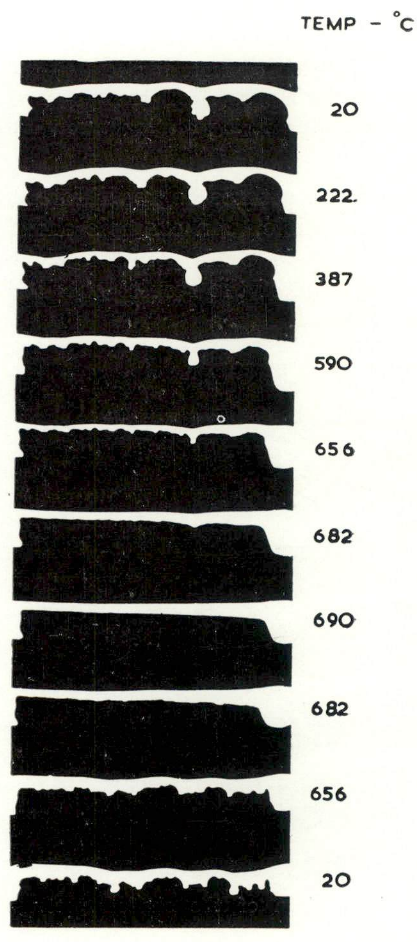

Fig. 1. The effect of temperature on the shadow pattern of the surface of a single crystal of hematite, origin Elba. The length of the crystal in the direction perpendiculur to the electron beam is 7 mm.

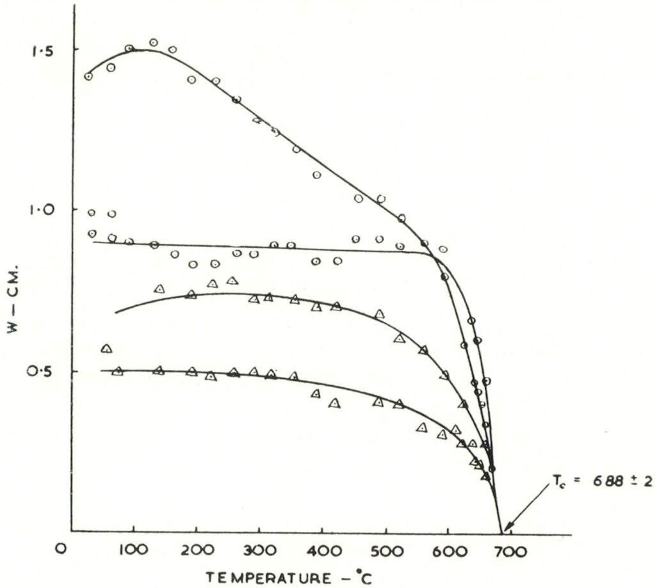

Fig. 2. Variation of feature size (W) with temperature for two prominent features. The lower two curves are obtained during the cooling of the crystal from above the Curie point, the upper curves during heating through the Curie point.

ably accurately, a three degree difference being just perceptible. An example showing a large difference in Curie points is shown in Fig. 3, and some of the results obtained are given in Table 1.

