Physical Separation in Science and Engineering, 2003,
Vol. 12, No. 3, pp. 133–143

THE EFFECT OF ROASTING TEMPERATURE ON ILMENITE PARTICLE ROTATION CHARACTERISTICS IN A ROTATING MAGNETIC FIELD

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(Received 24 March 2003; Accepted 25 April 2003)

Individual ilmenite particle measurements of magnetic susceptibility and magnetic rotation index show that, as ilmenite roast temperatures increase, both the magnetic anisotropy and the coercive force increase. By around 650°C the particles are behaving magnetically, in magnetic fields up to 0.3 T as though they contain magnetic elements with a single unidirectional magnetic axis.The presence of some magnetic field, even that of the earth, during the cooling of ilmenite samples roasted above 500°C may be essential to the magnetising roast process.

Keywords: Magnetising roast; Ilmenite; Magnetic separation; Rotation index

INTRODUCTION

The particle measurements presented here were made on individual ilmenite particles from a series of fluid bed oxidising roasts at roast temperatures between 350 and 650°C [1]. Samples from these roasts had already been subjected to a series of lift and rotation magnetic separations, and particles for measurement were chosen from the magnetic fractions produced by these separations The measurement of magnetic susceptibility and magnetic rotation index on individual particles, and the concepts behind the interpretation of the measurement results, have been described by Allen [2]. The particles are placed individually in small water-filled glass tubes, which are then placed horizontally in the magnetic field produced by a rotating magnet rotor. The field and gradient at which the particles commence rotation, and the field and gradient at which they are lifted are measured, and are used to calculate an approximate magnetic susceptibility and ferromagnetic rotation index. The rotation index relates the actual magnetic torque placed on a particle to the maximum possible torque that could be placed on a particle of that total magnetic moment. The rotation index is based on the sine of the maximum achievable angle between

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ISSN 1478-6478 print: ISSN 1478-6486 online
DOI: 10.1080/1478647031000154234

particle magnetisation and external field. Although the theoretical maximum value for any rotation index is therefore 1, uncorrected factors such as particle shape can produce values greater than unity. An average shape correction factor has been applied to the measurements here.

EXPERIMENTAL METHODS

Fifty ilmenite particles from each roasted sample (4–6 particles from each magnetic lift fraction) were measured for approximate magnetic susceptibility and magnetic rotation index, using the method illustrated in Fig. 1.

For each particle, measurements were carried out at five field rotation frequencies (10, 20, 30, 40 and 50 Hz). For purposes of plotting in the graphs below, the five measurements were averaged for each particle.

MEASUREMENT RESULTS

The measurement results are plotted as scattergrams in Figs. 2–7. Logarithmic scales are used on both axes. An average 'shape factor' of four has been applied across the rotation index measurements (i.e. the raw rotation indices have been divided by four) as an approximate average correction for the departure of the particle shape from the basic cubic shape assumed by uncorrected rotation index measurements. Particles with rounded edges rotate easier than a cube, and the rotation index will therefore measure high. The application of a 'shape factor' across all the particles does not change their relative distribution in the graphs, but moves the whole pattern downwards.

While measurements of the distance for commencement of rotation vary, and are expected to vary, across the frequency range, measurements of lift distance rarely show variation that is outside of the scale measurement error (± 0.25 mm), and then only for the more magnetic particles where sideways rotation (precession effects) can make the lift point less accurate to judge.

The roasting experiments, on which these rotation measurements are based, also examined the effects of cooling a roasted sample in a static magnetic field of approximately 0.15 T. Particle measurements on these 'magnetically treated' samples showed no convincing difference from the traces in Figs. 3–7, except perhaps for the 650°C roast, where the group with low rotation indices (in Fig. 7) around a

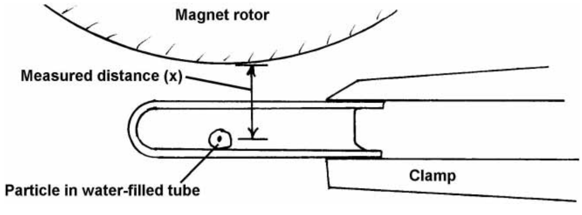

FIGURE 1 The arrangement used for the measurement of particle lift and rotation distances and the calculation of mass magnetic susceptibility and rotation index.

