Structural, magnetic and electronic state characterization of L1$_0$-type ordered FeNi alloy extracted from a natural meteorite

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Received 21 July 2013, revised 21 October 2013
Published 27 January 2014

Abstract
To understand the hard magnetism of L1$_0$-type ordered FeNi alloy, we extracted the L1$_0$-FeNi phase from a natural meteorite, and evaluated its fundamental solid-state properties: sample composition, magnetic hysteresis, crystal structure and electronic structure. We executed multidirectional analyses using scanning electron microscopy with an electron probe micro-analyzer (SEM-EPMA), a superconducting quantum interference device (SQUID), x-ray diffraction (XRD) and magnetic circular dichroism (MCD). As a result, we found that the composition was Fe: 50.47 ± 1.98 at.%, Ni: 49.53 ± 1.49 at.%, and an obvious superlattice peak is confirmed. The estimated degree of order was 0.608, with lattice constants $a = b = 3.582$ Å and $c = 3.607$ Å. The obtained coercivity was more than 500 Oe. MCD analysis using the K absorption edge suggests that the magnetic anisotropy could originate from the orbital magnetic moment of 3d electrons in Fe; this result is consistent with that in a previous report obtained with synthetic L1$_0$-FeNi.

(Some figures may appear in colour only in the online journal)

1. Introduction
For the realization of a sustainable society, the development of rare-metal-free and functional material is strongly desired. The L1$_0$-type ferromagnet has been of great promise as a material for magnetic memories or permanent magnets because of its strong magnetic anisotropy and coercivity [1]. Meanwhile, typical L1$_0$ phases such as CoPt or FePt have used large quantities of rare metals to obtain strong magnetic anisotropy [1]. From the viewpoint of resource depletion and spiraling prices, there is a need for the introduction of a substitutable material. Here, we are looking into an L1$_0$-type ordered alloy consisting of Fe and Ni, which are both abundant and inexpensive. L1$_0$-FeNi is known to have high coercivity (500–4000 Oe) and magnetic anisotropy ($1.3 \times 10^7$ erg cm$^{-3}$), and its magnetic properties are comparable to those of CoPt [2–6]. Common disordered FeNi alloys, which usually have small coercivity and weak magnetic anisotropy, are classified as soft magnetic materials [7]. On the other hand, L1$_0$-FeNi has high coercivity and strong magnetic anisotropy, and it is classified as a hard magnetic material; therefore, shedding light on this phenomenon is a matter of great interest in the field of magnetic materials science.

L1$_0$-FeNi is described as a chemically ordered alloy with an fct structure consisting of Fe and Ni. It has a superstructure alternating monatomic layers of Fe and Ni...
along the c-axis direction. The phase diagram shows that FeNi is an all-proportional solid solution, and the L1₀ phase is exhibited as an equilibrium state below 320 °C [8, 9]. The formation process of natural L1₀-FeNi has been discussed by planetary scientists based on the metallurgy of the FeNi system [10, 11]. The inner core of the mother body consists primarily of a FeNi alloy, and it is thermally isolated by a silicate mantle. According to the FeNi phase diagram, long-range thermal diffusion encourages metamorphic evolution of FeNi from the γ₁ phase (the low-Ni paramagnetic fcc phase) to the γ₃ (L1₀-FeNi) phase; then L1₀-FeNi is formed by subsequent phase transitions at grain boundaries between α and γ₃ lamellae. To complete the entire equilibrium reaction, extremely slow cooling (4.6 billion years) is necessary due to the extremely slow Ni diffusion [2–4].

In a previous study on the synthesis of L1₀-FeNi, it was reported by Néel et al in the 1960s that the L1₀-FeNi ordered structure could be constructed by neutron irradiation at high temperature [5]. However, this approach is probably not an ideal choice for device applications. Recently, Shima et al demonstrated successful production of the synthesis of single-crystalline L1₀-FeNi by alternate monatomic layer deposition using molecular beam epitaxy [12]. Kojima et al reported that, for alternate atomically deposited FeNi, the magnetic anisotropy and degree of order are proportional, and they reported a magnetic anisotropy of 7.0 × 10⁶ erg cm⁻³ with degree of order of 0.48 [13–16]. Kotsugi et al analyzed the origin of the magnetic anisotropy of their sample using synchrotron radiation [17–20], and reported that the magnetic anisotropy arises from spin–orbit interaction in the 3d electrons of Fe [21]. Theoretical approaches were taken by Miura et al and Mitsumata et al, who reported on the relationship between magnetic anisotropy and lattice structure [22, 23]. However, the magnetic anisotropy of L1₀-FeNi has not reached the upper limit yet. There is still very much an unclear issue for the improvement of magnetic anisotropy; in particular, the fundamental physical properties of the L1₀-FeNi phase are not yet accurately clarified.