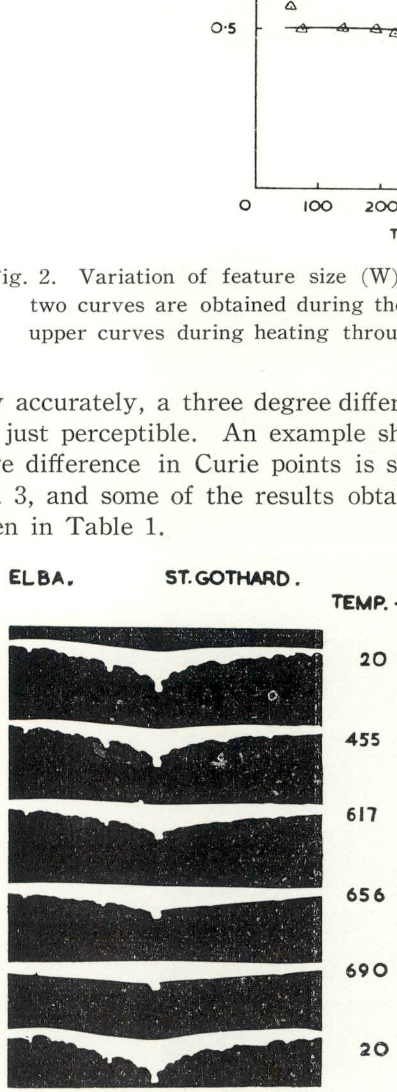

Fig. 3. Comparison of the shadow patterns of two natural single crystals of hematite having widely differing Curie points, one (origin Elba) having a Curie point of 688°C, the other (origin St. Gothard) having a Curie point of 643°C. The length of each of the crystals in the direction perpendicular to the electron beam is 4 mm.

All the specimens were analyzed spectrochemically and the titanium content so obtained is also shown.

Measurements of the lattice parameters of the above crystals were also carried out, and these showed the general change in lattice parameters which would be expected from the titanium content.

The lowest Curie point found was 643°C, for a number of crystals from different localities and deductions from the spectrochemical analyses and the X-ray data suggest that this is consistent with 1.5% titanium, at the most, being incorporated in the lattice. It seems reasonable to conclude that this amount of titanium is the maximum which can be accommodated in the hematite lattice at room temperature. In this case the solidus

Table I. Curie Temperatures of various Hematite Crystals.

Specimen No.	Origin	Major Impurity	Curie Temp. °C
E-H(H)1	Elba	None	688±2
S-H(E)	Unknown	0.3% Ti	673±3
S-H(G-B)C1	Unknown	1% Ti	661±3
S-H(S-G)A1	St. Gothard	2.5% Ti	656±3
S-H(S-G)B1	St. Gothard	3.0% Ti	643±3

curve should pass through an x value of 0.05 (where a typical member of the hematite-ilmenite series can be written as x $FeTiO_3$, $(1-x)$ Fe_2O_3. This would imply a slight modification of the solidus curve determine by Uyeda.

DISCUSSION

S. OGAWA: Is there no any temperature difference between the surface and the thermocouple position?

M. BLACKMAN: While I would not wish to insist on the accuracy of our Curie point determination, the specimen was very well surrounded by the furnace and independent tests showed that the thermocouple readings were reasonably accurate. However, the real interest lies in the differential Curie point measurements and for these the accuracy of the thermocouple measurements is not really important.

JOURNAL OF THE PHYSICAL SOCIETY OF JAPAN VOL. 17, SUPPLEMENT B-II, 1962
PROCEEDINGS OF INTERNATIONAL CONFERENCE ON MAGNETISM AND CRYSTALLOGRAPHY, 1961, VOL. II

An Electron Microscope Capable of Simultaneous Recording of an Electron Microscopic Image and an Electron Diffraction Pattern

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A new device of the three-stage electron microscope capable of simultaneous recording of an electron microscopic image and an electron diffraction pattern is explained and an example of transformation due to heating of an evaporated film of Al 50 wt.%/Cu 50 wt.% alloy is shown.

It is well-known that we can obtain, by means of a three-stage electron microscope, an electron microscopic image and an electron diffraction pattern from a thin film specimen by varying the strength of the intermediate lens. Recently, an electron microscope with a cine-camera has been developed and the continuous recording of a transformation which occurs in a crystalline specimen by heating, cooling, or tension, etc., has become possible. But, in such an apparatus, it is difficult to observe an image and a diffraction pattern simultaneously, and either an image or a diffraction pattern is recorded alternately. On the way of the transformation it is quite impossible to know to which crystallographic stage the image under observation corresponds. The important advantage of the three-stage electron microscope is partly lost in such an apparatus. In order to cover this defect, we have designed an electron microscope permitting the simultaneous recording of an image and a diffraction pattern.

Fig. 1 is a block diagram showing the cinematographic apparatus 4 attached to a three-stage electron microscope. The strength of the intermediate lens is varied respectively by means of the two regulators 22 and 23,