Low Temperature Characteristics of Remanent Magnetization of Magnetite

—Self-reversal and Recovery Phenomena of Remanent Magnetization—

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Abstract

Behaviours of remanent magnetization of magnetite are studied for low temperature range from room temperature to liquid nitrogen temperature (-190°C). The remanent magnetization produced at room temperature on magnetite decreases sharply when magnetite is cooled from room temperature to liquid nitrogen temperature, and recovers a certain percentage of the intensity of the original remanent magnetization when heated to the room temperature (recovery phenomenon), or decreases further in the heating. In some of single crystal magnetites, however, the remanent magnetization was found to reverse its direction at about -145°C in the cooling in non-magnetic space.

In order to explain the anomalous behaviours of a remanent magnetization at low temperatures, it is assumed that the heterogeneity in the stress distribution or in other physical conditions in a grain would result in a heterogeneous distribution of isotropic points (temperatures at which crystal anisotropy energy becomes minimum) within a grain. On the basis of these assumptions, it is shown that the regions with different isotropic points then would interact magnetostatically with each other resulting in the self-reversal or the recovery phenomenon.

Cobalt was found to show the similar recovery and reversal phenomena of a remanent magnetization when it is cooled from 400°C to room temperature in non-magnetic space. Since cobalt has an isotropic point at about 250°C , the self-reversal and the recovery phenomena may be explained in the same way as in magnetite.

1. Introduction

Because of its central importance as a rock forming ferromagnetic mineral in palaeomagnetism, titanomagnetite series have been most extensively studied. However, owing to the undue feeling that TRM was produced only in a cooling process from a certain high

temperature to atmospheric temperature, the study on the magnetic properties of rock forming minerals has been mainly confined to higher temperature region: namely, above atmospheric temperature.

Domenicali (1950) published a detailed study on the low temperature properties of magnetite. His studies were devoted to such properties as magnetization, magnetostriction, electrical conductivity and others. For palaeomagnetist, however, the remanent magnetization is the primary concern. Few work has been carried out as to the low temperature behaviours of the remanent magnetization of titano-magnetites.

In a recent paper by Nagata, Ozima and Yama-ai (1963), it was shown that a stable remanent magnetization was acquired by heating magnetite from low temperature to atmospheric temperature in a weak magnetic field. For the further understanding of a remanent magnetization as a basis for palaeomagnetism, it may therefore be important to extend the study of the remanent magnetization of rock forming ferromagnetic minerals to low temperature range.

In this report, some of the low temperature characteristics of the remanent magnetization of magnetite is given. One of the most interesting behaviours of the remanent magnetization of magnetite is the self-reversal phenomenon (Yama-ai, Ozima and Nagata, 1963). Several examples of self-reversal of a remanent magnetization in natural rock forming minerals have been reported (Uyeda, 1958; Carmichael, 1961; Everitt, 1962), in which hematite-ilmenite solid solution (Uyeda and Carmichael) and pyrrhotite (Everitt) were responsible for the self-reversal phenomenon. The mechanism which is responsible for the self-reversal of a remanent magnetization of magnetite seems to be very different from those for hematite-ilmenite and pyrrhotite cases.

In the next section, samples which were used for the experiments are described. In § 3, experimental results about the self-reversal and the recovery phenomenon of a remanent magnetization of magnetite will be given. Deducing from the experimental results of § 3, an interpretation is offered in § 4, to account for the recovery phenomenon and the self-reversal phenomenon of a remanent magnetization of magnetite.

2. Description of samples studied and experimental procedure

Samples; The samples studied here are as follows.

(a) Single crystal of magnetite collected at Mitsuishi, Hokkaido, Japan. The chemical composition of this sample is shown below.

Fe ₂ O ₃	50.5 mol%
FeO	49.5
TiO ₂	0.5
total	100.5

The lattice parameter, the Curie point and the saturation magnetic moment are $a=8.391$ Å, $\theta=565^{\circ}\text{C}$ and $\sigma=95$ emu/g respectively. These data indicate that the sample is nearly a stoichiometric magnetite.

(b) Sixteen single crystals of magnetite from various places, Japan.

All of them were examined by means of X-ray analysis and they were found to be almost pure magnetite.