In this paper, we analyze natural L1₀-FeNi, which originally exhibits hard magnetism, and describe the complete set of fundamental physical properties – composition, magnetic hysteresis, structure, and electronic structure – of natural L1₀-FeNi, which has never been discussed. The results obtained are also compared with those in a previous report on synthetic L1₀-FeNi. These physical properties are essential for understanding the fact of magnetic anisotropy, because magnetic anisotropy is mainly correlated with spin–orbit interaction, and it is strongly associated with symmetry breaking of the crystal field through lattice distortion [24, 25]. In particular, the analysis of natural L1₀-FeNi from a materials science perspective has not been carried out yet. An accurate characterization of L1₀-FeNi will provide us with important information, and we aimed to obtain guidelines for improving the magnetic function of L1₀-FeNi.

To examine the L1₀-FeNi phase, we took measurements with an electron probe micro-analyzer (EPMA) and a superconducting quantum interference device (SQUID), and used synchrotron x-ray diffraction (XRD) and magnetic circular dichroism (MCD) to estimate the chemical composition, magnetic hysteresis, crystal structure and electronic structure, respectively [26–30].

EPMA can quantitatively estimate the chemical composition by measuring the energy and intensity of specific x-rays generated by electron beam irradiation. Since its electron lens is in common with that of a scanning electron microscope, it provides us with the metallographic fine structure of sample simultaneously. A SQUID was used to measure the magnetic hysteresis of L1₀-FeNi. It is operated by quantum effects in a Josephson junction to measure tiny magnetic fields in a sample. XRD can investigate the crystal structure with high accuracy. Advantages of synchrotron radiation-based XRD are high energy, high flux, and a sharp beam to achieve signal-to-noise ratios much higher than with laboratory-scale XRD. It thus enables us to investigate very weak diffraction peaks from minute quantities of sample material.

MCD spectroscopy is the differential x-ray absorption spectroscopy of left and right circular polarized x-ray, induced in a specimen by a magnetic field applied parallel or antiparallel to the light incidence. Since the photon energy of synchrotron radiation can be freely tunable, it is possible to acquire element-selective information. Analysis of MCD spectra provides us directly with the electronic structure of specific elements as an origin of the magnetic moment. In particular, at the K absorption edge, MCD can observe transitions from 1s to 4p, thus allowing the 4p magnetic moment to be studied. Polarization of the empty 4p level in Fe and Ni is not normally expected, but an MCD signal is observed due to hybridization with the 3d orbitals of neighboring atoms, which causes a small amount of polarization in 4p state too [30]. Accordingly, an important characteristic of this technique is that it is possible to obtain information about 3d electrons indirectly through MCD of the K absorption edge. Since the K-edge MCD technique can be applied to polycrystalline materials and the effects of surface oxidation can be ignored, it is suitable for the evaluation of our natural samples. Furthermore, natural L1₀-FeNi samples are obtained as flakes of approximately 1 µm in thickness, thus facilitating transmission measurements. It is therefore the ideal technique for the characterization of the natural L1₀-FeNi.

2. Sample preparation

We extracted natural L1₀-FeNi from the Toluca meteorite, which shows a clear Widmanstätten structure with wide lamellae. Long-range segregation is exhibited in this meteorite and sufficient L1₀-FeNi phase is included [8]. Samples were extracted by chemical etching in a 5% aqueous HCl solution for one week, and foil-like samples showing a metallic gloss were obtained. L1₀-FeNi is more resistant to acid than other FeNi phases, and can thus be extracted by selective etching. The shape and composition of the extracted flake samples were investigated using SEM-EPMA. SEM-EPMA was also used to eliminate unsuitable specimens with foreign impurities and inclusions. We also prepared disordered FeNi phase as a reference. The reference samples were produced by simple heating using an arc furnace and a muffle furnace. Raw materials of Fe and Ni were calibrated as 50 at.% to 50 at.%.
Figure 1. (a) SEM image of meteorite surface before etching. (b) SEM image of meteorite surface after etching for 3 min in a 5% aqueous solution of HCl. (c) SEM image of a flake extracted from the iron meteorite; the EPMA result gives the chemical composition Fe: $50.47 \pm 1.98$ at.% and Ni: $49.60 \pm 1.49$ at.%. (d) SQUID measurement showing a magnetic moment of $1100 \text{ emu cm}^{-3}$ and coercivity more than 500 Oe.