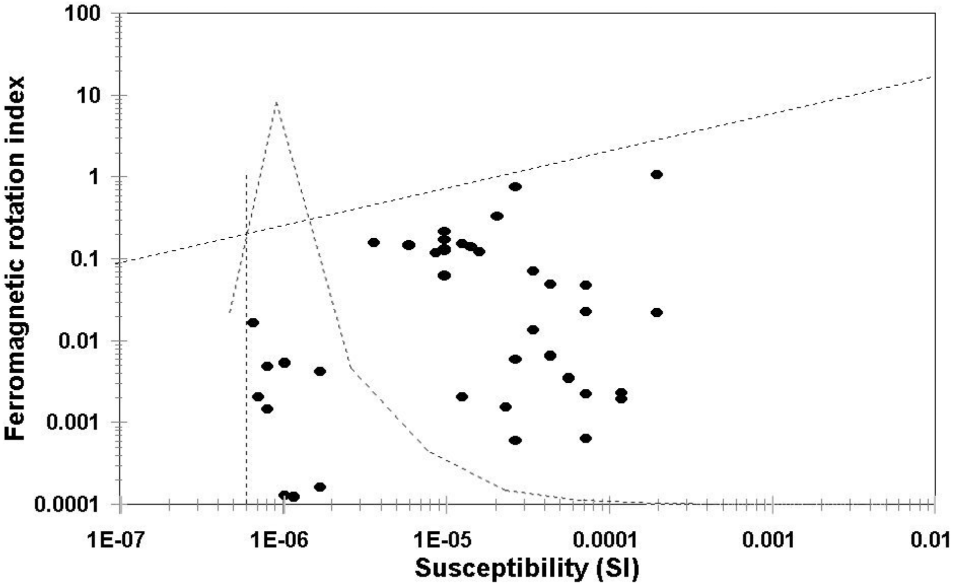

FIGURE 2 Measurements on an unroasted sample. Note: the dotted diagonal straight line represents the upper theoretical limit for ferromagnetic components in a paramagnetic matrix (see also Fig. 11), and the dotted vertical line marks the lower susceptibility limit below which the particle should be completely paramagnetic. The irregular line represents the magnetic susceptibility frequency distribution for the sample (from previous lift separations, [1]).

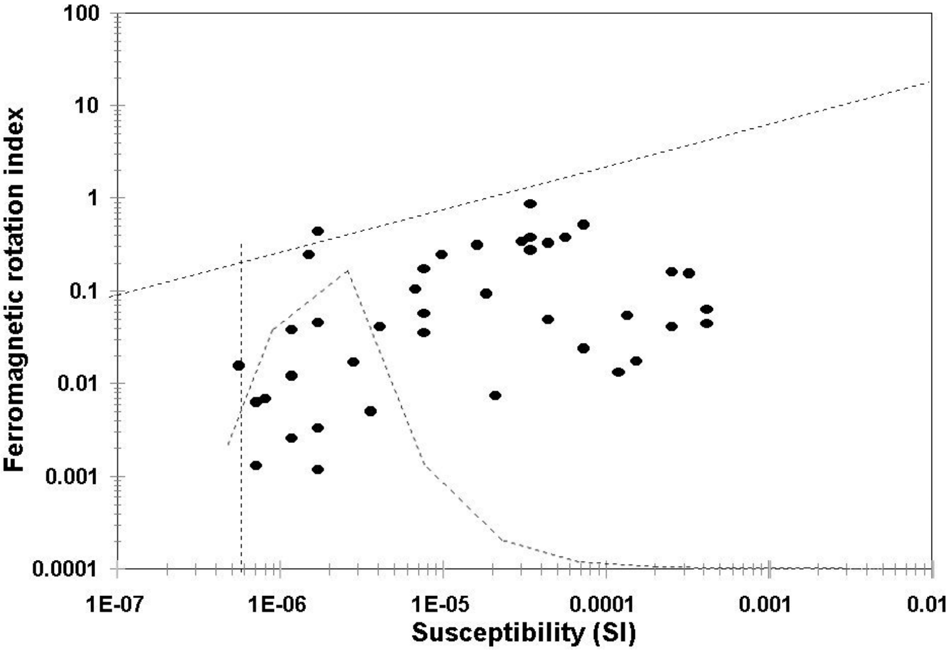

FIGURE 3 Measurements on the 450°C roasted sample.