(c) Synthetic magnetite powder.

The specimen was prepared by sintering fine powder of mixture of pure hematite and electrolytic iron in an assigned proportion which were previously sealed in a quartz tube in vacuum of approximately 10^{-8} torr. Thus prepared sample was then quenched from 1150°C after being maintained at this temperature for 15 hours.

Composition of the sample thus prepared was examined by means of chemical and X-ray analyses.

(d) Cobalt chips.

Cobalt chips were prepared by lathing a cobalt metal bar.

Measurement procedure:

The measurement of the remanent magnetization for low temperatures was carried out with an astatic magnetometer and a ballistic method. For a high temperature measurement a ballistic method was used.

All temperature treatment were carried out in non-magnetic space in order to avoid possible effect of inverse TRM which might be produced in geomagnetic field in the heating.

3. Experimental results on self-reversal and recovery phenomena of a remanent magnetization of magnetite.

It is usually observed that the intensity of a remanent magnetization increases when a ferromagnetic body is cooled in non-magnetic space owing to the increase of the spontaneous magnetization. In the case of some iron oxides, however, anomalous behaviour is observed. For example, $\alpha\text{Fe}_2\text{O}_3$ has a transition point at about -15°C where the remanent magnetization almost vanishes in cooling from room temperature and reappears in heating to the room temperature from the low temperature (memory phenomenon). The recovered remanent magnetization has the same direction as that of the initial remanent magnetization. The mechanism of the transition and the "memory phenomenon" for hematite have been extensively studied (Tasaki, 1962; Iwata, 1963).

Recently, it is found (M. Yama-ai, 1962) that magnetite and titanomagnetites also show similar anomalous behaviours at low temperatures. When magnetite or titanomagnetites are cooled in non-magnetic space from room temperature to liquid nitrogen temperature, the remanent magnetizations (IRM, TRM) decrease sharply. In a heating process from liquid nitrogen temperature to the room temperature, the remanent magnetization once largely reduced at the low temperature again increases to recover a certain percentage of the original intensity of the remanent magnetization. In Fig. 1-a, the typical example of the recovery phenomenon of IRM ($H=200\text{ oe}$) produced at room temperature is shown for synthesized magnetite grain assemblage. In some cases, however, the reduced remanent magnetization at the low temperatures further decreases in the heating process.

The latter example is shown in Fig. 1-b.

In some cases, the direction of the remanent magnetization of several single crystal magnetites produced at room temperature was reversed at about -145°C in a cooling

Fig. 1-a Temperature variation of IRM ($H=200$ oe) of synthetic magnetite grain assemblage (original sample)

Fig. 1-b Temperature variation of IRM ($H=63$ oe) of synthetic magnetite grain assemblage (heat-treated in vacuum at 600°C for 1 hour).

process in non-magnetic field. When the sample is heated to the room temperature, the once reversed remanent magnetization at the low temperature recovers to the original direction with a certain percentage of the initial intensity of the remanent magnetization. A typical example of the self-reversal phenomenon of IRM ($H=5000$ oe) produced at room temperature is shown in Fig. 2 for the case of a Mitsuishi magnetite single crystal disk.

The experimental results on the self-reversal phenomenon and the recovery phenomenon are summarized below. For experiment (1)–(10) the Mitsuishi magnetite single crystal was used and for the experiment (11) other magnetites were studied.

Experiment (1)

A disk of single crystal magnetite (diameter = 1.3 cm, thickness = 0.3 cm) was magnetized in a magnetic field of $H_{ex}=5000$ oe at room temperature in the disk plane. The sample was cooled to -196°C in non-magnetic space and subsequently heated to the room temperature. Fig. 2 shows the change of the intensity of the remanent magnetization for this temperature range. As seen in the figure, the remanent magnetization once vanishes at about -145°C and then re-appears into the opposite direction in further cooling, namely, the direction of the remanent magnetization is reversed at -145°C in a cooling process in non-magnetic space. When the magnetite is heated to the room temperature in non-magnetic space, the direction of the remanent magnetization is again reversed to the original one at the same

Fig. 2 Temperature variation of IRM produced at $H=5000$ oe along $[100]$ axis of single crystal magnetite from Mitsuishi, Japan.

critical temperature where the direction change of the remanent magnetization took place in the cooling.

