PLANETARY SCIENCE

Ferromagnetism and exchange bias in compressed ilmenite-hematite solid solution as a source of planetary magnetic anomalies

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We propose a compressed ilmenite-hematite solid solution as a new potential source of Earth's magnetic anomalies. The $0.5FeTiO_3 \cdot 0.5Fe_2O_3$ solid solution compressed by collision synthesis with super-high-energy ball milling showed a decrease in molar volume of approximately 1.8%. Consequently, the sample showed a saturation magnetization of 1.5 ampere square meter per kilogram (Am^2/kg) at 300 kelvin, a Curie temperature of 990 kelvin, and a magnetic exchange bias below 100 kelvin, e.g., 1.7×10^5 ampere per meter at 60 kelvin. Ilmenite-hematite solid solutions are common mineral systems in most mafic igneous and metamorphic rocks, and the compressive force in the rocks is generated by the high pressure in the upper mantle or by shock events with high pressure such as the collision of these rocks with meteorites. Therefore, we consider that the compressed ilmenite-hematite solid solution is an additional candidate source of other planetary magnetic anomalies including those in the Moon and Earth.

INTRODUCTION

Earth's magnetic anomalies are commonly attributed to the presence of magnetite, hematite, titanomagnetite, and pyrrhotite minerals. Magnetite (Fe_3O_4) shows strong ferromagnetism, and hematite (α-Fe_2O_3) shows antiferromagnetism with superimposed weak ferromagnetism (*1*). Ilmenite ($FeTiO_3$) is a common mineral in nature and usually shows solid solutions toward α-Fe_2O_3; therefore, ilmenite-hematite (IH) solid solutions exist in most mafic igneous and metamorphic rocks. The intermediate members of the IH mineral series have strong ferrimagnetism (*2*), which is preserved only in rapidly cooled volcanic rocks. In addition, the IH natural mineral intergrowth series usually has large and stable magnetism, the so-called lamellar magnetism (*3*), formed through a natural process of phase separation during slow cooling over millions of years. Despite extensive advances in the study of this series including the recent investigations of lamellar magnetism (*4*–*7*), understanding the full nature of magnetism in the minerals and intergrowths in the IH solid solution series remains a great challenge. In this study, we provide experimental evidence of a new type of ferromagnetism with high Curie temperature and a large magnetic exchange bias in the compressed IH solid solution. These magnetic properties are of crucial importance in the study of planetary magnetic anomalies.

RESULTS

Ferromagnetism in compressed IH solid solution

The compressed IH solid solution was prepared by super-high-energy ball milling (*8*–*11*). The $0.5FeTiO_3 \cdot 0.5Fe_2O_3$ solid solution sample shows a decrease in molar volume of approximately 1.8%, which corresponds to that of a single crystal of $FeTiO_3$ under a high pressure of 5 GPa at 300 K. Consequently, the oxidation state in $FeTiO_3$ changes to almost $Fe^{3+}Ti^{3+}$ from $Fe^{2+}Ti^{4+}$, resulting in the emergence of ferromagnetism (fig. S1 and table S1). The details were described in our previous report (*11*).

Figure 1A shows the magnetization M as a function of the applied magnetic field H at 300 K. The sample showed a mixed magnetic behavior between ferromagnetism and paramagnetism. The saturation magnetization M_s estimated from the straight line extrapolated to H of 0 A/m was approximately 1.5 Am^2/kg. This value is three times higher than that for antiferromagnetic hematite with weak ferromagnetism, e.g., 0.5 Am^2/kg (*1*) but lower than those of strongly ferromagnetic magnetite (*1*) and ferrimagnetic IH solid solution series (*2*). The saturation remanent magnetization M_r and coercivity H_c of the sample at 300 K were 0.3 Am^2/kg and 24×10^3 A/m, respectively (Fig. 1B). We measured the magnetization of the sample at 100 and 5 K (fig. S2), and the M_s, M_r, and H_c values are plotted in Fig. 1C. The slope of M_s versus temperature for the sample is gentler than those for the synthetic IH solid solutions without lattice compression (*12*–*15*). Therefore, we consider that the ferromagnetism in the compressed IH solid solution sample is not related to conventional ferrimagnetism in the IH solid solution series.