The specimen was first melted in an arc furnace, and annealed to $1050 \, ^\circ \mathrm{C}$ for one week in a muffle furnace; then the samples were allowed to cool naturally to room temperature.

3. Experimental results

Figure 1 shows the images obtained by SEM-EPMA. Figure 1(a) is an SEM image of the meteorite surface before sample etching. This is a metallurgical structure called a Widmanstätten structure, which is a typical microstructure in iron meteorites. The image shows the region of an interface between $\alpha$-FeNi and $\gamma$-FeNi lamellae. It is known that $\text{L}_1\text{O}$-FeNi generally exists segregated at the interfaces of $\alpha$ and $\gamma$ phases. Figure 1(b) shows an SEM image of the same region after etching for 3 min in a 5% aqueous solution of HCl. Although etching has progressed in the $\alpha$ and $\gamma$ phases, the interface region has not dissolved, and it remains. Other regions apart from $\text{L}_1\text{O}$-FeNi have been turned black by etching, so the selective nature of etching is readily apparent.

Next, after etching continuously for one week in aqueous HCl, we extracted the residual flakes with a metallic gloss. Figure 1(c) shows an SEM image of one such flake obtained in this way. Although there were still small impurities, we were able to extract samples of the order of millimeters in size, and we confirmed that these took the form of foil-like flakes.

Next, we used EPMA to quantitatively evaluate the composition ratio. Measurements were performed by selecting 23 points in a flat region with no inclusions on six flake samples. As a result, the composition was found to be Fe: $50.47 \pm 1.98$ at.%, Ni: $49.60 \pm 1.49$ at.%, and Co: $0.049 \pm 1.53$ at.%. Although the samples included a small amount of Co, we confirmed that the Fe and Ni were uniformly distributed with a composition ratio of almost 50:50.

Figure 1(d) shows an example of the magnetic hysteresis obtained with the SQUID. The magnetic field is applied in both the in-plane and out-of-plane directions of the flake. Shape anisotropy in the out-of-plane hysteresis loop was eliminated by assuming an infinite film here, because the typical sample shape was $W \times 3 \, \text{mm} \times L \times 1 \, \text{mm} \times T$ several $\mu \text{m}$, as shown in figure 1(c). The hysteresis exhibited ferromagnetic behavior with a magnetic moment of $1100 \, \text{emu cm}^{-3}$. The coercivity is 900 and 500 Oe for the out-of-plane direction and the in-plane direction, respectively. The coercivity has a fairly large value, despite eliminating the effect of shape anisotropy. The coercivity was highly dependent on the sample, and varied widely over the range from 500 Oe to 3 kOe. We here present the lower estimation, but this value is significantly larger than that of disordered FeNi, which is a soft magnetic ferromagnet with several Oe coercivity. This magnitude of the magnetic moment corresponds well with the value of $H_c = 1200 \, \text{emu cm}^{-3}$ obtained for synthetic $\text{L}_1\text{O}$-FeNi as described in [12]. Since this sample is polycrystalline, it is difficult to evaluate its magnetic anisotropy, but its coercivity is a fairly high value, and we confirmed that it exhibits hard magnetic behavior. The actual origin of the increase in coercivity is still unclear, but it is presumed to originate from pinning by impurities included in the material.
at room temperature. Also, when pulverizing the L10 was sealed inside a capillary and then exposed for 120 min, the lattice parameters were determined. The resulting superlattice peak is also confirmed at the shoulder. The resulting superlattice peak is confirmed in the 001 direction. (c) The Figure 2.

Figure 2. (a) Result of synchrotron radiation-based XRD. (b) The obvious superlattice peak is confirmed in the 001 direction. (c) The 110 superlattice peak is also confirmed at the shoulder. The resulting lattice parameters were $a = b = 3.582$ Å and $c = 3.607$ Å. The degree of order was estimated as $0.608 \pm 0.04$.

Figure 2 shows the results of synchrotron radiation XRD measurements for structural determination. The measurements were performed using the Debye–Scherrer camera installed at the BL19B2 beamline of SPring-8 [26]. With the x-ray energy set to 30 keV, the powdered natural L10-FeNi sample was sealed inside a capillary and then exposed for 120 min at room temperature. Also, when pulverizing the L10-FeNi flakes, sample screening was performed by using a permanent magnet to remove non-magnetic inclusions. Measurements were performed under the same conditions for natural L10-FeNi and disordered FeNi. Figure 2 shows the XRD results for L10-FeNi and disordered-phase FeNi. Fundamental peaks of FeNi were clearly observed and impurity peaks were mostly suppressed. This suggests that the HCl screening process worked well.

We carried out a Rietveld analysis of the wide-angle XRD patterns in figure 2(a); the lattice parameters of L10-FeNi were estimated as $a = b = 3.582$ Å and $c = 3.607$ Å ($c/a = 1.007$), and those of disordered FeNi were $a = b = c = 3.603$ Å. The superlattice peak position was also estimated from these lattice constants, indicated as the vertical solid lines in figures 2(b) and (c). A 001 superlattice peak is clearly confirmed for L10-FeNi, but no 001 peak was observed for the disordered-phase FeNi (figure 2(b)). In figure 2(c), a weak signal is also confirmed as a shoulder at the position of the 110 peak, but there is no peak for the disordered FeNi. The neighbor peaks at 9.5° may originate from impurities.

According to the extinction rule, the 001 and 110 peaks originate from superstructure and tetragonality of the L10-type structure. By considering the structure factor, the diffraction intensity of the 001 superstructure peak is in the order of 1/1000 with respect to the 220 fundamental peak; such a weak signal is very hard to investigate by laboratory-scale-based XRD. The observed results show a clear signal at both 001 and 110, although the 110 peak is overlapping with an impurity peak. There is no way to investigate the extremely weak signal except highly brilliant synchrotron radiation, because our Debye–Scherrer camera with imaging plate system has the dynamic range 1:10 000. Thus, our XRD results suggest that a superstructure is constructed in the meteoritic L10-FeNi specimen. By fitting a Voigt function to the 001, 110, and 220 peaks, we estimated the degree of order to be $0.608 \pm 0.04$. Since the degree of order in synthetic L10-FeNi is 0.48 at most, natural L10-FeNi has a higher degree of order. This confirms that the obtained FeNi flakes by HCl etching indeed show the L10-type superstructure, and that it is the equilibrium L10 state of the FeNi binary system [31].

Next, we performed K-edge MCD measurements to investigate the origins of magnetic anisotropy in L10-FeNi from a standpoint of electronic structure [27, 30]. K absorption edge MCD is known to contribute indirectly to the 3d orbital magnetic moment via 4p polarization. K-edge MCD of Fe and Ni was performed at the BL39XU beamline of SPring-8 [29]. Measurements were performed for natural L10-FeNi and disordered FeNi, and the photon energy was scanned at the region of the K absorption edge of Fe and Ni. A magnetic field of 2 T was applied parallel to the optical axis, and the x-ray absorption was detected using the transmission method. The circular polarization of the x-rays was switched by using a 0.45 mm diamond phase retarder, and an MCD signal synchronized with the circular polarization switching was detected with a lock-in amplifier, allowing weak signals of $10^{-5}$ or less to be obtained with high accuracy.

Figures 3(a) and (b) show the x-ray absorption spectra of Fe and Ni at the K absorption edge in natural L10-FeNi and disordered FeNi. Let us focus on the structure at the absorption edge; we can see that there are two peaks in all cases. In the FeNi system, it is known that the x-ray absorption fine structures drastically change, reflecting the electronic states that contribute to the bonding state, with a single peak in FeNi alloy with a bcc structure (an Ni composition ratio below 25 at.%), and double peaks in FeNi alloy with a fcc structure (an Ni composition ratio more than 25 at.%) [32]. This results shows that natural L10-FeNi adopts an fcc structure and not a bcc structure from the standpoint of the electronic state. It is difficult to distinguish between fcc and bcc from the x-ray absorption fine structure only, but the XRD results of figure 2 suggest that an L10-type structure is obtained; therefore both experimental results are consistent with each other.