The intensity of the remanent magnetization is reduced to about one third of the original one after the heating to the room temperature. For the remanent magnetization which previously cooled and then heated in non-magnetic space, the temperature change of the remanent magnetization proceeds almost reversibly for cooling and heating in non-magnetic space. There observed no systematic correlation between the self-reversal phenomenon and the direction in the magnetite disk along which the initial remanent magnetization was produced.

Experiment (2)

The single crystal magnetite disk was magnetized at room temperature and then cooled to -196°C in the presence of various intensity of magnetic field of which direction is parallel to that of the original remanent magnetization. In Fig. 3, the intensity of the remanent magnetization at -196°C was plotted against the external field. As seen in the figure, the reversal phenomenon is found to be significantly affected even by the presence of a weak magnetic field comparable to the geomagnetic field. The magnetic field of 0.5 oe is sufficient to suppress the self-reversal phenomenon.

Experiment (3)

Temperature variation of IRMs produced in various magnetic field were examined from room temperature to liquid nitrogen temperature. In Fig. 4 the intensity of the recovered magnetization at room temperature and the intensity of the reversed magnetization at -196°C are plotted against the initial intensity of the remanent magnetization. Both the degree of the recovery and the reversal is enhanced with increasing intensity of the initial remanent magnetization.

Experiment (4)

Total TRM produced in the geomagnetic field was studied. No reversal phenomenon

Fig. 3 The relationship between the value of the IRM at -196°C and the intensity of the magnetic field in which the sample was cooled. It is seen from the figure that the magnetic field of 0.5 oe is sufficient to suppress the self-reversal phenomenon.

Fig. 4 The relationship between the intensity of the initial IRM and that of the recovered and reversed IRMs. Both the degree of the recovery and the reversal is enhanced with increasing intensity of the initial remanent magnetization.

was observed. However, it must be taken for granted that the process to produce the TRM, or the high temperature treatment might have brought in the change of the physical or chemical state of the sample to cause the disappearance of the self-reversal phenomenon. This point will be further discussed in the following section.

Experiment (5)

The effects of the shape of the sample on the reversal and the recovery phenomenon were examined. A single crystal magnetite was cut into a flat rectangular of various dimension ratio (thickness = 0.3 cm, width = $a = 0.8$ cm, length = b). Then the sample was magnetized along the length of the rectangular at $H = 5000$ oe. The temperature change of the IRM thus produced was measured. The ratio of the intensity of the remanent magnetization at -196°C to that at the room temperature is plotted against to the dimension ratio b/a of the sample in Fig. 5. It is found that the reversal phenomenon is critically dependent on the shape of the sample; that is, the shorter the sample, the more is pronounced the self-reversal phenomenon. When the sample is long enough, the reversal phenomenon does not take place, the remanent magnetization decreasing without changing the sign on the cooling process.

The remanent magnetization reduced at the low temperature recovers a certain percent of the original intensity when heated to the room temperature.

Experiment (6)

Temperature variation of inverse TRM (remanent magnetization produced in a heating process from -150°C to room temperature in a magnetic field) is similar to that of IRM;

Fig. 6 Temperature variation of inverse TRM, (ITRM)
r.t.
-150°C, 0.6 oe

Fig. 5 The relationship between the shape of the sample and the reversal phenomenon. The shorter the sample, the more pronounced the reversal phenomenon.

Fig. 7 Temperature variation of partial TRM, (PTRM)
-196°C
150°C, 0.6 oe

ITRM once vanishes at about -150°C and reappears in the opposite direction on the further cooling. (see Fig. 6) When the sample is heated to room temperature from -196°C the reversed ITRM is again reversed to the original direction at about -150°C .

Experiment (7)

Partial TRM which is produced in the cooling process from -150°C to -196°C in a magnetic field is reversed at -150°C and is again recovered at the same temperature. (see Fig. 7)

Experiment (8)

In order to examine the nature of the recovered remanent magnetization, an a - c decay mode of the recovered remanent magnetization is compared with that of the original one. Fig. 8-a and 8-b show that the remanent magnetization after the recovery is magnetically much stronger than the original one, or the remanent magnetization becomes hard after the cooling to -196°C in non-magnetic space, as if a soft component of the remanent magnetization were washed off by the low temperature treatment.