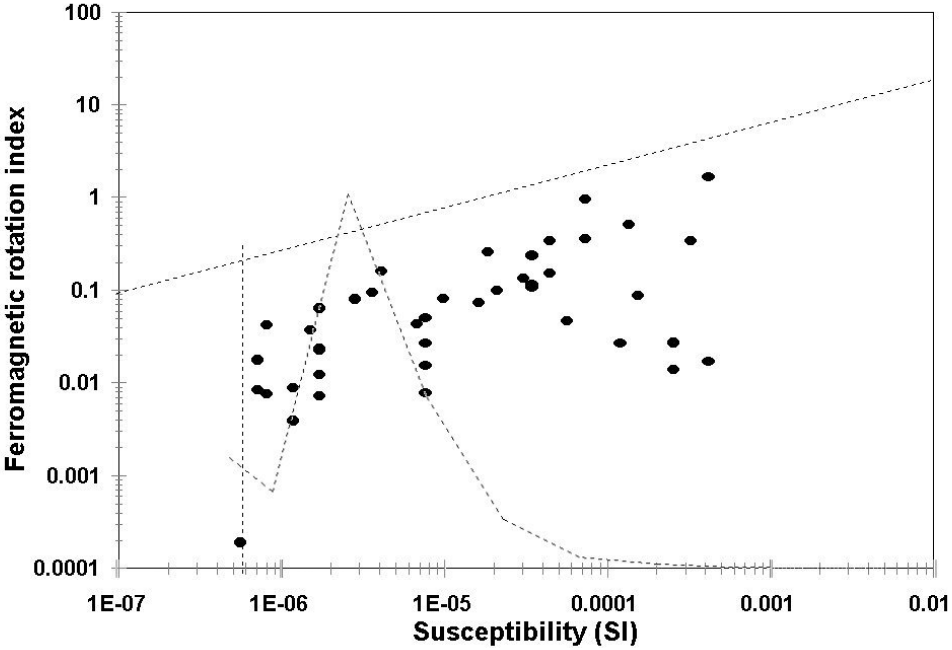

FIGURE 4 Measurements on the 500°C roasted sample.

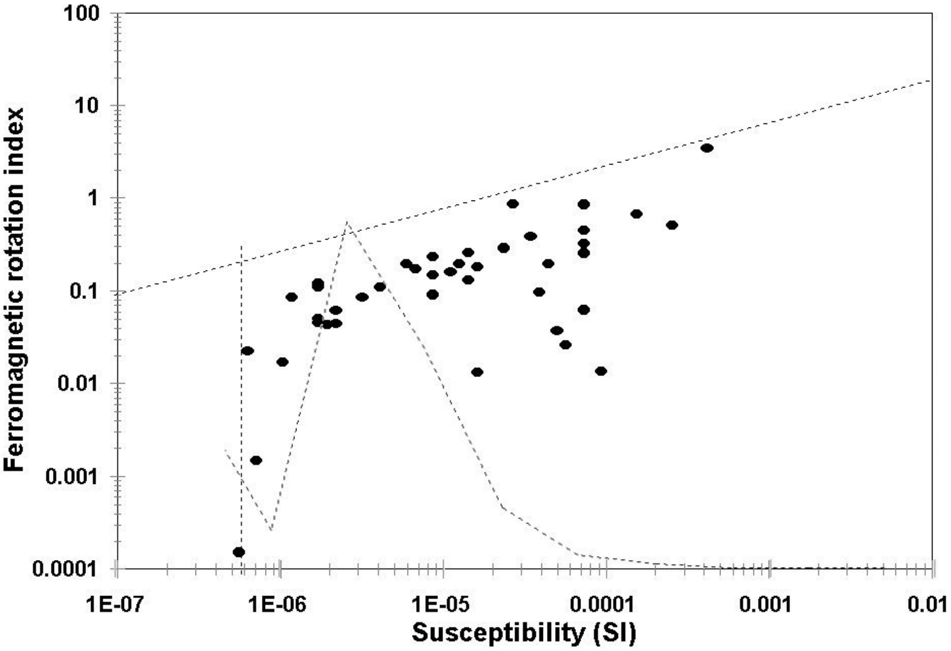

FIGURE 5 Measurements on the 550°C roasted sample.