High Curie temperature

Figure 2 shows the temperature dependence of magnetization at 4×10^5 A/m from room temperature to 1073 K. We found that the Curie temperature T_c of the compressed IH solid solution sample is 990 K. This value is much higher than that (500 K) for the synthetic $0.5FeTiO_3 \cdot 0.5Fe_2O_3$ solid solution without lattice compression (*12*, *13*), hence increasing the depth range in Earth's mantle across which this new type of magnetism would apply. Therefore, we propose that the ferromagnetism in the compressed IH solid solution is a new type of magnetism, and it is different from conventional ferrimagnetism. Notably, the T_c of the sample is higher than 843 to 853 K for strongly ferromagnetic magnetite and 943 K for weakly magnetic hematite (*1*). It is also higher than the unblocking temperature of 923 to 940 K for the lamellar magnetism in IH natural mineral intergrowth (*16*, *17*).

Large magnetic exchange bias

We consider that the ilmenite component acts as the ferromagnetic phase and the hematite component acts as the antiferromagnetic

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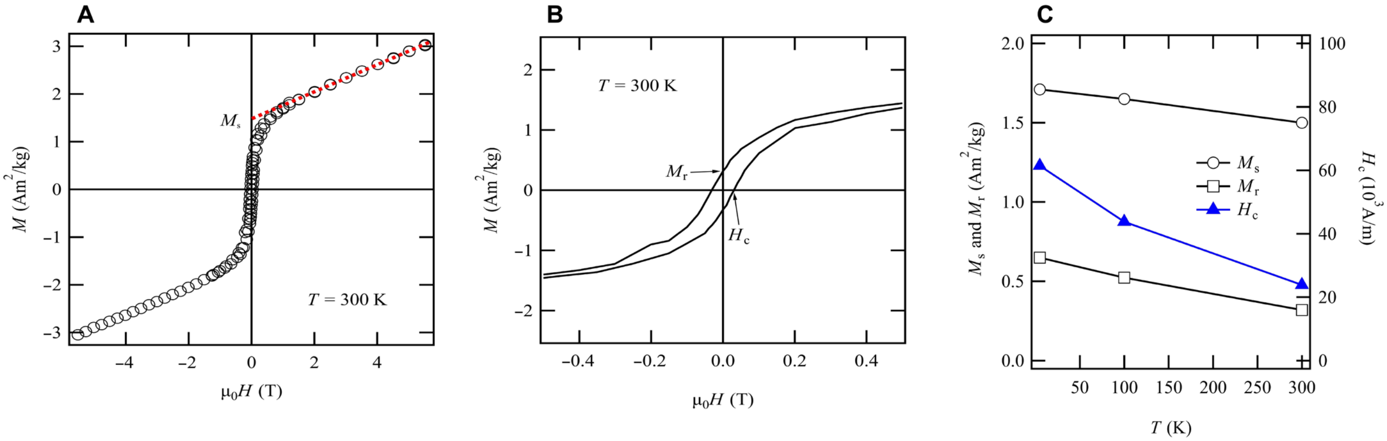

Fig. 1. Magnetic properties of compressed $0.5FeTiO_3{\cdot}0.5Fe_2O_3$ sample. (**A**) Magnetization measured at 300 K. (**B**) Hysteresis data for field strengths up to 3.5 × 10^5 A/m. (**C**) Temperature dependence of saturation magnetization M_s, remanent magnetization M_r, and coercivity H_c. M_s/M_r values are 2.63, 3.15, and 4.70 at 5, 100, and 300 K, respectively.

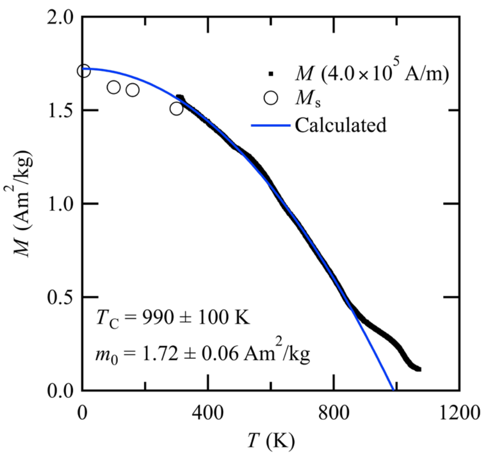

Fig. 2. Magnetic properties of compressed $0.5FeTiO_3{\cdot}0.5Fe_2O_3$ sample at high temperature. The magnetization was measured under a magnetic field of 4 × 10^5 A/m in vacuum. The Curie temperature T_c is approximately 990 K.