Figures 3(c) and (d) show the MCD spectra at the K absorption edge of Fe and Ni in natural L10-FeNi and disordered FeNi. It is clear from this result that a polarization signal of Fe 4p in L10-FeNi is weaker than that of disordered FeNi (figure 3(c)); on the other hand, the polarization signal of Ni 4p in L10-FeNi is stronger than that of disordered FeNi.
Figure 3. XAS spectra obtained at the K absorption edge of Fe (a) and Ni (b). MCD spectra obtained at the K absorption edge of Fe (c) and Ni (d). The double peak at the crest of the XAS spectra suggests that the crystallographic structure takes the fcc form. The K-edge MCD intensity of Ni is stronger in meteoritic L1\textsubscript{0}-FeNi than in disordered FeNi. This suggests that polarization of the Ni 4p state in L1\textsubscript{0}-FeNi is stronger than that of disordered FeNi. (figure 3(d)). In general, spin polarization of the 4p orbital in Ni occurs via hybridization of 3d orbitals in neighboring Fe atoms, and indirectly reflects the spin polarization of 3d orbitals in Fe. It is thus suggested that the 3d orbital magnetic moment of Fe is more strongly polarized in L1\textsubscript{0}-FeNi than in disordered FeNi. This implies that the magnetic anisotropy bears a stronger correlation to the 3d orbital magnetic moment of Fe than that of Ni. By comparison with the experimental results on synthetic L1\textsubscript{0}-FeNi, it appears that the magnetic anisotropy of L1\textsubscript{0}-FeNi is increased by spin–orbit interaction arising from the 3d orbital magnetic moment of Fe. It could be suggested that the results of K-edge MCD are consistent with the known L-edge results; therefore it can be concluded that the magnetic anisotropy originates from the orbital magnetic moment of Fe 3d electrons.

Let us consider this orbital magnetic moment behavior based on spin–orbit interaction. Low-dimensional systems such as thin films or quantum wires give symmetry breaking of crystal structure. This commonly causes anisotropy in the orbital magnetic moment associated with the bonding state, and this is generally said to cause increasing magnetic anisotropy due to spin–orbit interaction. The 3d orbital magnetic moment of Fe is normally frozen, but it has been reported that reducing its dimensionality causes the magnetic anisotropy and orbital magnetic moment to increase [33, 34]. Our result shows that natural L1\textsubscript{0}-FeNi exhibits a fairly high degree of order, and tetragonality in the crystal structure was confirmed. This indicates that symmetry breaking of the crystal structure occurs. The Ni 4p electron of L1\textsubscript{0}-FeNi gives a higher polarization signal compared to the disordered FeNi phase. It is suggested that structural ordering enhances the polarization of Ni 4p electrons. Although the MCD spectra obtained are indirect information, this could be possible evidence that the 3d electron of neighboring Fe is polarized. Such behavior in the electronic state supports the previous results obtained for synthetic L1\textsubscript{0}-FeNi. It can thus be concluded that spin–orbit interaction is not negligible, and it is thought that this Fe orbital moment behavior provides a consistent explanation for the expression of magnetic anisotropy. This implies that controlling the Fe 3d orbital magnetic moment plays a key role in the enhancement of magnetic anisotropy.

4. Conclusion

To understand the hard magnetic behavior of L1\textsubscript{0}-FeNi, we extracted L1\textsubscript{0}-FeNi from a natural meteorite, and executed multidirectional analyses using SEM-EPMA, SQUID, XRD, and MCD. The composition ratio of natural L1\textsubscript{0}-FeNi is Fe: 50.47 ± 1.98 at.%, Ni: 49.60 ± 1.49 at.%, and it has been confirmed to have a high coercivity, more than 500 Oe. As a result of structural analysis, we obtained lattice parameter values \(a = b = 3.582\,\text{Å}\) and \(c = 3.607\,\text{Å}\), and a degree of order of 0.608. Our MCD measurements suggest that the orbital magnetic moment of Fe 3d electrons could be one origin of the magnetic anisotropy. This result is consistent with previous results obtained on synthetic L1\textsubscript{0}-FeNi.

Acknowledgments

The synchrotron radiation experiments were performed at the BL19B2 and BL39XU beamlines of SPring-8 with the approval of the Japan Synchrotron Radiation Research
Institute (JASRI) (Proposal Nos 2007A2119, 2007A2032, and 2011B1472). This study was partly supported by a Grant-in-Aid for Young Scientists (B19740210) and the JST Industry-Academia Collaborative R&D Program. The EPMA measurements were performed at Hiroshima University’s National Science Center for Basic Research and Development (N-BARD). We thank Dr Kenji Yoshii of the Japan Atomic Energy Agency (JAEA) for the SQUID measurements. We would also like to thank Minoru Funaki of the National Institute of Polar Research (NIPR) for his helpful discussion of meteorites.

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