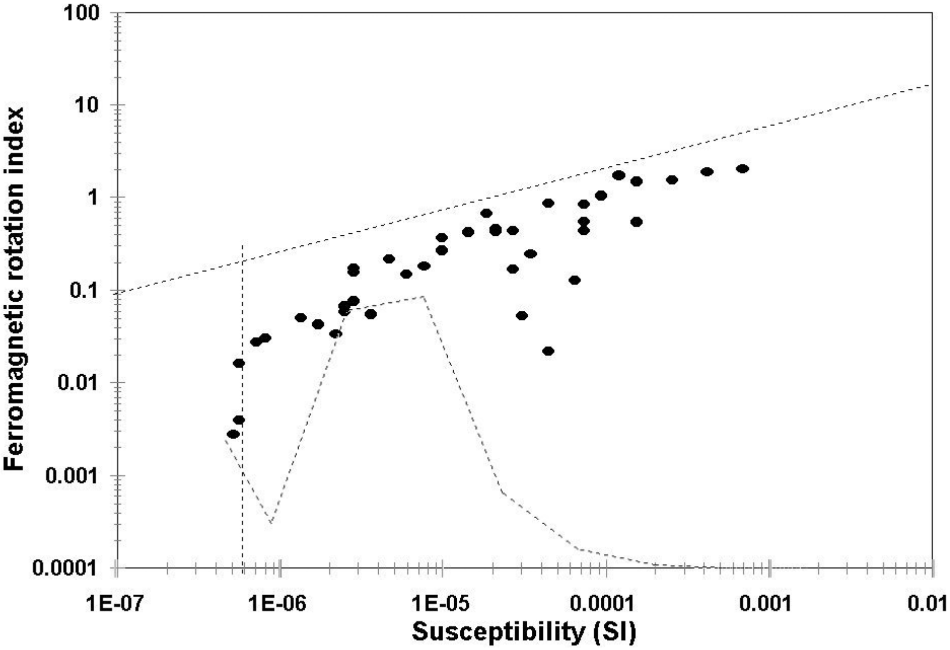

FIGURE 6 Measurements on the 600°C roasted sample.

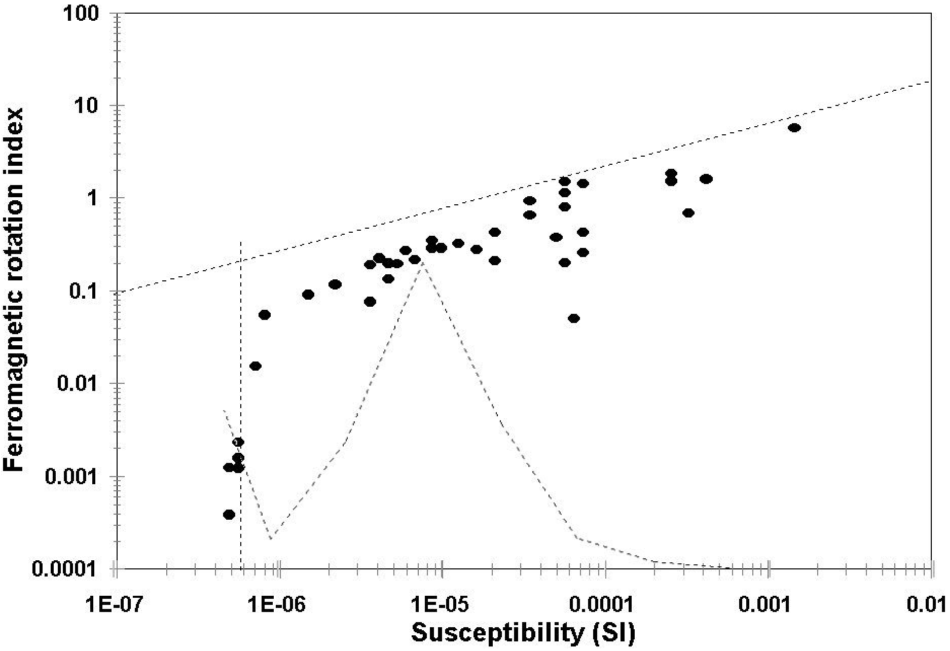

FIGURE 7 Measurements on the 650°C roasted sample.

