Anhysteretic Remanent Magnetization of Small Multidomain Fe_3O_4 Particles Dispersed in Various Concentrations in a Non Magnetic Matrix

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Abstract. The anhysteretic remanent magnetization (ARM) and hysteresis parameters of synthetic, small, multidomain particles (0.2–0.4 μ) of Fe_3O_4, dispersed at various concentrations in a non magnetic matrix (packing factors $p=0.01$–0.3), have been measured. The coercive force H_c is weakly dependent on p. At higher concentrations the shape of the sample has a strong influence on the magnetization. There appears to be an additional concentration dependence of the ARM. However, the independence of partial ARMs also holds for larger p.

Key words: Rock magnetism – Small particles – Anhysteretic remanent magnetization – Fe_3O_4.

Introduction

In recent years, in rock magnetism, a series of studies have been devoted to the problem of determining the palaeointensity of the geomagnetic field from the magnitude of the natural remanent magnetization (NRM) of rocks. In the models and methods used for this purpose it is suggested that the NRM is a thermoremanent magnetization (TRM). In order to determine palaeointensities, the method proposed by Thellier and Thellier (1959), or one based on it with minor or major modifications, is generally applied. For this procedure the samples are given a TRM in the laboratory, i.e., they have to be heated, so chemical alteration might occur.

For some years investigations of palaeointensity have been performed with the aim of replacing the laboratory TRM by the anhysteretic remanent magnetization (ARM). In order to produce an ARM, a sample is subjected to the combined action of a small, steady (D.C.) and an alternating (A.F.) magnetic field. The A.F. field initially saturates the sample during each cycle and decreases slowly to zero, with the D.C. field remaining constant. The resulting magnetization is called anhysteretic magnetization (AM). After reducing the D.C. field to zero the remanence is the ARM. Both the TRM and the ARM represent some kind of magnetic equilibrium state, although the two states may not be the same. This similarity, which may apply preferentially to single-domain particles (SDP), helps explain why both remanences show some similar properties: (1) Similar stability against A.F. demagnetization (Rimbert 1959; Levi and Merrill 1976): (2) Both are linearly proportional to a weak, inducing magnetic field (Rimbert 1959): (3) The addition law is valid for partial ARM and TRM (Patton and Fitch 1962; Dunlop and West 1969). On the other hand, there is a major difference between the states: the

TRM arises predominantly at high temperatures whereas the ARM is usually produced at room temperature (Dunlop et al. 1975).

Up to 1969, the AM and the ARM were explained, in particular for SDP, in terms of static models including magnetostatic interaction between grains in the specimens (review by Wohlfarth 1964; Kneller 1968). Jaep (1969, 1971) introduced a kinetic analysis for the ARM of SDP based on the concept of fluctuation phenomena, as developed by Néel (1949). In this model the analogy between ARM and TRM becomes clear and, the attempt to replace TRM by ARM measurements is justified in principle. In this picture the initial anhysteretic susceptibility, $\chi_{AM} = \lim_{H \to 0} dI_{AM}/dH$ (I_{AM} = anhysteretic magnetization, H = steady magnetic field), remains finite even for highly diluted SDP, contrary to the old models. Using Jaep's results, Banerjee and Mellema (1974) derived formulae for the ratio ARM/TRM of SDP, including interactions between particles. Shcherbakov and Shcherbakova (1977) presented similar formulae. Kneller and Köster (1977) investigated the relation of χ_{AM} to the static magnetic parameters of SDP. For the ARM of multidomain particles (MDP), a microscopic theory like that developed for SDP is still missing. In this case the magnetization process is complicated by the presence of domain walls, whose motion is determined by factors such as lattice imperfections (e.g., stacking faults) and surface irregularities. A phenomenological theory of ARM was given by Gillingham and Stacey (1971).

