MAGNETIC PROPERTIES OF LAMELLAR TETRATAENITE IN TOLUCA IRON METEORITE

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Abstract: Magnetic studies were conducted using lamellar tetrataenite extracted from the Toluca octahedrite by a diluted HCl etching technique. Natural remanent magnetization (NRM) in the lamellae is very stable against AF demagnetization and is quite intense, ranging from 2.58 to 37.42×10^{-2} Am^2/kg. This NRM is completely demagnetized at about 550°C of temperature. The most characteristic change in magnetic properties on heating up to about 550°C is a significant decrease in magnetic coercivity. This observation is consistent with the results obtained from chondrites. The paramagnetic component in lamellar tetrataenite, which is estimated by Mössbauer spectrum analyses, was not detected by conventional magnetic studies.

1. Introduction

Atomically ordered FeNi, which is detected as a tetragonal super lattice, was first discovered by ALBERTSEN *et al.* (1978a) in isolated lamellae from the Cape York and Toluca iron meteorites by Mössbauer spectroscopy and X-ray diffraction studies. ALBERTSEN *et al.* (1978b) showed that the ordered FeNi could be disordered by heating at 460°C for 10 h. CLARKE and SCOTT (1980) described ordered FeNi, which was given a new mineral name-tetrataenite, in over 50 chondrites, mesosiderites and iron meteorites, including Toluca, by microscope observations, where it is commonly distributed as 10–50 μm regions in contact with kamacite, troilite, taenite and silicate.

Recently the magnetic properties of tetrataenite in chondrites have been clarified by WASILEWSKI (1982, 1985) and NAGATA and FUNAKI (1982). The thermomagnetic curves (I_S-T curves) of tetrataenite-rich chondrites are characterized by a very flat heating curve up to 400–450°C and then an abrupt decrease to the Curie point which ranges 550–580°C, depending on composition. Chondrites containing tetrataenite grains have a highly stable NRM component against alternating field (AF) demagnetization up to 180 mT. Magnetic coercivity of the tetrataenite phase is much larger than that of ordinary (disordered) taenite.

Magnetic properties of bulk samples of ordinary chondrites are due to kamacite, taenite, cloudy taenite, tetrataenite, plessite and other ferromagnetic materials, and occasionally it is difficult to distinguish the magnetic properties of the tetrataenite from these integrated properties. Since LIN *et al.* (1977) reported the existence of tetrataenite in the plessite field by the Mössbauer spectrum analyses, where the magnetic properties of plessite may be similar to those of the tetrataenite. From the viewpoint of elucidating the intrinsic properties of tetrataenite, magnetic studies of pure tetrataenite have been performed.

The Toluca iron meteorites is a polycrystalline coarse octahedrite with Widmanstätten pattern of bandwidth 1.4±0.2 mm and Neumann bands (BUCHWALD, 1975). The high-nickel lamellae were prepared from the bulk sample of Toluca by using 2.5 N diluted HCl etching over a period of 15 days. The clear taenite (tetrataenite) lamellae are prepared by a further 5 days etching. By consecutive microscopical observations, we observe that the clear taenite sandwiched the plessite and cloudy taenite, and these are etched away completely. By this method a couple of clear taenite (tetrataenite) lamellae are obtained from one high nickel lamella.

There are probably two different mechanisms of tetrataenite formation in meteorite. The most popular one is tetrataenite lamella resulting from the Widmanstätten pattern formation; it is formed as rims on high-nickel taenite grains, which are characterized by the M-shaped Ni distribution profile in taenite. Another one is discrete tetrataenite grains which may be formed in shock-melted meteorites including high-nickel Fe-Ni grains such as Yamato-74160 (LL7) (TAKEDA and YANAI, 1980; TAKEDA *et al.*, 1984) and St. Séverin (NAGATA *et al.*, 1986). In the case of Toluca the former origin is identified by the microscopic observations.

2. Characteristics of Natural Remanent Magnetization

Natural remanent magnetization (NRM) of ten lamellae was measured using the superconducting rock magnetometer. The weight of the lamellae ranges from 0.08 to 1.39 mg, with an average 0.53 mg. Occurrence frequency of the NRM intensities is illustrated in Fig. 1, where the maximum and the minimum values are 37.42 and 2.58×10^{-2} Am^2/kg, respectively. The intensities of 50% occurrence are between 1 and 5×10^{-2} Am^2/kg. Since the bulk sample was 2.24 g in weight, and has 8.727×10^{-4} Am^2/kg of NRM intensity, the intensity of lamellae is about 100 times stronger than that of bulk Toluca.