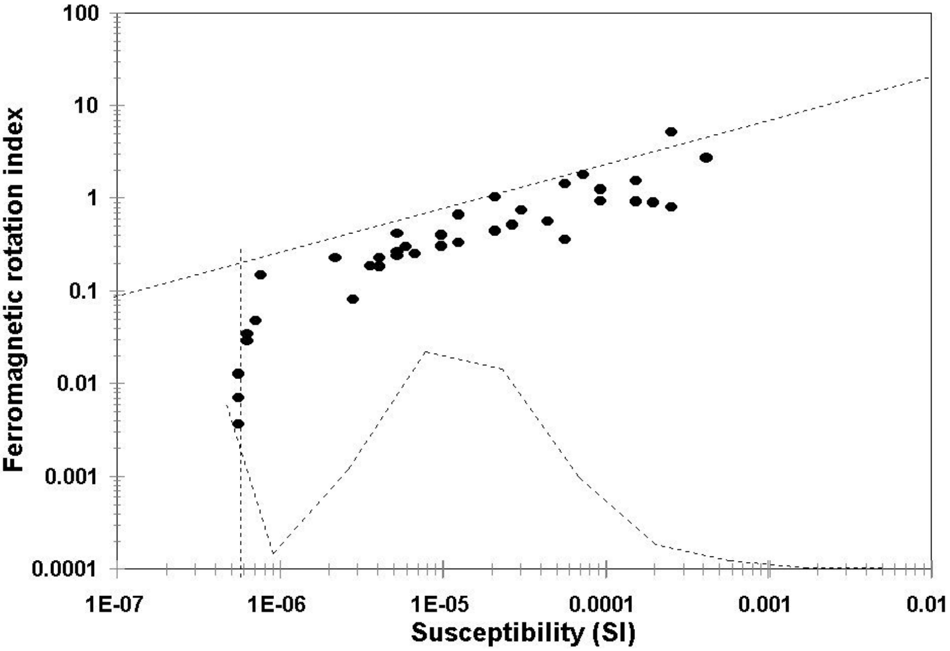

FIGURE 8 Measurements on the 650°C roasted sample that was cooled in a magnetic field of approximately 0.15 T.

mass susceptibility of $6 \times 10^{-5}\,\mathrm{m^3/kg}$ is absent. Keeping in mind that, for the 650°C roast, the total number of particles in this susceptibility fraction has been more than tripled by the magnetic treatment, the apparent absence of this low susceptibility group could be simply because none of these particles were chosen for measurement. The measurement results for the 650°C 'magnetically treated' sample are plotted in fig. 8.

Rotation indices are expected to decrease as the field rotation frequency increases, due to the effects of inertia. Other factors can modify this frequency response, such as slow domain wall velocities. However, in the absence of any overall change in the structure of the ferromagnetic components within the particles, the average frequency response for each sample should stay the same across the roast temperatures. If it does not, then there has been a change in the properties of the magnetic elements within the particles. This could include changes to anisotropy, coercive force or magnetic axes. The volume or number of the ferromagnetic components within the particles has little effect on the rotation indices, as these are in proportion to the magnetic moment of the particle. Figure 9 shows the average sample frequency response across the roast temperature range.

COMMENTS ON THE RESULTS

The rotation index distribution (fig. 2) for the unroasted ilmenites is very dispersed, with particles spread widely right across the total range so far known for all ilmenites. The range of values so far known for ilmenites is shown in fig. 10.

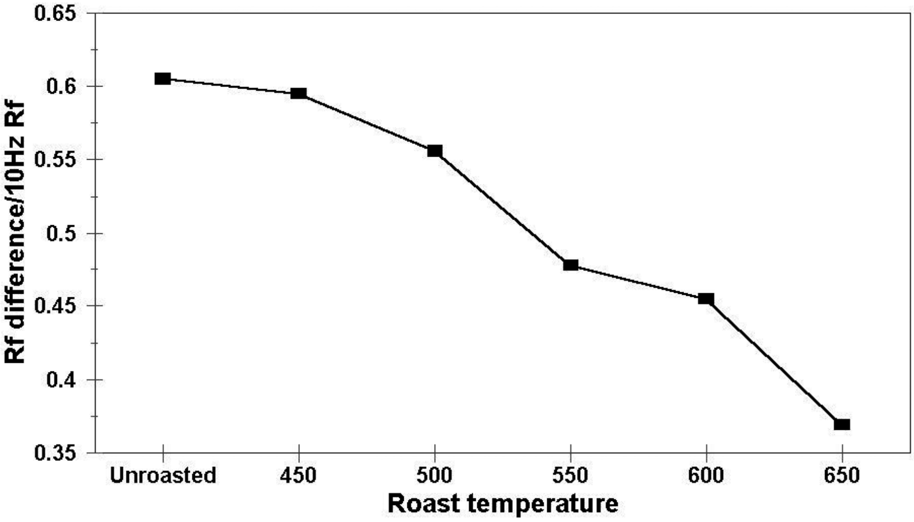

FIGURE 9 Average sample rotation index frequency response for different roast temperatures. This is the difference between the 10 and 50 Hz rotation indices divided by the 10 Hz rotation index.