In many rocks, the magnetization is carried by magnetic grains consisting of Fe_3O_4 or spinels which are close to Fe_3O_4 in composition. The Fe_3O_4 grains in rocks are frequently above single-domain (SD) size (300 Å–500 Å, Morrish and Yu 1955; Dunlop 1973; Butler and Banerjee 1975) and below multidomain (MD) size (<20 μ, Stacey 1963; Parry 1965. These grains are small MDP which show neither pure SD nor pure MD behaviour. In rock magnetism they are sometimes called pseudo-SDP. These grains can occur in quite high concentrations in oxidized titanomagnetite intergrowths (Evans and Wayman 1974; Davis and Evans 1976).

We have investigated the ARM and hysteresis properties of synthetic equidimensional Fe_3O_4 grains in the grain size range 0.2–0.4 μ, in relation to their concentration in specimens with volume fractions (packing factors) $p=0.01$–0.30.

The ARM of small Fe_3O_4 MDP in low concentrations has been investigated in the past by Rimbert (1959), Patton and Fitch (1962), Gillingham and Stacey (1971) and Johnson et al. (1975) The last mentioned authors found no difference between the demagnetization properties of the ARM carried by dispersed SDP and small MDP. Levi and Merrill (1976, 1978), in their study

of ARM and TRM characteristics of small Fe_3O_4 grains, gave curves of the ratio ARM/TRM with varying grain size. Their Fe_3O_4 grains, in the small MD size range were diluted to less than 1 wt.%, in a non-magnetic Al_2O_3 matrix. Sugiura (1979) investigated the concentration dependence of ARM in small Fe_3O_4 MDP with packing factors $p = 3 \cdot 10^{-6} - 2 \cdot 10^{-2}$. He had a mixture of SDP and small MDP in his samples. Surprisingly he found a marked concentration dependence of the ARM even for these high dilutions.

Sample Characterisation

The theoretical domain wall thickness in bulk Fe_3O_4 is approximately 0.15μ (Morrish and Yu 1955; Butler and Banerjee 1975). Therefore Fe_3O_4 grains in the size range of $0.2-0.4 \mu$ contain only one or a few domain walls. This and surface effects prevent the development of MD characteristics. In a detailed model one has to consider furthermore whether the grains are equidimensional, of predominantly cubic or spherical form, or irregularly shaped. In all three cases the thickness, the number and the structure of the domain walls may be different.

The magnetic structure of such two- or more-domain particles, shaped as parallelepipeds or spheres, has been investigated theoretically by Amar (1957, 1958 a, b) and Stapper (1969), respectively. For Fe_3O_4 grains, Morrish and Yu (1965), Stacey (1963), Dunlop (1973), Stacey and Banerjee (1974) and Butler and Banerjee (1975) have estimated the size for the transition from SDP to MDP and the possible structure of small MDP. In the last mentioned two papers the authors have emphasized the effect of surface terminations of domain walls. They conclude that SD-like moments occur at the surface of a grain. According to their model, the magnetization of a grain can be described by a superposition of SD and MD behaviour caused by different parts of the grain. Dunlop et al. (1974) found that equidimensional Fe_3O_4 grains of 0.22μ are two-domain grains and those of less than 0.2μ (still above SD size) consist essentially of one domain wall which behaves like one SDP. Shcherbakov (1978), assuming a particle to have the shape of a parallelepiped and using Amar's formulae, estimated that Fe_3O_4 grains of $0.2-0.4 \mu$ would be two- and three-domain grains. According to this concept the ratio of the saturation remanent magnetization (I_{RS}) to the saturation magnetization (I_S) should be $I_{RS}/I_S = 0.2-0.6$ for two-domain grains and $I_{RS}/I_S = 0.1-0.18$ for three-domain grains. A similar estimation concerning the number of domain walls has been carried out by Moskowitz and Banerjee (1979). A circular configuration of spins for small, spherical MDP, as discussed by Morrish and Yu (1955), implies very low saturation remanent magnetization.