A cubic sample of bulk Toluca (a) and three lamella samples (b), (c) and (d) were demagnetized by AF field up to 120 mT as shown in Fig. 2. Although the lamella samples were not mutually oriented, the directions appeared to be oriented along the plane of lamella. This can been seen in the vector orientation along the 90–270°

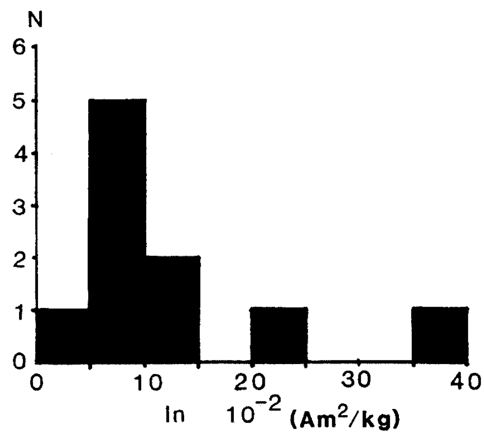

Fig. 1. Occurrence frequency of the NRM intensities of tetrataenite lamellae in Toluca.

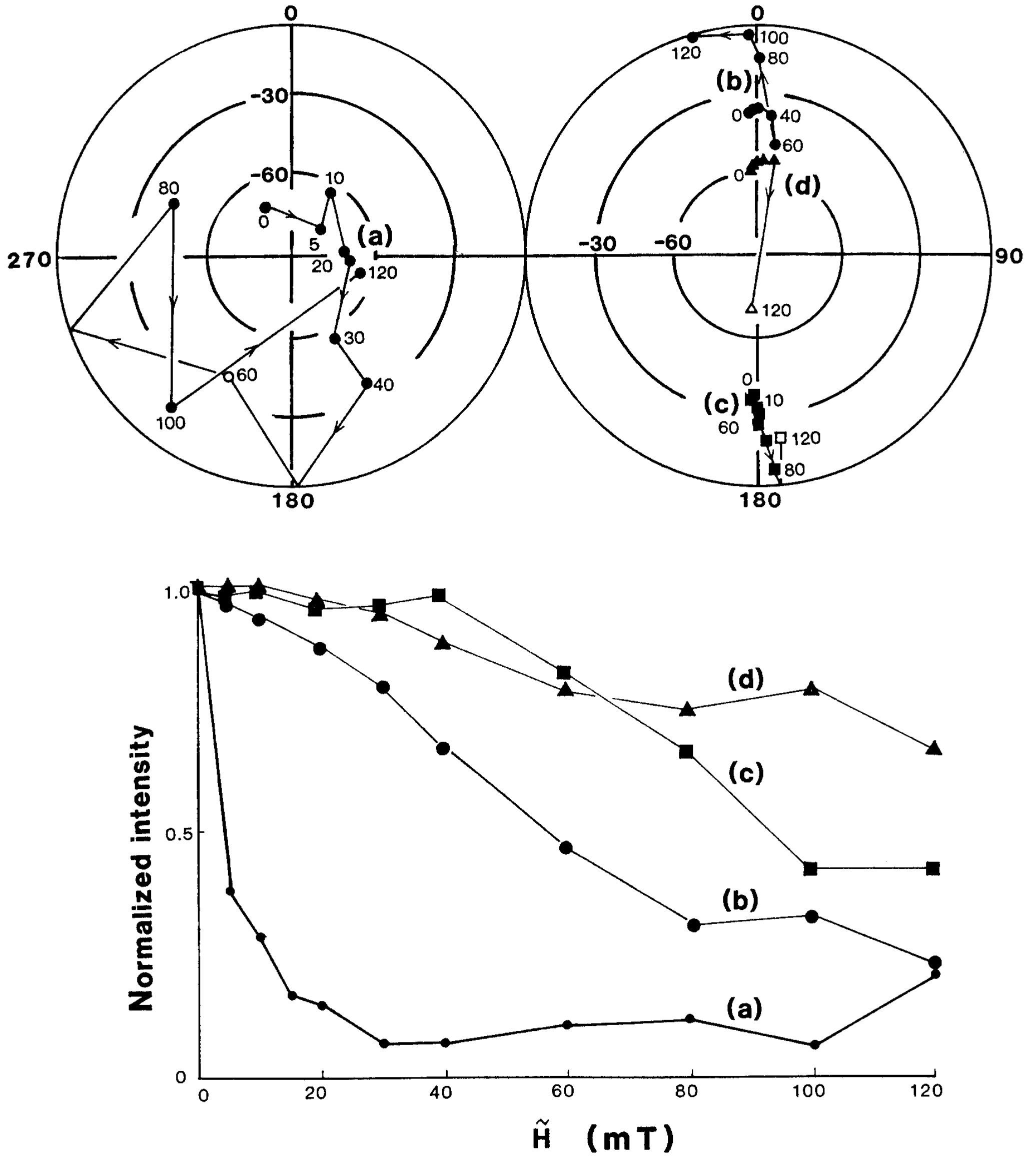

Fig. 2. AF demagnetization curves of NRM of Toluca bulk (a) and its tetrataenite lamellae (b), (c) and (d). Equal area projection.