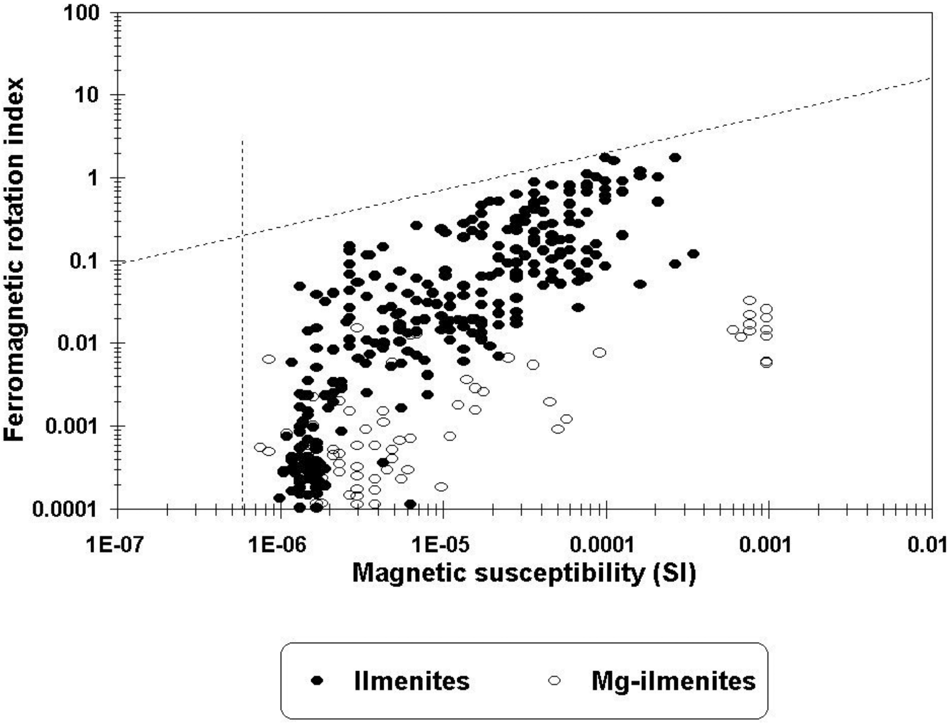

FIGURE 10 Rotation index distribution for approximately 700 ilmenite particles from sites across Australia. The Mg-ilmenites contain >2% MgO, and generally have low rotation indices but high susceptibilities.

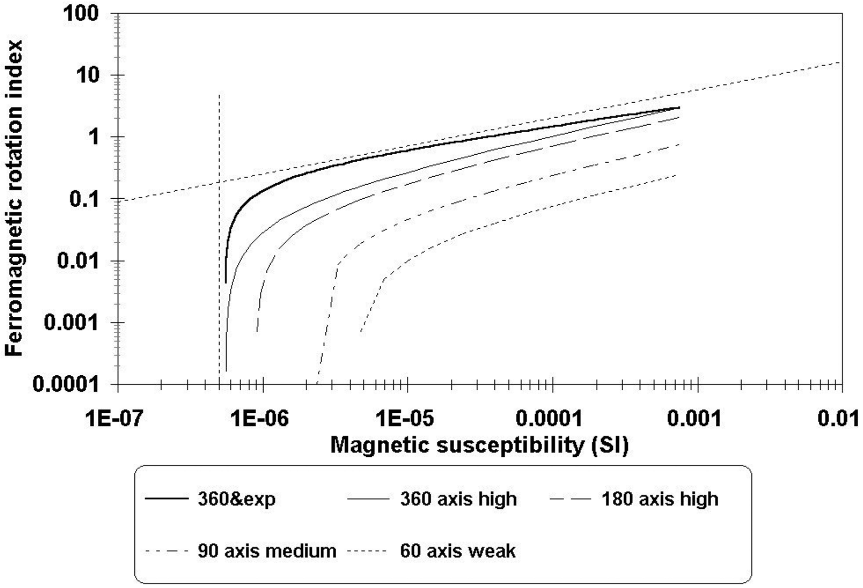

FIGURE 11 Expected rotation index traces for ilmenite particles containing ferromagnetic components in a paramagnetic matrix.