Effective Field in a Sample

It has been found that the ARM for heterogeneous samples is related to internal magnetic interaction effects. For this reason it is suited to the study of structural properties. The ARM depends also on the external demagnetizing factor of the sample. The latter effect is significant when the magnetic material is dispersed in high concentration. Both effects may possibly be separated when the ARM is measured as a function of the sample shape and of the concentration.

In order to describe magnetic interaction and demagnetizing effects one can aplly the 'effective field method' In this model one considers the internal magnetic field H_i acting on each individ-

ual grain. H_i can be thought to be approximately composed of the following terms

$$H_i = H_{ex} + H_d^o + H_{Lor}^o + H_{Lor}^i + H_d^{gr}.$$

H_{ex} = External magnetic field
H_d^o = Demagnetizing field of the sample. When the sample is macroscopically homogeneous and has the outer form of an ellipsoid, H_d^o is uniform and is given by $H_d^o = -N_g I$. N_g = demagnetizing tensor of the sample, I = average magnetization of the sample
H_{Lor}^o = Field inside a spherical cavity centered on the grain in question (Lorentz sphere). When the magnetization of the sample, I, is macroscopically homogeneous and a sufficient large sphere is taken, the effect of the grains outside the cavity can be described by a uniform field $H_{Lor}^o = +4\pi I/3$ within the sphere
H_{Lor}^i = Field of the grains inside the Lorentz sphere acting on the central grain. This field depends on the properties and distribution of the grains and on the interaction between them. It is called the local or interaction field
H_d^{gr} = Demagnetizing field due to the magnetization of the grain itself. This field will vary in some complicated way inside a small MDP

The concept of Lorentz sphere is, in general, valid if the grains are arranged isotropically and their orientations are statistically random.

Experimental Method

Several commercially available Fe_3O_4 powders, with grain sizes of $1-5 \mu$, were checked for purity using magnetization data and Mössbauer spectroscopy. With the help of the latter it was found that the powders contained up to about 20% of either γ-Fe_2O_3 or a solid solution of Fe_3O_4-γ-Fe_2O_3. Similar observations concerning commercial Fe_3O_4 powders have been made by other workers (Levi and Merrill 1978).

For this reason, and to reduce magnetic interaction which may hinder the dispersal of magnetic grains in a non-magnetic matrix, hematite (α-Fe_2O_3) was taken as the starting material of the samples. After the dispersal α-Fe_2O_3 was reduced to Fe_3O_4. Commercial α-Fe_2O_3 powder (Fluka AG) and ZrO_2 (Fluka AG), which was previously milled to a grain size $<1\mu$, were dispersed and mixed ultrasonically in acetone. The mixing was continued in a slowly rotating plastic cylinder, containing a few hundred small glass spheres, while the acetone evaporated. Under the optical microscope the α-Fe_2O_3 grains appeared to be well dispersed (magnification $\times 1,100$). The mixture was pressed into pellets (2–3 kbar). The reduction of the α-Fe_2O_3 to Fe_3O_4 was attempted at first using hydrogen at 300° C. In addition to the Fe_3O_4, however, the samples contained a certain amount of either Fe_2O_3 or FeO and metallic Fe. Better Fe_3O_4 was obtained using a mixture of approximately 90% N_2 and 10% H_2 at 500° C for about 40 min. The quality of Fe_3O_4 grains, prepared in this way, has been studied by Weisweiler and Alavi (1977). By examining some dozens of specimens, thus produced, a series of samples was found to contain fairly pure Fe_3O_4. Others had the correct saturation magnetization but were found to be contaminated by FeO and Fe (Mössbauer spectroscopy). These findings proved the samples to be too critically dependent on the gas mixture and on temperature variations. Finally, for sample preparation, a mixture of H_2 and H_2O vapour
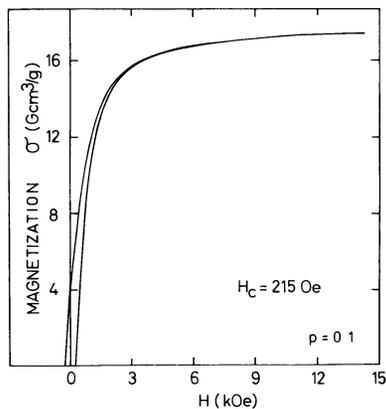
Fig. 1. Upper branch of a hysteresis loop for a spherical sample with $p=0.1$, measured at room temperature