meridian. Original intensity of bulk sample (a) is demagnetized by 65% steeply from 0 to 5 mT and then gradually from 5 to 30 mT. Although the changes of direction of this sample are relatively small from 5 to 20 mT, the NRM is unstable on the whole. Compared with the bulk sample, the NRM stability of the lamellae against AF demagnetization is fairly high, not only in intensity but also in direction as shown in Fig. 2. The original NRM is demagnetized gradually, having a median demagnetization field (MDF) of more than 50 mT. The NRM directions in this figure suggest that the lamellae have NRM oriented almost parallel to the lamella plane development.

NRM of the lamella samples was thermally demagnetized in steps of 50°C from 50 to 650°C. The samples were embedded completly by LARC-TPI adhesive to prevent oxidation, and then they were bonded on the glass holders for thermal demagnetization from 50 to 600°C. If the heating time is less than 5 min at a tempera-

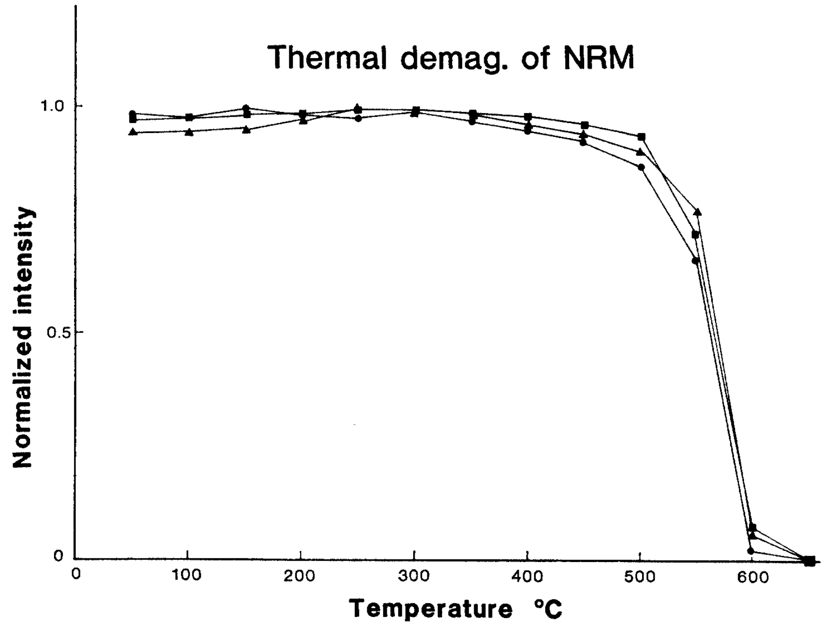

Fig. 3. Thermal demagnetization curves of NRM intensity of tetrataenite lamellae from Toluca.

ture lower than 600°C, the adhesive may protect the samples from excessive oxidation. When the samples were demagnetized at 650°C, they were enclosed in silica tubes under 10^{-5} torr of pressure for the same reason. Thermal demagnetization curves of NRM intensities for three lamellae show very similar characteristics as shown in Fig. 3; they show an almost flat demagnetization curve up to 500°C and then an abrupt breakdown at 600°C. Significant residual remanence is not observed at 600 and 650°C. The changes of directions are only few degrees up to 550°C, and then a larger shift at 600 and 650°C.

3. Anhysteresis Remanent Magnetization

Anhysteresis remanent magnetization (ARM) was given to three lamellar tetrataenites, using steady magnetic field $\bar{h}$=0.042 mT, and maximum alternating field $\tilde{H}$=120 mT. The directions of $\bar{h}$ and $\tilde{H}$ were parallel to each other and were perpendicular to the lamella plane. These samples were then demagnetized by AF field to 120 mT. After the AF demagnetization tests, the samples were heated up to 650°C repeating the ARM tests to check the differences.

Obtained results are summarized by curves (1)–(4) in Fig. 4. Curves (1): ARM acquisition in the original samples takes place gradually to 60 mT and then steeply increases to 120 mT. It is unsaturated even at $\tilde{H}$=120 mT. Curves (2): These ARMs are demagnetized gradually to 40–60 mT and then steeply to 120 mT. The ARMs disappeared completely at the maximum field. Curves (3): ARM acquisitions of heated samples were saturated in weak fields, about $\tilde{H}$=15 mT. Curves (4): Then their ARMs are completely demagnetized by 20 mT.