The effect of increasing roast temperature (figs. 3–7) shows up very clearly as a progressive move towards higher and more restricted rotation indices, until at 650°C the trace, except for a small group of particles around a susceptibility of $6 \times 10^{-5}\,\mathrm{m^3/kg}$, is approaching a single line trace. At 650°C, the narrow width of the trace is very surprising indeed, given that the measurement error for rotation indices was expected at around a factor of 2.

The height of the trace indicates a combination of the anisotropy force and the number and relationship between axes of easy magnetisation. The slope of the trace (down to the left) indicates ferromagnetic components or regions within a paramagnetic matrix, with the paramagnetic matrix mass susceptibility given by the point at which the trace cuts the susceptibility axis (in this case around 5 or $6 \times 10^{-7}\,\mathrm{m^3/kg}$). This situation can be modelled for a number of easy magnetisation axes and for some varying anisotropy forces. Figure 11 shows the rotation index traces that might be expected for a number of situations.

Figure 11 assumes the presence of ferromagnetic components within a surrounding paramagnetic structure. The ferromagnetic component decreases progressively from total on the right of the graph to zero at the cut-off susceptibility shown by the vertical dotted line.

- The lowest trace (‘60 axis weak’) represents what should be measured for particles containing ferromagnetic components with three axes of easy magnetisation in the plane of particle rotation, with relatively weak magnetocrystalline anisotropy (angle between field and particle magnetisation about equal to angle between particle magnetisation and direction of easy magnetisation).
- The ‘90 axis medium’ trace represents the case of two easy magnetisation axes at right angles in the plane of rotation, with anisotropy forces such that the angle

between field and particle magnetisation is about three times the angle between particle magnetisation and direction of easy magnetisation.

- The ‘180 axis high’ trace represents a single bi-directional axis of easy magnetisation, with high anisotropy forces (angle between particle magnetisation and direction of easy magnetisation is very small compared to the angle between particle magnetisation and external field).
- The ‘360 axis high’ trace represents the situation of a single unidirectional magnetic axis, with high anisotropy forces. For this situation the measuring field must be less than the coercive force, and the magnetic structures are then behaving as though they are small permanent magnets.
- The ‘360&exp’ trace is based on the same situation as above, but on an initially faster increase in the bulk ferromagnetic component with increasing susceptibility, rather than on a linear proportional relationship. This implies that the ferromagnetic components at lower overall particle susceptibilities are lower in specific magnetic moment, but maintain the same anisotropy.

Although fig. 11 models the effect of different arrangements of magnetic axes, the same modelled traces could be obtained by considering only a single magnetic axis, and modelling the effects of increasing magnetic anisotropy forces. Then the ‘60 axis weak’ trace corresponds to a single bidirectional axis with a very weak anisotropy (angle between particle magnetisation and direction of easy magnetisation is very large compared to the angle between particle magnetisation and external field), and the ‘90 axis medium’ trace corresponds to an increased anisotropy on the same single bidirectional axis.

Most of the measured unroasted ilmenites (fig. 2) show a very weak anisotropy, even if a 60° angle is assumed between easy magnetisation directions (magnetisation in the basal plane), but some of the more susceptible particles do give a high rotation index that implies a single bidirectional axis.

As the roast temperature increases, the anisotropy appears to increase first, pushing the rotation indices up from below, to remain fairly constant from 450 to 500°C with a lower limit that implies higher-anisotropy 60° axis magnetisation in the basal plane or lower-anisotropy 90° axis magnetisation for many particles, along with some single-axis magnetisation for others. By 500°C most of the particles in the sample have rotation indices that would guarantee separation by magnetic rotation, even though the bulk sample susceptibility may still be too low for a good attraction separation from minerals such as chromite.

Above 500°C even the possibility of magnetisation axes at 60° disappears, and the particles move towards a single unidirectional axis that by 650°C fits about half way between the first trace in fig. 9 (‘360&exp’) and the second trace (‘360 axis high’). First, this implies a movement towards coercive forces that are comparable with or higher than the measuring fields (an ideal magnetic rotation separation situation), and second it implies a higher shape anisotropy but lower mass (or specific) magnetic moment at lower particle susceptibilities for the ferromagnetic components within the ilmenite particles.

By 650°C almost all the rotatable ilmenite particles are behaving as though they contain small elongated permanent magnets of differing sizes.