Fig. 2. The ratio I_{RS}/I_S vs the geometrical demagnetizing factor N_g of samples with various packing factors p

was used at 400° C (ratio of partial pressures $p_{H_2}/p_{H_2O} \simeq 12$). The Fe_3O_4 prepared in this way was reproducible and the samples were homogeneous. The specimens were cooled down to room temperature in 5–10 min by pulling them into a cold extension of the furnace.

It is possible, in view of the low reduction temperature, that the Fe_3O_4 grains were clusters of still smaller grains arising from the reduction process. However, no evidence is seen from scanning electron micrographs (SEM). The SEM showed that the grain sizes were distributed between 0.2 and 0.4 μ. No larger grains were seen. On the other hand, it was not possible to rule out the presence of grains of much less than 1 μ (magnification × 50,000). Both the Fe_2O_3 and Fe_3O_4 grains are almost spherical.

Small cylinders and spheres were cut from the reduced, pressed pellets and fixed in polyester resin to protect them from physical damage and oxidation. The cylinders were 5–7 mm long with length to diameter ratios between 2 and 6; the spheres had an average diameter of 3 mm.

The ARM was generated using an air-cored coil with a maximum A.F. amplitude of 1,500 Oe. A superimposed magnetic D.C. field up to $\simeq 80$ Oe was produced parallel to the A.F. field by an additional coil. All remanences were measured with a Digico spinner magnetometer.

Hysteresis properties were determined with a PAR vibrating sample magnetometer. The I_s was ca. 5% lower than the nominal values. I_s was accurate to 1%–2%.

Results

In Fig. 1 the upper branch of a hysteresis loop, measured on a spherical sample, is presented; it is typical of all concentrations used. The coercive force H_c varies between 210 and 235 Oe with a tendency to the lower values for increasing packing factors p. The relative isothermal saturation remanent magnetization I_{RS}/I_S is plotted in Fig. 2 for varying p and geometrical demagnetizing factors N_g of the samples. The magnetometric demagnetizing factors (Zijlstra 1967) were taken for the cylinders. Values of N_g in Fig. 2 correspond to measurements on spheres and on cylinders
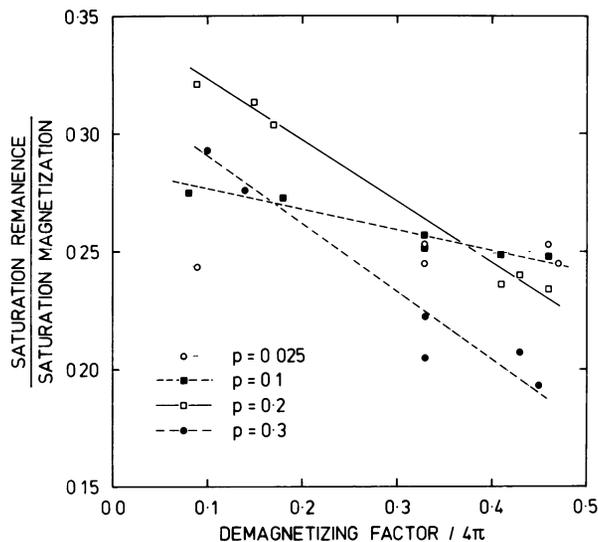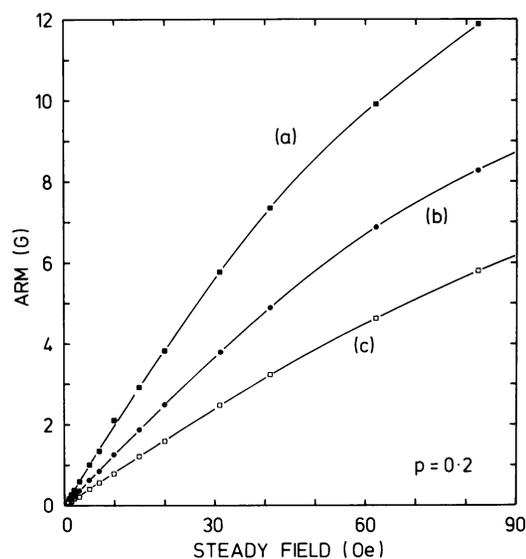
Fig. 3. Dependence of the anhysteretic remanent magnetization (ARM) on the steady field H for samples with $p=0.2$ and three values of the demagnetizing factor N_g: (a) $N_g/4\pi=0.08$, (b) $N_g/4\pi=0.33$, (c) $N_g/4\pi=0.46$