From these results, fairly high and small coercive forces can be estimated for the before- and after-heating lamellae, respectively. The increasing rate of ARM ac-

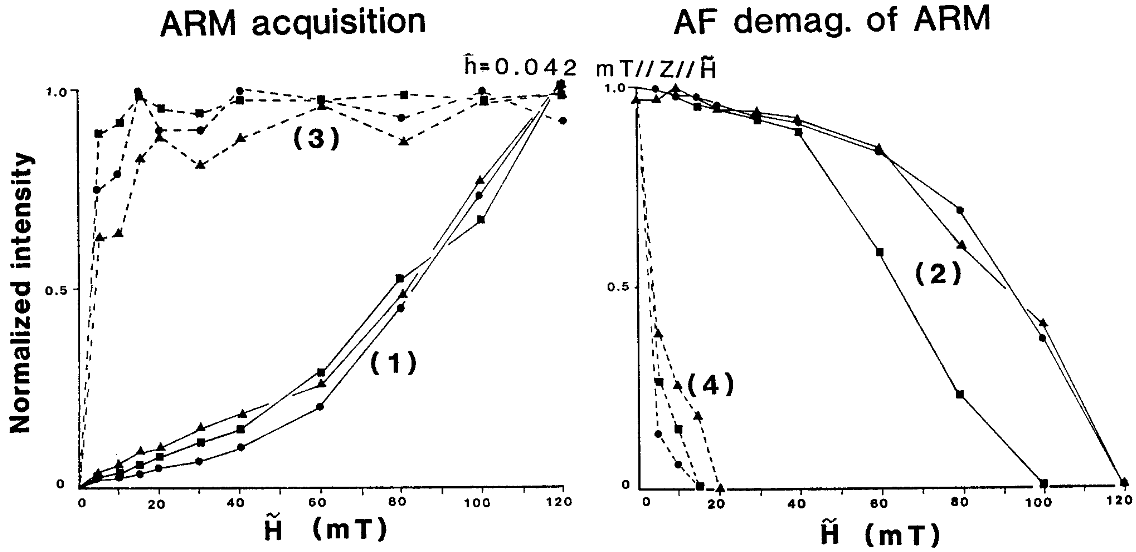

Fig. 4. Intensity change curves of ARM acquisition and AF demagnetization of ARM before and after heating (at 650°C) of three tetrataenite lamellae. Left: ARM acquisition curves, right: AF demagnetization curves of ARM. (1) and (2): pre-heating samples, (3) and (4) post-heating samples.

quisition, and the increasing demagnetization rate of ARM change taking place at about 60 mT suggest that the domain structure in these lamellar tetrataenite has a critical field response in the vicinity of 60 mT.

4. Thermomagnetic Curves (I_S-T curves)

Thermomagnetic curves (I_s-T curves) of a bulk sample were obtained in the range from room temperature to 800°C. An applied external field H=1000 mT, heating rate 200°C/h and pressure 10^{-5} torr were the experimental conditions. The 1st run I_S-T curves are shown in Fig. 5a. The sharp magnetic transitions at 735°C in the heating curve and 605°C in the cooling one correspond respectively to $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \alpha$ phase transition temperatures of kamacite, which corresponds to 7% Ni atomic ratio content in the FeNi alloy (HOSELITZ and SUCKSMITH, 1943). Two minor Curie points are observed at 550°C in the heating curve and at 240°C in the cooling curve. The former is a very unstable phase, because no detectable existence of this Curie point can be found in the cooling curve or in the 2nd run curves. The latter Curie point at 240°C is also observed in the 2nd run cooling curve, and it corresponds to 36% Ni in atomic ratio of an Fe-Ni alloy. The 2nd run I_S-T curves are fairly similar to the 1st run curves, except over the range between 30 and 550°C in the heating curve.

Representative I_S-T curves of a lamella sample, (which is shown in Fig. 5b), were obtained from −269 (4 K) to 650°C (933 K) under the same conditions as the bulk sample, but the heating was performed in helium gas from 30 to −269°C. In addition to this sample, I_S-T curves for seven lamellae were measured from 30 to 650°C. Significant characteristic behavior in the heating curves is a very flat decreasing curve from −269 to 550°C and then it abruptly breaks down from 550 to 575°C. In the

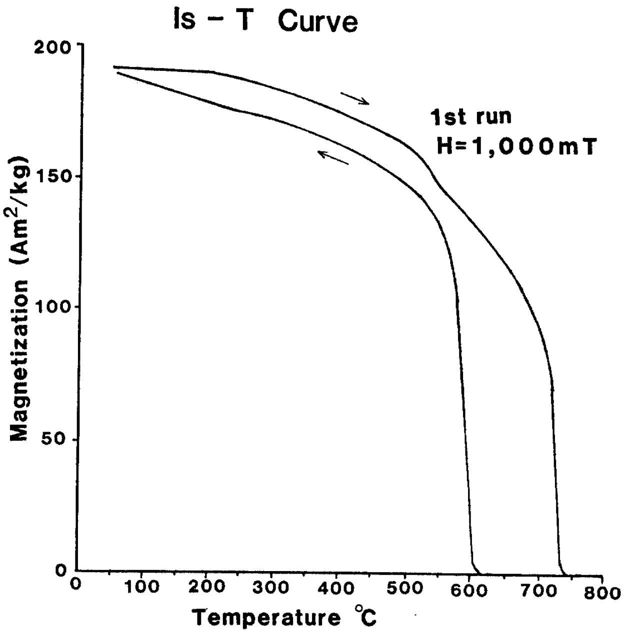

Fig. 5a. Thermomagnetic curves of a Toluca bulk sample.

