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MAGNETIC PROPERTIES OF LAMELLAR TETRATAENITE IN
TOLUCA IRON METEORITES

by

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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} emu/g. This NRM is completely demagnetized thermally at about 550°C. The most characteristic change in magnetic properties on heating 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.

Key-words: Magnetic; Tetraenite; Meteorite.

1. Introduction

Atomically ordered FeNi, which is detected as a tetragonal super lattice, was first discovered by Alberston et al. (1978a) in isolated lamellae from the Cape York and Toluca iron meteorites by Mössbauer spectroscopy and X-ray diffraction studies. Albersten et al. (1978b) showed that the ordered FeNi could be disordered by heating at 460°C for 10 hours. Clark 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, 85) 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 between 550-580°C, depending on composition. