Low-temperature cycling of isothermal and anhysteretic remanence: microcoercivity and magnetic memory

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Abstract

This paper reports low-temperature cycling (LTC) through the Verwey transition of anhysteretic remanence (ARM), partial ARMs and partially demagnetised saturation isothermal remanence (SIRM) induced at room temperature in pseudo-single-domain and multidomain (MD) magnetite. The remanences were cooled in zero field to 50 K and then heated back to room temperature. By inducing partial ARMs over different field ranges and by partially alternating field demagnetising SIRM, it was possible to isolate both low-coercive-force and high-coercive-force fractions of remanence. On cooling through the Verwey transition, a sharp increase in the remanence was observed. The relative size of the jump increased as the high-coercive-force fraction was increasingly isolated. This behaviour is interpreted as being due to both an increase in the single-domain/multidomain threshold size on cooling through the Verwey transition and to the reduction or elimination of closure domains in the low-temperature phase. In addition, the memory ratio, i.e. the fraction of remanence remaining after LTC divided by the initial remanence, was found to be higher for the high-coercive-force fraction than the low-coercive-force fraction. In our interpretation, the high-coercivity fraction behaviour is associated with reversible domain re-organisation effects, whilst the low-coercive force fraction’s behaviour is associated with irreversible domain re-organisation and (de-)nucleation processes. Due to the decrease in magnetocrystalline anisotropy on cooling to the Verwey transition, the high-coercive-force fraction is likely to be magnetoelastically controlled. Thus, a rock displaying high-coercive-force behaviour is likely to carry a palaeomagnetically meaningful remanence with high unblocking temperatures. In addition, LTC analysis can be used to identify the domain state dominating the natural remanence in magnetite-bearing rocks.

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1. Introduction

Zero-field low-temperature cycling (LTC) of magnetite-bearing rocks from room temperature to liquid nitrogen temperature causes varying degrees of partial demagnetisation of remanence depending on domain state [1–8]. The partial demagnetisation is thought to be primarily related to
changes in the magnetocrystalline anisotropy, but
the exact processes governing LTC behaviour are
still unresolved. There is a need for greater under-
standing if LTC is to become a reliable palaeo-
magnetic or environmental magnetic technique.
To understand the mechanisms controlling de-
magnetisation, it is revealing to study the LTC
behaviour of various initial remanence states.
The direct measurement of remanence during
LTC has not been extensive; previous studies
have predominantly considered saturation iso-
thermal remanence (SIRM) [1–4, 6–8]. It is found that
SIRM induced in a multidomain (MD) sample at
room temperature gradually decreases on cooling
to the magnetocrystalline energy isotropic point
\( T_K \) at 130 K and the Verwey transition
\( T_V \) at 120–124 K [9, 10]. This decrease in remanence
has been associated with domain re-ordering ef-
sic, i.e. domain wall re-equilibration or domain
nucleation [4, 7]. On cooling through \( T_V \), an in-
crease in magnetisation or ‘jump’ is observed.
The size of the jump for SIRM carried by assem-
blages of crystals is relatively small compared to
that of SIRM induced in orientated single crystals
\( \sim 1–4 \) mm in diameter [6, 8]. Below the mono-
clinic/triclinic phase of magnetite, SIRM displays
thermally reversible behaviour. On warming
through \( T_V \), the jump is found to be mainly,
though not always, reversible. On warming from
\( T_K \) to room temperature, SIRM displays irreversible
behaviour, with some increase in magnetisa-
tion (recovery). The degree of recovery is found to
be dependent on internal stress [11] and has been
associated with the stiffening of domain walls on
heating [7].
There are a limited number of studies of the
low-temperature cycling of thermoremanent mag-
netisation (TRM) [1, 7, 12], of which by far the
most extensive is the paper of Muxworthy and
McClelland [7], who measured LTC behaviour
of TRM and partial TRMs (pTRMs) induced in
well characterised synthetic and natural stoichi-
ometric pseudo-single-domain (PSD) and MD
magnetites. TRM and pTRM carried by assem-
blages of magnetite crystals consistently displayed
a large jump on cooling through \( T_V \). The size of
the jump was influenced by grain size, the temper-
ature range over which the pTRM was acquired
and inducing field intensity, e.g. high-temperature
pTRMs induced in small fields in PSD samples
displayed larger jumps than pTRMs induced in
larger fields in large MD samples at lower tempera-
tures. The size of the jump was on average larger
than that observed for the same samples carrying
an SIRM [7].
In addition to experiments, micromagnetic and
other numerical models of single sub-micrometre
crystals predict the jump behaviour at \( T_V \) for sim-
ulated SIRM, TRM and pTRM structures [5, 13].
However, due to the simplifications of the model
the size of the simulated jump was larger than
that observed for assemblages of small PSD mag-
netites.
The behaviour at \( T_V \) was explained by a shift in
domain state to a more single domain-like (SD)
structure and the removal of closure domains in
the monoclinic phase [5, 7, 8]. This was concluded
by examining hysteresis data, micromagnetic so-
lutions and by considering the relative anisotropy
which indicates the favourability of closure do-
 mains [5, 8, 14]. It was suggested that closure do-
 mains play a more important role in reducing the
demagnetising energy in small grains than larger
grains and in pTRM structures acquired at high
temperatures.
The results of TRM LTC curves have been re-
vealing, but our current knowledge of MD TRM
acquisition is incomplete, making it difficult to
fully interpret such LTC data. To quantify and
improve our understanding of the behaviour of
remanence at low temperatures, in this paper
LTC curves of partially alternating-field (AF)
demagnetised SIRM, anhysteretic remanence (ARM)
and partial ARM (pARM) are presented. It is the
first time LTC curves for such remanences have
been reported. Although these remanences in MD
magnetite are not fully understood [15], our
knowledge is better than that of TRM as we
can more accurately identify which part of the
coercivity spectrum is affected during the induc-
tion.

2. Sample description
Magnetite samples from two different origins
were utilised in this study; three small commercial MD samples \(W(1.7 \mu m)\), \(W(7.0 \mu m)\) and \(W(11 \mu m)\), acquired from Wright Industries, and two MD synthetic samples made by hydrothermal recrystallisation (H (39 \(\mu m))\) and (H (108 \(\mu m))\).

The Wright samples were obtained 6 months before the experimentation and stored in a desiccator. Grain size distributions were found to be log-normal from scanning electron microscope photographs (Table 1). X-ray diffraction (XRD) spectra measured shortly after receiving the samples appeared to be those of pure magnetite within experimental limits. However, during the LTC measurements at the Institute for Rock Magnetism, spectra measured using a \(^{57}\)Co source identified non-stoichiometric magnetite (Fig. 1). It is uncertain whether this partial oxidation occurred during the 6 months of storage or if the samples were initially non-stoichiometric.

Mössbauer parameters were determined by fitting two sextets (A and B) using a Lorentzian fitting programme (Table 2). The oxidation state of the samples was estimated by considering the ratio of the area of the two sextets (\(F_A\) and \(F_B\)). For stoichiometric magnetite the ratio is experimentally \(\sim 1.9\) [16]. Two possible interpretations are considered; the first is that there are two separate phases within the sample, i.e. a stoichiometric maghemite phase and a stoichiometric magnetite phase. The second interpretation is that the samples are homogeneous cation-deficient magnetite with a formula given by \(\text{Fe}^{3+}_{3z/2}\text{Fe}^{2+}_{1-z}\square_{Z/3}\text{O}_4\), \(0 \leq z \leq 1\), where \(\square\) = cation vacancy. In reality it is likely that neither model is correct and the mineralogy is a mixture of both. It is possible to differentiate between two such configurations by

![Fig. 1. Room-temperature Mössbauer absorption spectra for samples \(W(1.7 \mu m)\) and \(W(11 \mu m)\). Two Lorentzian sextets have been fitted to each spectrum. \(W(11 \mu m)\) is nearly stoichiometric magnetite, whereas \(W(1.7 \mu m)\) is slightly non-stoichiometric. The difference between the two spectra is most clearly seen by comparing the peaks on the extreme left. The difference in absorption percentages is simply due to differences in sample mass.](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Size ((\mu m))</th>
<th>Mean AR</th>
<th>(H_c) (mT)</th>
<th>(H_{cr}) (mT)</th>
<th>(M_{rd}/M_s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(W(1.7 \mu m))</td>
<td>(1.7^a)</td>
<td>0.2(^a)</td>
<td>1.4</td>
<td>16.1</td>
<td>39.1</td>
</tr>
<tr>
<td>(W(7.0 \mu m))</td>
<td>7(^a)</td>
<td>3(^a)</td>
<td>1.0</td>
<td>6.2</td>
<td>24.9</td>
</tr>
<tr>
<td>(W(11 \mu m))</td>
<td>11(^a)</td>
<td>3(^a)</td>
<td>1.8</td>
<td>4.6</td>
<td>20.4</td>
</tr>
<tr>
<td>(H(39 \mu m))</td>
<td>39</td>
<td>9</td>
<td>–</td>
<td>1.2</td>
<td>24.7</td>
</tr>
<tr>
<td>(H(108 \mu m))</td>
<td>108</td>
<td>31</td>
<td>–</td>
<td>0.94</td>
<td>18.2</td>
</tr>
</tbody>
</table>

The Wright samples had log-normal distributions. These were transformed into \(\log_{10}\) space before the mean and standard deviation were calculated. The hydrothermally produced samples displayed a more Gaussian size distribution. \(\sigma\) is the standard deviation of the normal distribution in either case, but \(\sigma\) given in the table for the Wright samples is not symmetric about the mean on a linear scale. The aspect ratio (AR) is the ratio of the long axis over the short axis. No aspect ratio was measured for the hydrothermally produced samples as the grains were nearly all symmetrical. The hysteresis parameters \(H_c\), \(H_{cr}\) and \(M_{rd}/M_s\) are shown.

\(a\) Converted from \(\log_{10}\) space.
measuring Mössbauer spectra in the presence of an external field [16]. However, this facility was not available. The two-phase model is relatively straightforward to determine. The oxidation parameter \( z \) was determined by comparing the \( F_B/F_A \) ratios in Table 2 to the average of the experimental data in Ramdani [17] and Schmidbauer and Keller [18]. The degree of oxidation was found to decrease with grain size. For the two-phase model, the percentage of maghemite was determined to be 12% in \( W(1.7 \mu m) \) and only 2.3% in \( W(11 \mu m) \). In the cation-deficient model, \( z \) drops from 0.045 for \( W(1.7 \mu m) \) to 0.009 for \( W(11 \mu m) \).

The stoichiometry was checked magnetically by examining both the Curie and Verwey temperatures. The Curie temperatures were determined by measuring thermomagnetic curves on a Princeton Measurements vibrating sample magnetometer (VSM). The Curie temperatures were 583 ± 1°C, which is a little above that of stoichiometric magnetite of 575–580°C [19].

The Verwey transition was examined by measuring susceptibility using a Lakeshore Cryotronics AC susceptometer (Fig. 2). The transition was relatively sharp in \( W(11 \mu m) \), indicating near-stoichiometric magnetite, but broader in \( W(1.7 \mu m) \). It is uncertain whether this broadening in the smaller grains is due to differences in stoichiometry and/or grain size.

The second set of samples, \( H(39 \mu m) \) and \( H(108 \mu m) \), were produced by the hydrothermal recrystallisation method [20]. The magnetic properties of these samples have been reported several times previously [7,21]. Grain size distributions and magnetic parameters of the samples are summarised in Table 1. XRD analysis and Mössbauer spectroscopy found that the samples were pure magnetite. The samples were dispersed in KBr pellets, and then vacuum-sealed in quartz capsules. As the samples were stored in vacuum, oxidation was not expected. However, to check, warming curves for an SIRM at 35 K were measured using a Quantum Design MPMS. A very sharp Verwey transition was observed, indicating stoichiometric magnetite.

Magnetic hysteresis parameters were measured at room temperature for all six samples using the VSM (Table 1). The hydrothermally grown samples have very low values for coercive force (Hc) and low saturation remanence (Mr) to Ms ratios, suggesting low dislocation densities. The Wright samples have slightly higher Hc and Mr/Ms values, indicating an increased level of internal stress.

![Fig. 2. Low-temperature susceptibility curves for samples \( W(1.7 \mu m) \) and \( W(11 \mu m) \). The susceptibility was measured during warming for two frequencies: 400 Hz and 4000 Hz. No frequency dependence was observed. Only the 400 Hz data are shown. The samples were cooled in zero field.](image-url)
which may be related to the non-stoichiometry. In addition to the hysteresis properties, first-order reversal curve (FORC) diagrams have been measured for these samples at room temperature [21].

3. Experimental procedures

A series of LTC experiments were made; samples were given either an ARM, pARM or partially demagnetised SIRM, and the behaviour of the remanence was measured during LTC. These experiments were designed to examine how different fractions of the coercivity spectrum behave during LTC.

ARMs and pARMS were induced with a DTech pARM inducer/demagnetiser. The maximum AC field was 200 mT. The effect of applying different biasing DC fields over different field ranges was examined. Partially demagnetised SIRMs were examined by first inducing an SIRM in a field of 1 T using an ASC pulse magnetiser. The samples were then partially demagnetised using the AF demagnetiser.

Low-temperature behaviour of remanence was measured using an MPMS. Before measuring the LTC curves, the internal field inside the MPMS was reduced to ±0.5 μT. To test the effects of small biasing fields on LTC curves, Muxworthy and McClelland [7] applied fields up to ~500 μT during LTC of SIRM. They found that although the field induced a magnetisation which shifted the LTC curves in the direction of the biasing field, the overall behaviour of the low-temperature thermomagnetic curve was not affected, i.e. the jump feature at \( T_V \) still existed regardless of the bias field direction. They concluded that small biasing fields of the size ±0.5 μT were not the cause of the jump behaviour on cooling through \( T_V \).

4. Results

4.1. Low-temperature cycling of partially AF-demagnetised SIRMs

For three samples, \( W(7.0 \, \mu m) \), \( H(39 \, \mu m) \) and \( H(108 \, \mu m) \), LTC curves were measured for partially AF-demagnetised SIRMs (Fig. 3). The general shape of the undemagnetised SIRM LTC curves was in good agreement with previously published data for assemblages of PSD and MD magnetite [2-4,7]. As the peak AF demagnetisation field increases, several features are observed. First, the demagnetisation which occurs on cooling to \( T_K \), associated with domain wall re-ordering, decreases. Second, a sudden increase or jump occurs on cooling through \( T_V \) for peak AF ≥ 50 mT. Third, the memory ratio, i.e. (memory remanence)/(initial remanence), increases. The width of the Verwey transition was greater in sample \( W(7.0 \, \mu m) \) than in \( H(39 \, \mu m) \) and \( H(108 \, \mu m) \), most likely because of differences in stoichiometry.

The sharp change in magnetisation at \( T_V \) is characterised by a new parameter, the ‘Verwey jump’ \( (\Delta V_J) \), which is defined as the size of the increase or decrease in magnetisation across the Verwey transition on cooling normalised by the room temperature magnetisation. Specifically, we calculated \( \Delta V_J \) as the jump in magnetisation over a 10 K interval, starting at \( T_V \) and ending 10 K below \( T_V \), divided by the initial magnetisation. Because \( T_V \) decreases if magnetite contains impurities or is partially oxidised [9], \( T_V \) should be iden-
tified by eye for each individual sample as the temperature at which the magnetisation begins to change sharply. Commonly $T_V$ is associated with a minimum or a ‘wild zone’ [3] in the temperature range 110–130 K. For all the samples in this study, $T_V$ was at ≈124–125 K. Both positive and negative values for $\Delta V J$ have been previously observed [1,6,7].

The absolute values of the jump, i.e. $\Delta V J \times$ the initial remanent magnetisation, increase for $W(7.0 \mu m)$ and $H(39 \mu m)$, showing a peak at an AF of 50 mT, before decreasing gradually with increasing AF (Fig. 4a). For $H(108 \mu m)$ the absolute jump size decreases with alternating field. In contrast, $\Delta V J$ is seen to increase as a function of peak AF for samples $W(7.0 \mu m)$, $H(39 \mu m)$ and $H(108 \mu m)$ (Fig. 4b). That is, the relative size of the jump increases with peak AF, suggesting that $\Delta V J$ is associated with the high-coercivity fraction.

The memory ratio is also seen to increase with peak AF (Fig. 4c), supporting the idea that the high-coercivity component of remanence displays reversible behaviour to LTC [7]. The AF-demagnetised memory ratio increases with peak AF for $W(7.0 \mu m)$, reaching a value of 1 at 100 mT.

From the complete data set for each sample, it is possible to construct standard AF demagnetisation curves for both initial remanence and memory (Fig. 5). The initial remanence is more easily demagnetised than its memory until some AF level is reached where the two AF demagnetisation curves become identical. Similar contrasts between AF demagnetisation curves of initial remanence and memory have been reported previously [7,22–24].

4.2. Low-temperature cycling of pARM

LTC curves were measured for pARM induced
in samples $W(1.7 \mu m)$ and $H(39 \mu m)$ using a DC field of 200 $\mu T$ (Fig. 6). The maximum AF was always set to 200 mT. $\Delta V_J$ and the memory ratios increased with the AF field range over which the pARM was induced for both $W(1.7 \mu m)$ and $H(39 \mu m)$ (Figs. 6 and 7). For pARM$_{20}^0$, i.e. a pARM induced between AFs of 20 and 0 mT, $\Delta V_J$ for $H(39 \mu m)$ was observed to be negative. Negative $\Delta V_J$ values have been observed before for various types of TRMs induced in large MD magnetites [1,7]. That $\Delta V_J$ is a maximum for high AF ranges supports the idea that $\Delta V_J$ is related to the high-coercive-force fraction.

For sample $W(1.7 \mu m)$, the memory ratio for pARM$_{20}^0$ is smaller than the ARM memory ratio, i.e. pARM$_{200}^0$. The ratios were 40% and 92% respectively, supporting the idea that low-temperature demagnetisation is related to the low-coercivity fraction as suggested by Figs. 3 and 4. $H(39 \mu m)$ displayed different behaviour, and the memory ratios for pARM$_{20}^0$ and ARM were, respectively, 18% and 4%. It is unclear why the memory ratio for ARM was less than any of the pARM memory ratios (Fig. 7).

From the pARM data it is possible to determine remanence acquisition distributions. For $W(1.7 \mu m)$ the peak in the distribution was for pARM$_{20}^{50}$, whereas $H(39 \mu m)$ displayed a maximum in the distribution at low-field ranges, i.e. pARM$_{20}^0$. These correspond well with cross-sections of the FORC distribution along the $H_C$-
axis (x-axis) reported in Muxworthy and Dunlop [21].

4.3. Effect of ARM acquisition field on LTC behaviour

In Fig. 8, LTC curves for $W(11 \mu m)$ are shown for ARMs produced with a range of DC fields. As the DC biasing field increases, the shape of the LTC curves evolves, with $\Delta V_J$ decreasing (Fig. 9). For the larger DC fields, ARM LTC curves resembled SIRM LTC curves (Fig. 3). The memory ratio also decreases with DC field (Fig. 9). This field-dependent behaviour is similar to that observed for TRM [7].

ARM and ARM memory acquisition curves were linear with applied field up to 200 $\mu$T, in agreement with Muxworthy and McClelland [7], who measured such linear trends for acicular SD magnetite, and hydrothermal and natural MD magnetite.

LTC curves for ARM induced in samples $W(1.7 \mu m)$ and $W(7.0 \mu m)$ in a DC field of 200 $\mu$T displayed similar behaviour to $W(11 \mu m)$ (Fig. 8), i.e. $\Delta V_J = 0$. The memory ratio gradually decreased with grain size, i.e. 92%, 47% and 31% for samples $W(1.7 \mu m)$, $W(7.0 \mu m)$ and $W(11 \mu m)$, respectively. It is uncertain what contribution the differences in stoichiometry made to these memory ratios.

4.4. Reversibility of ARM LTC

To check for reversibility, an ARM (DC field = 200 $\mu$T) was induced in sample $W(11 \mu m)$ at room temperature and the remanence gradually cycled between room temperature and increasingly lower temperatures (Fig. 10). The induced ARM is seen to partially demagnetise on cooling to temperatures $> T_V$ and $T_K$, e.g. cooling to 150 K is sufficient to demagnetise 22% of the initial ARM on warming to room temperature. Similar data have been reported for low-temperature cycled SIRM [4,7]. This result strongly supports the theory that domain wall re-organisation during cooling above $T_K$ is a major contributor to the total low-temperature demagnetisation. On cooling through $T_V$, the memory ratio is seen to drop sharply, indicating that significant demagnetisation occurs on cooling through $T_K$ and $T_V$.

5. Discussion

There are large variations in both the initial remanences and LTC curves shown in this paper.
However, there are two clear trends. First, initial remanences associated with higher-coercivity fractions display the highest $v_{VJ}$ values, e.g. $v_{VJ}$ is largest for SIRM AF-demagnetised to 100 mT (Fig. 3) and pARM$_{200}^{180}$ (Fig. 6). Secondly, the high-coercivity fraction is generally reversible during LTC, i.e. the high-coercivity remanences have the highest memory ratios (Figs. 4 and 7). Irreversible behaviour is observed when the lower-coercivity fraction of remanence is present.

$\Delta_{VJ}$ displays a grain-size dependence (Figs. 4 and 7). $\Delta_{VJ}$ for $H(39 \mu m)$ and $H(108 \mu m)$ is greater than $\Delta_{VJ}$ for $W(1.7 \mu m)$ for all peak AFs of SIRM. However, as the peak AF increases, $\Delta_{VJ}$ for $H(39 \mu m)$ becomes greater than $\Delta_{VJ}$ for $H(108 \mu m)$ (Fig. 4). $\Delta_{VJ}$ for all the pARMs is greater for $H(39 \mu m)$ than $W(1.7 \mu m)$ except for the pARM$_{0}^{50}$ $\Delta_{VJ}$, which is negative for $H(39 \mu m)$ and zero for $W(1.7 \mu m)$.

The memory ratio displays a consistent grain-size dependence (Figs. 4c and 9). Such grain-size dependence of the memory fraction is well documented for other types of remanence [22, 23, 25]. $v_{VJ}$ decreases strongly with increasing ARM DC field (Fig. 9). The memory ratio displays a weaker dependence on ARM DC field. Muxworthy and McClelland [7] observed a similar field dependence of $\Delta_{VJ}$ for pTRM induced in hydrothermally grown MD magnetites for fields between 100 $\mu$T and 5 mT. They related the LTC behaviour of the high-field remanence to a shift in domain structure to a more SIRM-like structure. SIRM structures generally have smaller $\Delta_{VJ}$ values [5, 7]. $\Delta_{VJ}$ for an initial SIRM was zero for Wright sample $W(1.7 \mu m)$, although for some samples $\Delta_{VJ}$ SIRM $> 0$, e.g. $H(108 \mu m)$ (Fig. 4b). Drawing together the results in this study, the effect of decreasing the ARM DC field is to increase the relative size of the high-coercivity fraction of remanence.

5.1. Origin of $\Delta_{VJ}$

In previous papers, simple mutually compatible models were developed to explain positive $\Delta_{VJ}$ behaviour [5–8]. $\Delta_{VJ}$ was attributed to both a shift to a more SD-like domain state and the removal of closure-like domains in the monoclinic phase [5, 7, 8].

The shift in domain state is related to the large increase in the magnetocrystalline anisotropy on cooling through $T_V$, causing the SD/MD threshold size to increase [5, 8]. On cooling through $T_V$ the domain structure tries to denucleate walls, or at least re-equilibrate walls to reduce the total energy. This will on average increase the measured magnetisation of a sample. Generally, denucleation of domain walls is thought to be irreversible, whilst wall re-equilibration is reversible.

That the isolated high-coercive-force fraction of remanence displays reversible behaviour at $T_V$ suggests that its behaviour is controlled by domain wall re-equilibration processes (e.g. Figs. 3 and 6).

The contribution of removing closure domains to $\Delta_{VJ}$ is more debatable, especially for small PSD grains. Closure domains have been clearly observed in large MD magnetite [26]. In small PSD magnetite such features are generally not observed [27, 28]. However, grain-edge features which reduce the magnetic flux leakage in PSD magnetite are predicted to exist and to be difficult to observe experimentally [29]. On simulated cooling through the Verwey transition, the removal of such features was the main contributor to $\Delta_{VJ}$ [5].

The high-coercive-force fraction of remanence is more likely than the low-coercive-force fraction to be associated with domain configurations where domain walls are pinned far from their equilibrium positions. To reduce the energy of these states in the cubic phase, closure domains are likely to form. The removal of such closure domains will result in an increase in $\Delta_{VJ}$. Experimentally, $\Delta_{VJ}$ becomes relatively larger as the higher coercive fraction becomes increasingly isolated (Figs. 4 and 7).

The relative increase in $\Delta_{VJ}$ from both of these effects will be greater for small grains, e.g. reducing a domain structure from two domains to a SD will lead to a bigger increase in $\Delta_{VJ}$ than decreasing from, say, 20 domains to 18, and the importance of closure domains is also greater for domain structures with only a few domains. In contrast, the results in this paper show that $\Delta_{VJ}$
increases with grain size (Figs. 4 and 7). This discrepancy may be due to the very different microcoercivity distributions of the Wright and hydrothermal samples; the influence of microcoercivity may mask the expected grain-size trend. Özdemir et al. [8] found a similar grain-size relationship for SIRM, whereas Muxworthy and McClelland [7] found that $\Delta V_J$ for TRM increased as the grain size was decreased.

In addition to these two mechanisms, twin domain (TD) structures are thought to be important to the low-temperature magnetic behaviour due to the high magnetocrystalline anisotropy [6–8]. However, it is difficult to assess their importance as certain key facts are unknown. Firstly, there may or may not be TD boundaries as there is a single-TD to multi-TD critical size [30,31]. The critical size depends on the boundary conditions of each grain, and it is not possible to roughly estimate the critical size as this is a highly non-linear calculation (A. Jacobs, personal communication, 2002). Secondly, the interaction of remanence structures with TDs is unknown. Either the twin or the magnetic domains could dominate the remanence behaviour depending on whether the strain or magnetic energy dominates (A. Jacobs, personal communication, 2002). In addition, it is possible due to the symmetry considerations, for certain orientations of TD and magnetic domain walls to re-adjust independently (C. Medrano, personal communication, 1999).

5.2. Origin of high memory ratios

High memory ratios reflect reversible behaviour through the entire LTC curve, including on cooling through $T_V$ (Figs. 3, 6 and 8). Irreversible behaviour is thought to be due to demagnetisation of domain walls, both on cooling from room temperature to $T_K$ due to the decrease in the magnetocrystalline anisotropy (Fig. 10), and on cooling to below $T_V$. High memory ratios were found to be related to high coercivity remanences (Figs. 4 and 7). This is in agreement with AF demagnetisation curves of both initial and memory remanence (Fig. 5, [7,22,23]). These high-coercivity remanences are more likely to be stress-controlled, and hence relatively unaffected by changes in the magnetocrystalline anisotropy. When palaeomagnetic remanences are thermally demagnetised, a magnetoelastically controlled remanence decays much less rapidly than a magnetocrystalline controlled one. Thus a high memory ratio, implying magnetoelastic control, is likely to be an indicator of a palaeomagnetically meaningful remanence with high unblocking temperatures.

6. Conclusions

This study clearly demonstrates that LTC behaviour is influenced by the coercive force distribution of the remanence. It is shown that key features such as $\Delta V_J$ and high memory ratios are associated with the highest coercivity fraction of remanence. Some of the LTC behaviour can be explained by the application of previously reported theories [5–8].

The identification of high memory ratios or a significant $\Delta V_J$ peak for a natural remanent magnetisation (NRM) would indicate that the remanence was dominated by a magnetoelastically controlled high coercivity signal, and that the sample is suitable for palaeomagnetic investigations. The measurement of memory ratio does not require low-temperature measurements of magnetisation. It is recommended that LTC be used as a pre-selection technique in palaeomagnetic studies, and if applied judiciously it could be used as a cleaning technique [32].

In addition, LTC analysis can be used to identify the domain state dominating the NRM signal in magnetite-bearing rocks; high memory ratios and large jumps at the Verwey transition indicate a high-coercive force MD remanence, high memory ratios and small jumps at the Verwey transition suggest an SD remanence, whilst low-memory ratios indicate a low-coercive force MD remanence.

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