Experiment (9)

The relationship between the grain size and self-reversal phenomenon was examined. The single crystal magnetite was pulverized and two batches of magnetite powder of different grain size were prepared.

These are,

- single crystal of magnetite: dimension about 0.7 cm.
- magnetite powder prepared by pulverizing the single crystal magnetite (sample (a)): grain size about 1 mm.
- magnetite powder prepared by pulverizing the single crystal magnetite (sample (b)): grain size about 10μ —determined microscopically.

Fig. 8-a A-c decay mode of the original Irm and of the recovered one.

Fig. 8-b A-c decay mode of the original Irm and of the recovered one (in a normalized scale). A remanent magnetization becomes harder after cooled to liquid nitrogen temperature and heated to room temperature in nonmagnetic space.

The self-reversal is most conspicuous in the case of (a) and very weak in the case of (b) though it is still noticeable. The sample (c) did not show the self-reversal phenomenon.

Experiment (10)

Attempts were made to find if there is any relationship between the self-reversal phenomenon and the physical or the chemical state of the magnetite samples (Mitsuishi single crystal magnetite and other natural magnetites), such as bulk coercivity, chemical composition, Curie point, saturation magnetic moment, lattice parameter, microscopic and electron-microscopic observation. Except the bulk coercivity there observed no distinct relation between the self-reversal phenomenon and other physical or chemical properties. Even for the samples which have the same Curie point, saturation magnetic moment and lattice parameter, one show a self-reversal phenomenon and the other does not. However, there observed a distinct relation between the self-reversal phenomenon and the coercivity of the sample. This situation is represented in Fig. 9. If the sample is magnetically hard, the reversal phenomenon does not take place.

Experiment (11)

Other magnetites were also examined whether these magnetites show the self-reversal phenomenon.

The results are summarized below.

- (a) synthetic magnetite powder (grain size $\sim 10\mu$): No reversal phenomenon was observed.
- (b) natural single crystal of magnetite ($d=1\sim 0.2\text{cm}$): Sixteen natural single crystal (irregular shaped) from sixteen different locations were examined.

Three of them showed the self-reversal phenomenon. There appears, however, no clear relationship between the self-reversal phenomenon and the chemical composition or X-ray data of the samples. This fact again supports the view that the self-reversal phenomenon is essentially dependent on the physical state of the sample but not on the intrinsic properties such as chemical composition or lattice parameter.

Summarizing the above experimental facts, it may be said for any magnetite sample that the remanent magnetization decreases markedly at low temperatures and recovers or decreases when heated to room temperature. In some cases, the remanent magnetization reverses its direction at low temperature when cooled in non-magnetic space. The self-reversal phenomenon appears to be related to the physical state of the magnetite rather than to the intrinsic properties (Exp. (9), Exp. (10) and Exp. (11)). The mechanism of the recovery and the self-reversal phenomenon will be discussed in the following section.

Fig. 9 The relationship between the bulk coercivity of the sample and the reversal phenomenon.

4. Discussion

The phenomenon that the intensity of a remanent magnetization of magnetite decreases in cooling from room temperature may be explained by taking into account of the sharp decrease of the bulk coercivity caused by the decreasing crystal anisotropy energy at low temperatures (Ozima, Yama-ai and Nagata, 1963). The temperature change of the crystal anisotropy energy and the coercivity of magnetite are shown in Fig. 10. The decreasing coercivity will loosen the domain configuration to bring in the marked decrease of remanent magnetization when magnetite is cooled in non-magnetic space.

The experimental result (Exp. (8)) that the still remaining component of the remanent magnetization after the cooling (cooled to liquid nitrogen temperature and heated to room temperature in non-magnetic space) is magnetically much harder than that of the original remanent magnetization suggests that a remanent magnetization of magnetite consists of two kinds of component; a component whose coercivity is essentially determined by the crystal anisotropy energy and then susceptible to the low temperature demagnetization, and another whose coercivity is determined by other factors than crystal anisotropy energy and insensitive to the low temperature demagnetization.

In a previous paper (Yama-ai, Ozima and Nagata, 1963) we suggested that a magnetostatic interaction between the above two components (crystal anisotropy dependent and crystal anisotropy independent components) is responsible for the self-reversal and the recovery phenomena of magnetite at low temperatures. However, the experimental results (6) and (7) seem to rule out this possibility, since there still exist the recovery and reversal phenomena in the case of inverse TRM and the partial TRM produced by field-cooling from -150°C to -196°C , which are controlled solely by the crystal anisotropy energy and in whose acquisition the crystal anisotropy independent component does not take part.

In the light of these new experimental facts, it is here assumed that the magnetostatic interaction exists within the crystal anisotropy dependent component. Because of micro-

Fig. 10-a Temperature variation of crystalline anisotropy constants K_1 and K_2 of magnetite (after Bickford et. al., 1957)

Fig. 10-b Temperature variation of bulk coercivity of artificial magnetite grain (grain size $\sim 10\mu$).

scopic heterogeneity in the stress distribution or in other physical conditions within a magnetite grain, isotropic temperature (temperature at which the crystal anisotropy energy becomes minimum) may vary from points to points within a magnetite grain. Regions with different isotropic temperatures may interact magnetostatically with each other. For the simplicity, consider that the crystal anisotropy dependent component consists of two region A and B with the isotropic temperature T_1 and T_2 ($T_1 > T_2$), and they are magnetostatically coupled. The magnetic field acting on A due to the remanent magnetization of B would then be expressed as

$$H_B = -(D_A - \bar{D}_B)c_B J_B, \quad (1)$$

where D_A is the demagnetizing factor of the bulk specimen, $\bar{D}_B$ is the mean demagnetizing factor of B regions, c_B is a volume fraction of B regions and J_B is the intensity of the remanent magnetization of B . The interaction between A and B then would be either positive or negative depending on $D_A < \bar{D}_B$ or $D_A > \bar{D}_B$.

First consider that the interaction is negative. When magnetite is cooled from room temperature in non-magnetic space, A -region would then come to equilibrium with the ambient magnetic field, that is, the demagnetizing field of the persistent magnetization of B region at $T = T_1$. In the further cooling from T_1 , the magnetization of A will be fixed, as the coercivity of A -region increases with the decreasing temperature (Ozima, Yama-ai and Nagata, 1963). At $T = T_2$, the region B will be magnetized in the influence of the demagnetizing field of the A -region. Fig. 11-a and Fig. 11-b show schematically the change of the magnetizations of A and B regions with decreasing temperature. The total magnetization which is the algebraic sum of the magnetization A and B , would then change as shown in Fig. 11-c. Depending on the relative intensity of the remanetized A -region

Fig. 11-a Schematic representation of the temperature variation of the magnetization of A -region of crystal anisotropy dependent component of magnetite (negative interaction case).

Fig. 11-b Schematic representation of the temperature variation of the magnetization of B -region of crystal anisotropy dependent component of magnetite.

Fig. 11-c Schematic representation of the temperature variation of the total remanent magnetization of crystal anisotropy dependent component.

under the influence of the demagnetizing field of B -region to those of B -region and the crystal anisotropy independent component, the resultant magnetization at the low temperature may be positive or negative (self-reversal). In heating the magnetite, the magnetizations of A and B will change reversibly until T_2 . At $T=T_1$, the A region again comes to equilibrium with the ambient field, that is, the demagnetizing field due to B -regions. Since the magnetization of B at T_1 in the heating process is smaller than that at the same temperature in the previous cooling process, the magnetization of A above T_1 will be larger than that below T_1 (weaker negative interaction). The broken line in Fig. 11-a and Fig. 11-b show schematically the variation of the magnetization of A and B in a heating process. The net change of the magnetization would then as shown in Fig. 11-c by a broken line, which indicates the "recovery phenomenon". In the above discussion the magnetic field due to the crystal anisotropy dependent component is neglected. However, as the magnetic field due to the crystal anisotropy independent component is constant throughout the whole temperature range considered here, its effect is only to contribute a constant magnetization and it would not change the temperature variation "mode" of the remanent magnetization.

If the interaction is positive, the similar consideration shows that the variation mode of a remanent magnetization is of a type shown in Fig. 1-b.

The experiments (2) and (5) seem to support the above assumption of the magnetostatic interaction. The result of the experiment (2) shows that the interaction between the two regions should be of the order of one-oersted. This weak interaction seems to rule out the possibility of the exchange interaction observed in Haruna-type self-reversal (Uyeda, 1958) and support the assumption of the magnetostatic interaction. As shown in Eq. (1), the demagnetizing field which is responsible for the reversal phenomenon is proportional to $(D_A - \overline{D}_B)$. Hence, it is expected that the larger bulk demagnetizing factor would favour the self-reversal of a remanent magnetization. Exp (5) shows that as the sample is shortened, the demagnetizing factor D_A increases to result in larger demagnetizing field approaching to the condition to cause the self-reversal phenomenon.

Experiment (4) may be explained in the following way. The heat-treatment causes a considerable change in the relative amount of the two regions. Hence, it is not unreasonable to expect that the interaction between the two regions would be different from that for the original sample causing the disappearance of the self-reversal phenomenon.

Based on these experimental results, it may be safe to generalize that both the self-reversal and the recovery phenomena are caused by a magnetostatic interaction among the crystal anisotropy dependent component with different isotropic points.

In this connection, it is interesting to note that cobalt shows the self-reversal and recovery phenomena. Cobalt is known to have crystalline anisotropy constant which vanishes at about 250°C (Honda and Masumoto, 1931). In Fig. 12, the temperature change of the crystalline anisotropy energy of cobalt which is taken from the book by Bozorth and that of susceptibility are shown. From the foregoing discussions, it may be expected that IRM acquired by the cobalt chips at the temperature higher than 250°C would show the self-reversal phenomenon or at least the recovery phenomenon when it is cooled to a

Fig. 12-a Temperature variation of crystalline anisotropy constants K_1 and K_2 of cobalt (after Bozorth, 1951).

Fig. 12-b Temperature variation of susceptibility of cobalt chips measured at $H=10$ oe.

room temperature and afterwards heated in non-magnetic space. It is shown in Fig. 13 that the remanent magnetization produced at 400°C vanishes at 100°C and reverses its direction in the further cooling. In heating it from the room temperature to 400°C , the remanent magnetization once reversed at 100°C vanishes at 100°C and re-appears in the same direction as the initial remanent magnetization in the further heating.

5. Conclusion

It is found that a remanent magnetization (IRM, TRM) of magnetite sharply decreases when the magnetite is cooled from room temperature to liquid nitrogen temperature in non-magnetic space. In some cases the remanent magnetization reverses the direction at low temperatures. In heating from the low temperature to the room temperature, the once largely reduced or reversed remanent magnetization recovers a certain percentage of the intensity of the initial remanent magnetization. In some cases, the once reduced remanent magnetization at low temperatures further decreases in heating to the room temperature.

The anomalous behaviours may be interpreted that the diminishing anisotropy energy loosens some of the domains, which are subsequently re-magnetized in the further cooling in the opposite direction (negative interaction case) or in the same direction (positive interaction case) to that of the initial magnetization under the influence of the magnetic field due to the domains with lower isotropic temperatures. If the re-magnetized components in the opposite direction to that of the initial remanent magnetization overcomes

Fig. 13 Temperature variation of IRM of cobalt chips produced at 400°C in $H=950$ oe.

the intensity of the undisturbed persistent domains the self-reversal phenomenon may be observed. In heating from the low temperature to the room temperature, the reversely re-magnetized domains again will be partially demagnetized resulting in the recovery phenomenon. It is assumed here that the heterogeneity in the stress-distribution or in other physical conditions within a grain would result in heterogenic distribution of isotropic points within a grain. The regions with different isotropic points then would interact magnetostatically with each other resulting in the self-reversal or the recovery phenomenon.

Cobalt shows the similar recovery and reversal phenomena of a remanent magnetization, when it is cooled from 400°C to room temperature in non-magnetic space. Since cobalt has a zero anisotropic point (isotropic point) at about 250°C, the self-reversal and the recovery phenomena may be explained in the same way as in magnetite.

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