magnetized along and perpendicular to their axes. The points are connected, in a first approximation, by straight lines. Samples with high p show a clear dependence of I_{RS}/I_S on N_g. There is no dependence on N_g for the most diluted samples.

Figure 3 shows how the ARM depends on the applied steady field H for a specimen with $p=0.2$ and three values of N_g. Up to about 15 Oe the ARM varies linearly with H. The concentration dependence of the ARM in a field of 2 Oe for specimens with different N_g is shown in Fig. 4. The dependence of the ratio ARM (2 Oe)/I_S on N_g for high p is much more pronounced than for I_{RS}/I_S. As the law of the variation is not known, an extrapolation to $N_g \rightarrow 0$ cannot be made. A.F. demagnetization curves of I_{RS} and ARM, carried by spheres with $p=0.01$ and 0.2, are plotted in Fig. 5. The samples wth high p have lower median destructive
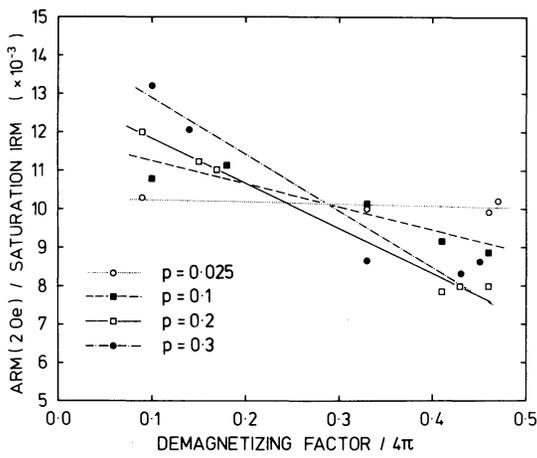
Fig. 4. ARM (2 Oe)/saturation magnetization vs the geometrical demagnetizing factor N_g for samples with various packing factors p

Fig. 6. ARM acquisition vs demagnetization of specimens with $p = 0.2$ and different demagnetizing factors. The magnetic fields given at the points are the A.F. peaks taken for the generation of the ARM and for the demagnetization of the full ARM

Fig. 5. A.F. demagnetization curves of the ARM and I_{RS} of spherical samples with $p = 0.01$ and 0.2

fields $H_{1/2}$ ($H_{1/2}$ = peak field for which half of the initial remanence is removed) for both I_{RS} and ARM. In each case $H_{1/2}$ is smaller for I_{RS} than ARM (2 Oe). Figure 6 shows the relation between the acquisition and the demagnetization of the ARM for a sample with $p = 0.2$ and three values of N_g. The partial ARM acquired in a peak alternating field ($\tilde{H}$) is plotted against the magnetization remaining after the full ARM is demagnetized in the same oscillating field. For all three N_g the points of varying alternating fields are on straight lines with slopes $\simeq -1$.