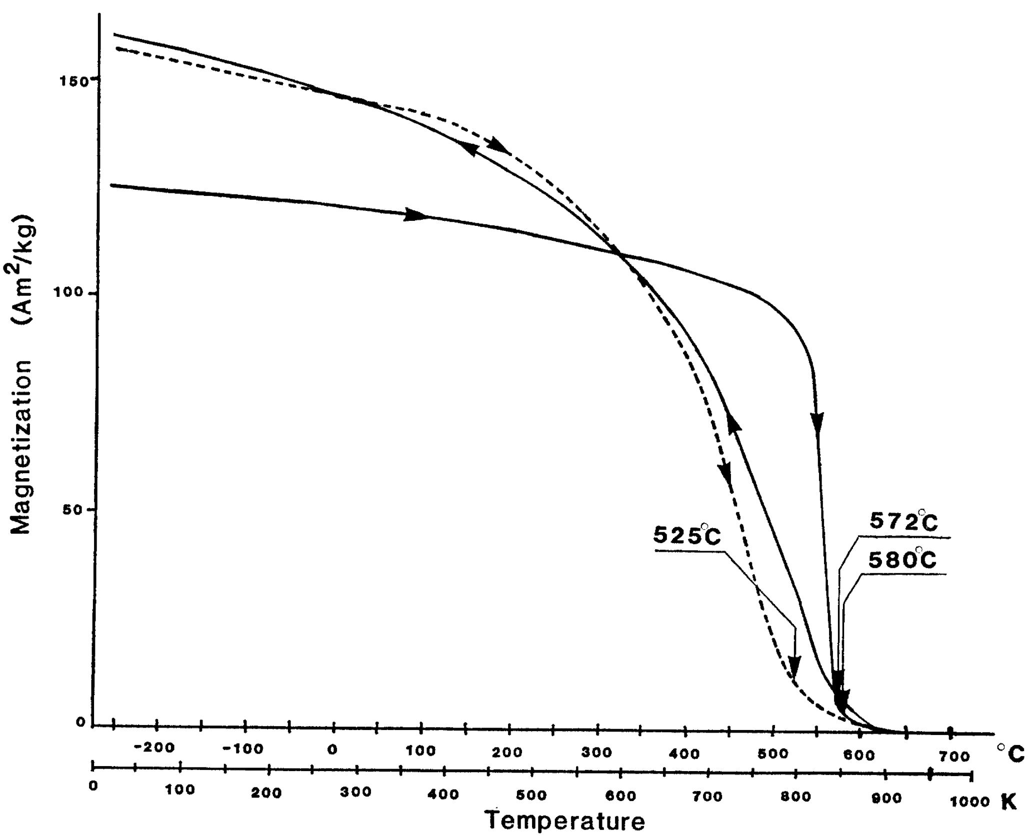

Fig. 5b. Thermomagnetic curves of Toluca tetrataenite lamella (solid line) and man-made 50Fe 50Ni alloy (dotted line).

cooling curve, the magnetization increases gradually from 620 to −269°C. Essentially the same tendency occurs among all other lamellae at temperatures higher than room temperature. The abrupt break for temperatures in the heating curves and the Curie points in the cooling curve for eight lamella samples are in the range of 550 to 585°C and 545 to 595°C, respectively. A small amount of magnetization, less than 5% of original one, is observed at the breakdown temperature and at the main Curie point, but no significant magnetization is observed at a temperature higher than 650°C. In the 2nd run heat treatment, the I_S-T curve is reversible and resembles the cooling curve of the 1st run treatment. The intensity of representative samples at room temperature increases 18% after the 1st run heating. This may be explained by the degree of saturation at 1000 mT in room temperature; before heating the sample is not saturated at 1000 mT for the high coercive force of tetrataenite but after heating it is saturated for the small coercive force of disordered taenite under the same conditions.

Figure 5b also shows the 1st run I_S-T curves of a man-made alloy of 50% Fe and 50% Ni. Its 515°C Curie point is reasonably consistent with the expected 517°C for the same composition, which was reported by CRANGLE and HALLAM (1963). The cooling curves for lamellae resemble the man-made alloy curves with regard to magnetization and aspect of the curve as shown in this figure. However, the Curie point at 575°C is higher than that of the alloy. Since the Curie point after heat treatment of eight lamella samples ranges from 545 to 595°C, the nickel contents are evaluated as 52 to 60% atomic percent. We measured the chemical compositions of pre-heating lamellae by EPMA, obtaining Fe=51.45 and Ni=49.05 wt% (Fe 52.44, Ni 47.56 at%) as well as minor amounts of Co=0.19 and Cr=0.02 wt%.

5. Magnetic Hysteresis Properties

Basic magnetic properties derived from magnetic hysteresis loops, saturation magnetization (I_S), saturation isothermal magnetization (I_R), coercive force (H_C) and remanent coercive force (H_{RC}), were measured at room temperature for the samples before and after heating up to 650°C as summarized in Table 1. The I_S values are

Table 1. Magnetic properties of Toluca iron meteorite.

Sample	Heating	I_S (Am^2/kg)	I_R (Am^2/kg)	H_C (mT)	H_{RC} (mT)	I_S*	I_R*	H_C*	H_{RC}*
Block	Before	208.4	1.0	2.45	129.6	1	0.02	0.16	0.05
	After	208.4	0.015	0.4	6				
Lamella 1	Before	143.5±8.6	28.0±1.68	45	72.9	0.09	0.18	0.03	0.04
	After	141.8±8.5	5.0±0.30	1.2	3				
Lamella 2	Before	152.5±19.8	70.8±9.2	69.5	87.7	1.07	0.68	0.05	0.03
	After	163.5±21.3	19.0±2.3	2.2	3				
Lamella 3	Before	133.5±21.4	27.0±4.3	44.5	70.4	0.98	0.57	0.04	0.03
	After	130.4±20.9	15.5±2.5	1.8	2.3				

I_S: Saturation magnetization, I_R: saturation remanent magnetization, H_C: coercive force, H_{RC}: remanent coercive force, I_S*, I_R*, H_C* and H_{RC}*: Rates of after heating values to before ones.

estimated by the law of approach to saturation magnetization. In general, the magnetic saturation curve is represented by

$$I = I_S(1 - a/H - b/H^2 \cdots) + \chi_0 \mu_0 H ,$$

where I, χ_0 and μ_0 are magnetization, relative susceptibility and permeability in vacuum. If the magnetocrystalline anisotropy of materials is very high (small), dominant influences by the term (b/H^2) should be high (low) in the field of I_S-T curve. Occasionally the second term (a/H) has large influences for that of small samples caused by unknown reasons, even though the materials are isotropic. We checked the effects of b/H^2 and a/H in the hysteresis curves ranging from 800 to 1450 mT by a method of extrapolating to $H=0$ mT.

The evaluated I_S values are shown in Table 1 together with other hysteresis data and the ratio of post-heating values to pre-heating ones which is denoted by * on each parameter. Values of $H_C=44.5$–69.5 mT and $H_{RC}=70.4$–87.7 mT decrease to $H_C=1.2$–2.2 mT and 2.3–3.0 mT by heating at 600°C. These coercivity values in the pre-heating samples are extremely high compared with chondrites, and the post-heating values are almost the same as those for low nickel chondrites (E, H chondrite). The values of $I_S{}^*$ is very close to 1.0, suggesting no great chemical composition changes in magnetic materials by heat treatment. Other rates $I_R{}^*$, $H_C{}^*$ and $H_{RC}{}^*$ are very small for all samples. The plausible reason for coercivity changes of such magnitude in FeNi alloys must be the transition from an ordered state to a disordered state by heat treatment up to 650°C. The small value of $H_C=2.45$ mT in the bulk sample illustrates the dominance of the coercive force by kamacite, because the dominant iron phase of Toluca is kamacite as estimated by I_S-T curves. The large value of $H_{RC}=129.6$ mT may be related to high coercivity materials such as lamellar tetrataenite and tetrataenite in plessite included in this meteorite.

6. Discussion

Measurements of magnetic properties of tetrataenite grains in chondrites were reported for Y-74160 and St. Séverin (NAGATA *et al.*, 1986). Y-74160 is an extremely recrystallized LL chondrite, classified as LL7 (TAKEDA and YANAI, 1980; TAKEDA *et al.*, 1984), and its magnetic minerals are estimated to be taenite and tetrataenite by magnetic studies. The model compositions of magnetic phase in the metal component in St. Séverin chondrite (LL6) are kamacite (18.5%), tetrataenite (34.8%) and taenite (46.7%). When the basic magnetic properties are obtained from chondrites using magnetic hysteresis curves from −1500 to 1500 mT, the results show the superposed values of kamacite, taenite, tetrataenite, plessite and other ferromagnetic materials. If chondrites include small amounts of materials of high coercive force, the I_S value is affected by magnetization of both the ferromagnetic and the paramagnetic components. NAGATA and FUNAKI (1982) attempted the separation of magnetic properties in chondrites which consisted of two ferromagnetic components. However, as many chondrites have several ferromagnetic components, it is difficult to separate the magnetic properties of one phase out of chondrite. Furthermore, it is necessary to check the differences in magnetic properties between discrete grains and lamellar tetrataenite.

