Determination of magnetite grain-size using the Hopkinson effect - examples from Botswana rocks

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Abstract
The Hopkinson effect is the increase in magnetic susceptibility with temperature from room temperature to near the Curie point. Although this effect has been known for more than a century, it has not been effectively utilised as an analysing tool in palaeo, rock, and environmental magnetic studies. This is partly due to the poor understanding of the influence of magnetite (Fe₃O₄) grain parameters on the Hopkinson effect. In an attempt to study the effects of grain size on the Hopkinson effect, magnetite samples with well-defined grain sizes have been used. It was found that in general, magnetic susceptibility enhancement factors (SEPs) obtained by heating the sample in a non-oxidising environment increase with decreasing grain size. The relation of SEP to grain size is linear when plotted on a log-log scale. This relation has been used to infer grain sizes (and hence magnetic domains) for some selected Botswana rocks. The inferred magnetic domains are consistent with independent predictions from hysteresis measurements for the same samples.

Keywords: magnetite, magnetic susceptibility, magnetic domains, Hopkinson effect, Botswana rocks.

Introduction
Hopkinson first reported the increase in magnetic susceptibility with temperature till near the Curie point in 1889 for the element iron (Collinson, 1983). This phenomenon (the so-called Hopkinson effect) has also been observed in other minerals, e.g. magnetite (Radhakrishnamurty and Likhite, 1970). Previous attempts at utilising the Hopkinson effect in rock magnetic studies include the determination of magnetic domains of magnetite (Radhakrishnamurty and Likhite, 1970 & Dunlop, 1974). Radhakrishnamurty and Likhite (1970) observed a gradual increase followed by a gradual decrease in magnetic susceptibility with increasing temperature for basalt samples. They interpreted this observation as being typical for single domain magnetite grains. In contrast, Dunlop (1974) observed a sharp increase in magnetic susceptibility near the Curie point using single domain and small multidomain grains of magnetite. In an attempt to systematically determine the effect of grain size of magnetite in the Hopkinson parameter SEP, samples with well-defined grain sizes have been used in this study.

Sample description
(a) Artificially produced magnetite particles.
SEM (scanning electron microscope) pictures of magnetite samples with well-defined grain sizes used in this study are shown in Figure 1. The method of production of the samples and their characterisation as magnetite have been described elsewhere (King and Williams, 2000 and King, 1996). Such characterisation included XRD (X-ray diffraction) the low temperature magnetic transition (the so-called Verwey transition, Verwey, 1939) that occurs near 120 K for stoichiometric magnetite, and the Curie point.

Fig. 1: SEM pictures for the following magnetite samples: (a) JG0.1 = 0.1 μm, (b) JG0.2 = 0.2 μm, (c) JG0.3 = 0.3 μm, (d) JG0.4 = 0.4 μm, (e) JH0 = 0 μm, (f) JH0.2 = 0.2 μm, (g) JH0.3 = 0.3 μm, (h) JH0.4 = 0.4 μm.
Magnetic hysteresis loops

As a check on the magnetic domain inferred using the Hopkinson parameter SEF for samples NS28a and NS60, magnetic hysteresis loops for the two samples were obtained (Figure 7). The Physics Department (UB) Alternating Gradient Magnetometer (AGM-micromag) equipment from Princeton Measurements was used to get the hysteresis loops for these samples. It can be seen from Figure 7 that these samples readily saturate in fields of about 250 mT. This is typical magnetic behaviour (King, 1996). From Figure 7 (inset), it can be seen that the coercivities for samples NS28a and NS60 are 5.5 mT and 10 mT respectively. These coercivity values are typical for pseudo-single domain maghemite.

It is clear that the magnetic domain prediction from hysteresis loop parameters concurs with predictions from the Hopkinson parameter SEF for these two samples.

It is emphasised here the application of the Hopkinson parameter SEF to grain size determination works well if samples have been heated in a non-oxidising environment. In oxidising environment (e.g. heating in air), chemical alteration can occur and hence the method may not work very well. The gradual increase and gradual decrease of magnetic susceptibility observed in the study of basaltic samples by Radhakrishnamurty and Likhite (1970) are most likely a result of chemical alteration.

Conclusions

Using magnetite samples with well-defined grain sizes, it was found that in general, magnetic susceptibility enhancement factor (SEF) obtained by heating samples in a non-oxidising environment, increases with decreasing grain size. The relation of SEF to grain size is linear when plotted on a log-log scale. This relation was used to infer grain sizes (hence magnetic domains) for some selected rocks from Botswana. The inferred magnetic domains are consistent with independent predictions from hysteresis measurements for the same samples. The Hopkinson parameter (SEF) method of determining magnetic domain is more appealing than other methods such as hysteresis loop parameters because it includes the determination of the Curie point at the same time.
<table>
<thead>
<tr>
<th>Sample name</th>
<th>Average grain size (µm)</th>
<th>SEF</th>
</tr>
</thead>
<tbody>
<tr>
<td>JGO.1</td>
<td>0.1</td>
<td>0.70</td>
</tr>
<tr>
<td>Ge10.2</td>
<td>0.2</td>
<td>0.42</td>
</tr>
<tr>
<td>JH0.2</td>
<td>0.5</td>
<td>0.22</td>
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<tr>
<td>JH1</td>
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<td>0.13</td>
</tr>
<tr>
<td>JH40</td>
<td>1.0</td>
<td>0.02</td>
</tr>
<tr>
<td>NS60</td>
<td>0.3</td>
<td>0.30</td>
</tr>
<tr>
<td>NS28a</td>
<td>0.6</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Table 1: Susceptibility enhancement factor (SEF) for magnetite samples with various grain sizes. Except for samples NS60 and NS28a whose average grain sizes were inferred from plotting of figure 9, the average grain sizes of the samples were determined from SEM pictures.

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References
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