Discussion

The weak dependence of H_c on p is in agreement with the findings of Morrish and Yu (1955) and of Levi and Merrill (1978) for small MDP of Fe_3O_4. For completely spherical SDP, H_c is determined by the crystalline anisotropy constant $K_1 \cdot H_c \simeq 2\, K_1/I_s$, which for Fe_3O_4, is, about 145 Oe, whereas for shape anisotropy

H_c is much larger. On the other hand, for MDP H_c is always found to be smaller than for SDP. Therefore, from H_c measured for highly diluted concentrations of Fe_3O_4 grains the conclusion may be drawn that, on average, a slight deviation of the grains from spherical shape might occur. The values of H_c are compatible with those measured by Morrish and Watt (1958). Dunlop (1973) found for $\simeq 0.22\,\mu$ monocrystalline, cubic Fe_3O_4 grains, at room temperature, $H_c \simeq 100$ Oe, which is much lower. A possible reason for increased H_c might be pores or voides in the grains, strain and imperfect conversion of α-Fe_2O_3 to Fe_3O_4 (exchange anisotropy).

For $p = 0.025$, $I_{RS}/I_S \simeq 0.22$. This value is probably much higher than one would expect from any circular spin configuration in the grains. It is close to the one predicted by Shcherbakov (1978) for two-domain grains of parallelepipedal or cubic shape. Therefore, in the spherical grains a similar arrangement of domains may be present. For higher p one has to consider agglomeration of grains in pairs or, to a lesser extent, in bridges. These clusters may have a magnetic structure different from the individual grains.

H_d^o, H_{Lor}^o, and H_{Lor}^i will all depend on the concentration of the magnetic grains, and any concentration dependence of the magnetic properties derives both from the outer shape of the measured sample and from the distribution and nature of the magnetic particles. The lack of a full model of the process of anhysteretic magnetization makes it difficult to subtract the shape effect and determine the interactions which occur between neighbouring particles. If the local field H_{Lor}^i is zero or can be approximated by a linear dependence on the average magnetization ($H_{Lor}^i = \alpha\, I$) there should be some shape of the sample such that $(-N_g + 4\pi/3 + \alpha)\, I = 0$ and the static field acting on an individual grain would be independent of p. Figures 2 and 4 show that, within the probable experimental uncertainties, such a crossing

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point may occur (perhaps with the exception of the $p = 0.3$ sample) at a demagnetizing factor of about 0.3. This suggests that the interaction effect between near neighbours is small for p less than 0.3. Due to the lack of data this conclusion is uncertain.

The difference between ARM and I_{RS} A.F. demagnetization curves for the highly diluted Fe_3O_4 powder (Fig. 5) had been observed in a similar form by Dunlop et al. (1973) on dispersed $0.1\,\mu$ Fe_3O_4 grains and by Johnson et al. (1975) on SDP and small MDP of Fe_3O_4 which may have contained a certain amount of γ-Fe_2O_3.

The linearity of the plots in Fig. 6 shows that, although shape-demagnetizing and structural effects influence strongly the intensity of remanence, they do not, in this case, invalidate palaeointensity techniques using partial demagnetization and remagnetization. This is, as mentioned earlier, important for rocks which bear highly concentrated Fe_3O_4 grains in titanomagnetite intergrowths and for archaeomagnetic specimens.

The acquisition of an anhysteretic remanent magnetization may be divided into two parts; the induction of a magnetization in an applied field (superposition of fields) and the partial self-demagnetization when the external field is removed. These processes depend on the demagnetization field of the sample and complicate the interpretation of the remanence data. For SDP the difference between AM and ARM is thought to be negligible, whereas for small MDP, due to domain wall motion, it cannot be neglected.

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