Tetrataenite grains have extremely high H_C and H_{RC} values, but the values decrease by heating to 550–580°C (NAGATA and FUNAKI, 1982). In Y-74160, for example, the respective values change from 22.5 and 40.6 mT to 0.8 and 24 mT by heating above 550°C. As summarized in Table 1, lamellae of Toluca have very high magnetic coercivities, H_C=44.5–69.5 mT, before heating and small coercivities, H_C=1.2–2.2 mT, after heating to 650°C. This is consistent with the results from tetrataenite grains. The values of H_C^*=0.03–0.05 and H_{RC}^*=0.03–0.04 suggest that the decreasing ratio of the coercive force to the remanent coercive force is very similar within each lamella. NÉEL *et al.* (1964) observed the I_S value of ordered FeNi is approximately the same as that of disordered FeNi (taenite). Since the I_S^* values of lamellae have a range of 0.98–1.07, this may support the above results.

ALBERTSEN *et al.* (1978a) studied Toluca by the X-ray technique and obtained essentially single crystals containing the superstructure L10 of FeNi. NAGATA and FUNAKI (1982) estimated the coercive force of single crystal of tetrataenite as about H_C=490 mT. The results of AF demagnetization of NRM and ARM and ARM acquisition of lamellae suggested they have very stable remanent magnetizations, but they are demagnetized to a certain degree up to 120 mT. From these viewpoints, observed values H_C=44.5–69.5 mT may show the coercive force of multidomain structure of single crystal of lamellar tetrataenite.

From microscopic observations and EPMA analyses, ferromagnetic minerals of kamacite, taenite, plessite and tetrataenite phases are defined clearly in the bulk Toluca sample. However, the first run heating I_S-T curves suggest only the existence of kamacite and tetrataenite. As the plessite phase is estimated to have very similar I_S-T curves (*i.e.* NAGATA and SUGIURA, 1976) and Mössbauer spectrum (LIN *et al.*, 1977) to tetrataenite one, the I_S-T curve of the tetrataenite phase in the bulk sample is composed of both plessite and tetrataenite phases. The chemical compositions of the taenite phase obtained by EPMA analyses ranges from 20.3 to 27.7 wt% in nickel content. If a sufficient amount of this phase exists in Toluca, there would be no detection of any magnetization (paramagnetics) due to lower Curie point than room temperature. Otherwise, if these taenite grains have the martensite structure, some magnetizations resulting from bcc phase should be observed in the I_S-T curves. From the observation results, we cannot check whether the taenite grains have some magnetization from the I_S-T curve due to the small amount of the taenite phase compared with kamacite. Consequently, only the kamacite and tetrataenite phases are recognized clearly in the I_S-T curve magnetically.

The characteristic flat 1st run heating curve of lamellae shows the typical I_S-T curve of a single component of magnetically homogeneous material. The abrupt breakdown of magnetization between 550 and 570°C is consistent with the transformation of an ordered phase (tetrataenite) to a disordered one (taenite) reported by WASILEWSKI (1982, 1985) and NAGATA and FUNAKI (1982) using Bjurbole (L4), Y-74160 (LL7), ALH-77260 (L3) and St. Séverin (LL6) chondrites and Estherville mesosiderite. For the Toluca lamellae, the breakdown of magnetization between 550 and 570°C appears to be influenced by a phase transition from order to disorder states.

The Mössbauer spectrum of Toluca lamellae (ALBERTSEN *et al.*, 1978b) indicated a superposition of two spectra of a paramagnetic γ-phase and an asymmetric six-line spectrum from the ordered phase. The Ni content of the paramagnetic γ-phase is

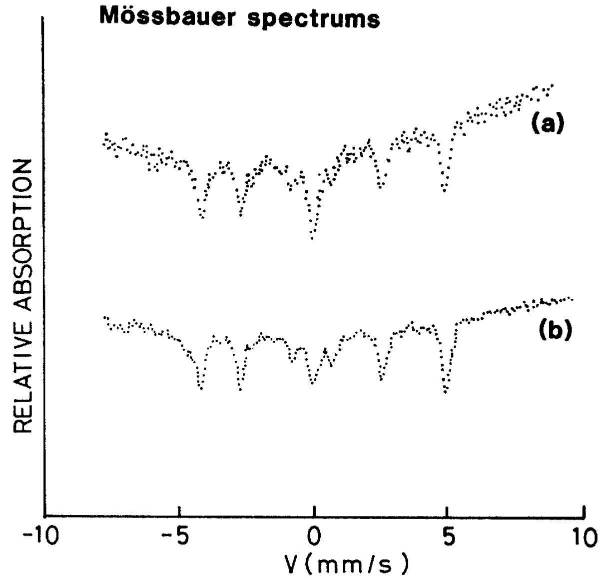

Fig. 6. Mössbauer spectra at room temperature of Toluca tetrataenite lamellae.

lower than about 30%Ni with a Curie point below room temperature. We obtained the results of Mössbauer spectrum of our Toluca lamellae at room temperature, as shown in Fig. 6, and found them to be essentially the same as their spectrum; the magnetic hyperfine field H_i and the electric quadrupole shift ε are $H_i=285$ kG and $\varepsilon=0.245$ mm s^{-1}. If the paramagnetic component is included in the lamella samples, it should be detected by some magnetization in the I_S-T curves from -269 to 800°C; (1) The curve shows that some gradually decaying magnetizations exceeded the Curie points; (2) The magnetization increases steeply at temperature lower than about -240°C as reported by NAGATA and FUNAKI (1982). However, we find no magnetization above 650°C and no steeply increasing magnetization below -240°C in the I_S-T curves. If lamellar tetrataenites include a paramagnetic component, the saturation magnetization, I_S, should be smaller than that of standard 50 wt% FeNi alloy; the I_S values of three lamellae in the range of 133.5 ± 21.4–152.5 ± 19.8 Am^2/kg, as shown in Table 1, are similar to the values 150.5 Am^2/kg of 50 wt% FeNi (HOSELITZ, 1952). These magnetic observations suggest that the lamellar tetrataenite does not include any large amount of paramagnetic component as observed in the Mössbauer spectrum analyses.

The bulk sample of Toluca has unstable NRM with weak NRM intensity (8.73×10^{-4} Am^2/kg) about 1/100 times the lamellar tetrataenite (2.58–37.42×10^{-2} Am^2/kg) intensity. As Widmanstätten structures in the octahedrite develop along (111) planes, those lamellae are aligned in the same directions. NRM directions in tetrataenite lamellae should be parallel to (111) planes of Toluca. However, we do not know the mutual orientation of the lamellae. It is important to decide the orientation of the lamellae in Toluca samples for evaluation of tetrataenite formation and for paleomagnetic studies of meteorites.

The NRM directions of lamellar tetrataenite are fairly stable up to 550°C but become unstable at 600°C against thermal demagnetization. The thermal demagnetization curves of these samples essentially resemble those of Y-74160 (NAGATA and

FUNAKI, 1982); the temperature at which unstable NRM is developed is 530°C; it has a very flat curve up to about 500°C and then an abrupt breakdown at 600°C. These breakdown temperatures are in the same range as the phase transition temperature of 550–575°C obtained from the I_S-T curves. From these viewpoints and the observed decreasing coercive force at that temperature, it seems likely that the breakdown of NRM is caused by phase transition from order to disorder rather than conventional NRM thermal blocking.

6. Conclusion

The NRM intensity of lamellar tetrataenite in Toluca is in the range of 2.58 to 37.42×10^{-2} Am²/kg. It is stronger by about 100 times than the bulk NRM intensity. The existence of tetrataenite lamellae in meteorites is important for NRM analyses. This NRM is fairly stable against AF demagnetization but it is completely demagnetized thermally by heating to 550–575°C. Magnetic hysteresis results obtained from lamellae before and after heating suggest that the phase transition from order to disorder takes place during heating up to about 550°C. During this transition, the original lamellae with fairly high H_C values of 44.5–69.5 mT decrease to 3–5% of the original values after heating to that temperature. This phenomenon is reflected in the other magnetic characteristics, such as thermal demagnetization of NRM, ARM acquisition, AF demagnetization of ARM and I_S-T curves, comparing the measurements made before and after heat treatment to about 550°C. Therefore, the characteristics of lamellar tetrataenite in Toluca are essentially the same as the results which were reported by WASILEWSKI (1982, 1985), NAGATA and FUNAKI (1982) and NAGATA *et al.* (1986).

Existence of the paramagentic component in Toluca, estimated by Mössbauer spectrum analyses, is not detected by conventional magnetic experiments such as magnetic hysteresis curve and I_S-T curves from −269 to 650°C. Further studies should be performed to consider the apparent discrepancy between Mössbauer identification of the paramagnetic component in tetrataenite and no identification of it magnetically.

Acknowledgments

The authors wish to thank Dr. P. WASILEWSKI (GSFS, NASA) for his discussion of tetrataenite magnetism and his correction of the manuscript in English and to Dr. H. NAGAI (Shinshu University) for his measurements and discussions of Mössbauer spectrum analyses.

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(Received August 30, 1985; Revised manuscript received November 29, 1985)