The persistent group of low rotation index particles with a susceptibility around $6 \times 10^{-5}\ \mathrm{m^3/kg}$ is interesting. This group appears for all three roasted samples above

500°C. Exactly why some particles at this particular magnetic susceptibility should show a tendency to lower rotation indices is not very clear. These particles are now showing evidence for either a bidirectional magnetic axis with very low anisotropy, or for more than one magnetic axis in the plane of rotation. As roast temperatures increase, more particles are moving into this susceptibility group, but the original roast separation results [1] also showed that above 500°C there was a puzzling movement of some particles back down to the non-magnetic and non-rotating fractions. It was suggested there that such a movement could occur with increasing roast temperatures if there was a coalescing of ferromagnetic elements within the ilmenite particles. If such a phenomena did occur, it would probably occur around a particular density of ferromagnetic components, i.e. around a particular magnetic susceptibility. There is therefore the possibility that the low rotation index group indicates this 'drop-out' susceptibility.

The above comments are all based on an averaged rotation index for each particle. An interesting trend is seen when the average rotation index frequency response is examined for each roasted sample, and this is illustrated in fig. 9. Here it has been calculated by dividing the differences between the 10 and 50 Hz indices by the 10 Hz index, averaged over all the particles for each roast temperature. Figure 8 indicates that this frequency response has quite significantly decreased by 650°C.

The rotation index for a particle (given by Allen [2]) can also be written to include domain wall and inertia effects as:

$$R_f = \frac{\sin(\phi + \phi_w)}{1 + I}$$

where: ϕ is the angle between particle magnetisation and external field; ϕ_w is the extra angle due to domain wall velocity; I is a term describing particle inertia.

As it is reasonably certain that by 650°C most particles have a single unidirectional magnetic axis, domain wall velocities are no longer a factor. The inertia term is inversely proportional to the fraction of a cycle available for the particle to accelerate to field rotation frequency. If there is a change from a single bidirectional magnetic axis to a unidirectional axis (a permanent magnet situation), the time available is effectively doubled, and the inertia effect is halved. This is approximately what is seen in Fig. 8.

Both the rotation index trace (figs. 7 and 8) and the drop in frequency response for particles roasted to 650°C (fig. 9) point to a single unidirectional magnetic axis for most particles at this roast temperature. The decrease in frequency response with increasing roast temperature can be seen as a gradual increase in the proportion of unidirectional magnetic axes as the roast temperature increases.

A unidirectional magnetic axis requires that the rotating field used for measurement be lower than the critical field for domain wall motion, which means very high coercive forces for the ferromagnetic components within the particles, especially for the particles of lower susceptibility where the measuring field is highest.

Figures 7 and 8 indicate that cooling a roasted sample in a static magnetic field does not cause any convincing changes in the rotation index plots, but does increase the sample susceptibility. This points to a simple reorientation of already existing, but previously opposing, high coercivity magnetic elements as the sample cools through its Curie temperature, but no change to their size or structure. It also suggests that the presence of some magnetic field during the cooling of roasted samples, even the earth's weak field, may be more important for a magnetising roast than has so far been realised.

It was reported by Allen [1] that cooling the roasted samples in a magnetic field had no effect for roast temperatures below 550°C. At the time this was seen as probably due to the rapid cooling of the sample below the Curie temperature before it could be placed in the static magnetic field. This may well have been the case for some particles, but now it can be seen as more probably due to lower coercive forces and the consequent existence of bidirectional rather than unidirectional magnetic axes.

CONCLUSION

An examination of the magnetic changes that occur in natural ilmenite particles as a result of roasting, indicates that the particles behave magnetically as though they contain discrete ferromagnetic components or elements within a paramagnetic matrix.

Prior to roasting, the ferromagnetic components in particles of natural ilmenite exhibit a wide range of magnetic anisotropies and coercive forces, with most particles in a sample having low magnetic susceptibilities. As oxidising roast temperatures increase up to 650°C, magnetic anisotropy forces in the ferromagnetic elements increase, and the ferromagnetic elements move towards a single magnetic axis. Coercive forces within the ferromagnetic elements also increase until, by 650°C, most of the ferromagnetic elements are behaving magnetically as though the single magnetic axis has become unidirectional in magnetic fields as high as 0.3 T.

For roast temperatures above 500°C, when many particles contain high-coercivity magnetic elements with unidirectional magnetic axes, the cooling of a sample in an external magnetic field, even the earth's weak field, may be an essential part of the magnetising roast process.

It is emphasized that these results have been produced by an oxidising roast in air, as described by Allen [1]. A reducing roast will produce different results.

References

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[2] N.R. Allen. Mineral particle rotation measurements for magnetic rotation separation. *Magn. Electr. Sep.*, **11**(3) (2001), 155–168.