phase in the compressed $0.5FeTiO_3{\cdot}0.5Fe_2O_3$ sample. The interface between the ferromagnetic and antiferromagnetic phases is to be expected for the generation of magnetic exchange bias, which is a phenomenon whereby the hysteresis loop of a soft magnetic phase is shifted by an amount H_E along the applied field axis owing to its interaction with a hard magnetic phase. Figure 3 shows the hysteresis loops measured at 5 K after zero-field cooling with different room temperature pretreatments. Before zero-field cooling, the examined samples were pretreated at 300 K by applying a magnetic field H of +44 or −44 × 10^5 A/m to establish positive or negative remanent, respectively. It is found that the two hysteresis loops are shifted by the positive (Fig. 3A) and negative (Fig. 3B) remanent treatments. This is the second observation of evident exchange bias in synthetic IH solid solution samples (*18*), although it has been observed in the lamellar magnetism in natural IH minerals (*5–7*, *19–23*). Figure 3C shows the difference between the upper and lower hysteresis branches (Fig. 3, A and B).

We measured the hysteresis loops from 5 to 160 K (fig. S3) to quantify H_E (fig. S4), and their values are plotted in Fig. 3D. The estimated shift of the loop center indicated an exchange bias (H_E) of approximately 1.7 × 10^5 A/m at 60 K. This large value is comparable to that for the lamellar magnetism at 45 K in natural mineral intergrowth with the most-fine nanometer-scale lamellae (*23*), and it is orders of magnitude larger than those in any other natural mineral intergrowths (*5*, *19–22*). In addition, it is found that the compressed IH solid solution showed H_E below 100 K, which is higher than the temperatures for the lamellar magnetism in natural mineral intergrowths (*5*, *23*). In these cases, H_E was observed below a Néel temperature of ilmenite lamellae, e.g., T_N of 57 K, because H_E is due to the interface between the ferrimagnetism in the contact layers of the titanohematite and the antiferromagnetism in the ilmenite lamellae (*23*). On the other hand, we consider that the two phases responsible for H_E in the compressed IH solid solution are the ferromagnetism in the ilmenite component acting as the soft magnetic phase and the antiferromagnetism in the hematite component acting as the hard magnetic phase, i.e., coupling between the compressed ilmenite and hematite components.

Then, we suppose that these two phases in the compressed IH sample contribute to the generation of self-reversed thermoremanent magnetization (SR-TRM), which was found in volcanic rocks from Haruna (*24*, *25*) and attributed to intermediate members of the IH solid solution series (*12*). Although the mechanism of SR-TRM in IH minerals remains poorly understood, it is considered to require the presence of two phases, namely, a strongly magnetic stable phase and a weakly magnetic metastable phase (*26*). We consider that the compressed IH solid solution may be potentially useful for the generation of SR-TRM induced by the coupling between the ferromagnetic phase of the ilmenite component and the antiferromagnetic phase with the superimposed weak ferromagnetism of the hematite component.

DISCUSSION

To confirm a new type of ferromagnetism with high T_c in the compressed IH solid solution, we released the lattice compression in the sample by heat treatment at 1273 K in vacuum. We found that the peak position for the sample after heat treatment was clearly shifted to a d-spacing higher than that for the compressed sample (fig. S5).

This shift in peak position suggests that the lattice compression in the IH solid solution can be released by heat treatment. Although the heat-treated sample showed a hysteresis loop (Fig. 4A), its T_c changed to 615 K (Fig. 4B). This temperature is much lower than 990 K for the compressed sample, and it is consistent with the conventional ferrimagnetism in the IH solid solutions without lattice compression (*12*, *13*). Moreover, the heat-treated sample showed no shift of the hysteresis loop at different room temperature pretreatments, i.e., no H_E (fig. S6). Therefore, we conclude that the ferromagnetism with high T_c in the compressed IH sample is a previously unknown magnetism,

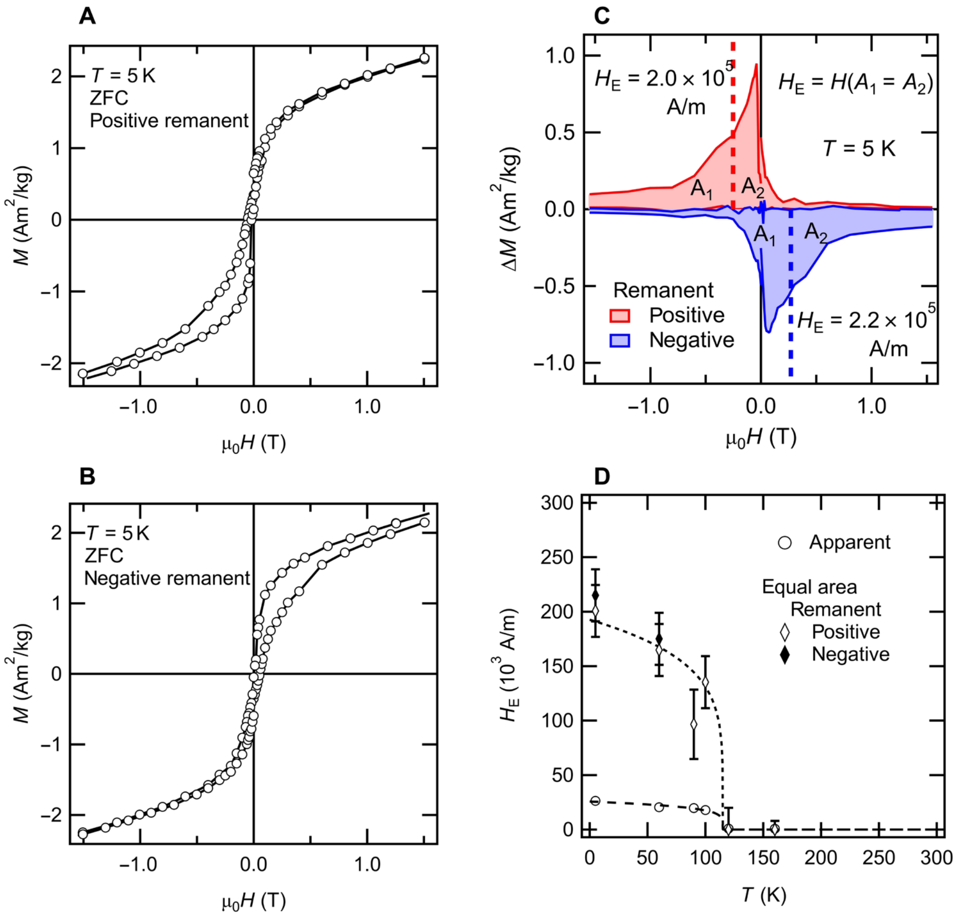

Fig. 3. Hysteresis loops measured at 5 K after zero-field cooling with different pretreatments. (**A**) Pretreatment with positive room temperature remanence. (**B**) Pretreatment with negative room temperature remanence. (**C**) The lower hysteresis branch is subtracted from the upper branch in the case of negative exchange bias. The estimated shift of the loop center (dashed line) indicates an exchange bias. (**D**) Temperature dependence of exchange bias H_E. ZFC, zero-field cooling.

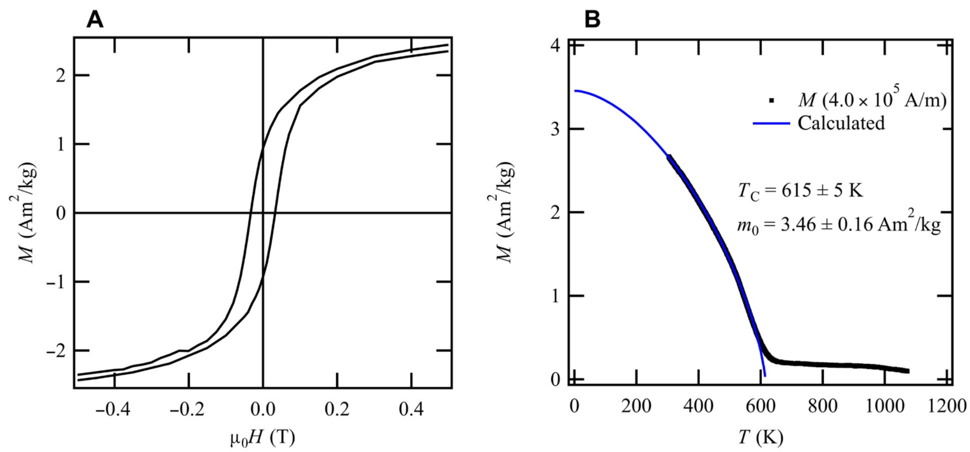

Fig. 4. Magnetic properties of heat-treated $0.5FeTiO_3{\cdot}0.5Fe_2O_3$ sample. (**A**) Hysteresis data measured at 300 K. (**B**) Magnetization was measured under a magnetic field of 4×10^5 A/m in the temperature range from 300 to 1073 K in vacuum.

and it clearly differs from the conventional ferrimagnetism in IH solid solutions series.

Overall, our results suggest that the compressed IH solid solution is responsible for the source of Earth's magnetic anomalies. Members of the IH solid solution series are common minerals in nature, existing as an accessory phase in most mafic igneous and metamorphic rocks. For example, they are derived from the upper mantle down to depths of approximately 400 km, and thus, with pressures of 12 to 13 GPa (*27*), their compressed state is obtained. The hydrostatic and isotropic pressure is formed in the mantle, leading to thermodynamic equilibrium. In the contrary, the nonhydrostatic pressure occurs between the balls uniaxially in the ball milling system. However, we consider that the thermodynamic state is obtained partially in the compressed IH solid solution prepared by super-high-energy ball milling, because some thermodynamic effects of nonhydrostatic pressure on the magnetic properties in magnetite are derived (*28*). Then, the compression rate of the sample is approximately 1.8%, which corresponds to that of a single crystal of trigonal $FeTiO_3$ under a high pressure of approximately 5 GPa at 300 K generated using a diamond anvil cell (*29*). It is reported that a pressure of 5 GPa occurs at a depth of 160 km, where the temperature is approximately 1073 K (*30*). Although this value is slightly higher than the T_c of the compressed sample at an ordinary pressure, it is reported that the T_c of magnetite increases with pressure (*31*, *32*). Therefore, we predict that the compressed sample shows the ferromagnetism at 5 GPa and 1073 K. On the basis of these findings, we suggest that some compressed IH mineral series at depths in the upper mantle contribute to in situ Earth's magnetic anomalies.

The magnetic anomalies are also observed on the Moon and Mars. It is known that both celestial bodies experienced shock events with high pressure upon collision with meteorites, such that intense compressive forces were added to those collision sites. We consider that the uniaxial pressure by collision with meteorites is similar to that by collision synthesis with super-high-energy ball milling. It is reported that geikielite-rich ilmenite rocks are important for the Moon's anomalies (*33*). It is considered that the anomalies in Mars require very large rock volumes over areas of hundreds of square kilometers and extending to depths of 20 to 30 km (*34*, *35*), where ilmenite exists as an accessory mineral. By taking together these findings and our results, we predict that the rocks containing the compressed ilmenite series are potential candidate sources of other planetary magnetic anomalies including those in the Moon and Earth.

Last, we discuss the relationships between the ferromagnetism in the compressed IH solid solution and the lamellar magnetism in the natural mineral intergrowth (*3*–*5*). It is considered that the lamellar magnetism is responsible for the unusually large and stable remanent magnetization in rocks. For example, it is observed in grains of titanohematite ($FeTiO_3$ bearing Fe_2O_3) that crystallized and phase-separated (exsolved) nearly 1 billion years ago (*23*). This mineral contains abundant nanoscale exsolution lamellae of ilmenite, and ferrimagnetism occurs at the interface between the paramagnetic ilmenite lamellae and the antiferromagnetic titanohematite host. At present, we assume that the compressive force is generated in the contact layers of the ilmenite lamellae by the heterogeneous interface between the ilmenite lamellae and the titanohematite host, i.e., their lattice mismatch. We believe that the ferromagnetism reported in this study could occur in the compressed contact layers of the ilmenite lamellae and that the charge ordering in contact layers of lamellar magnetism may be influenced by the charge transfer from Fe^{2+} to Fe^{3+} in $FeTiO_3$ with high pressure.

MATERIALS AND METHODS

Hysteresis loop measurements

We measured the magnetic properties using a conventional superconducting quantum interference device magnetometer (MPMS-XL, Quantum Design) under a magnetic field of up to 40×10^5 A/m in the temperature range from 5 to 300 K for dc magnetization.

Curie temperature measurements

We measured the magnetization using a conventional vibrating sample magnetometer (BHV-50H, Riken Denshi Co. Ltd.) under a magnetic field of 4×10^5 A/m in the temperature range from 300 to 1073 K in vacuum. Sample powder installed into a copper capsule was settled in a glass vacuum chamber. The Curie temperature T_c of the sample was determined by an empirical form with two exponents to reproduce the curve for the temperature dependence of magnetization below T_c.

X-ray diffraction measurements

Powder x-ray diffraction patterns were measured for the samples with Ni-filtered Cu Kα radiation (λ = 1.4506 Å).

SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at https://science.org/doi/10.1126/sciadv.abj2487

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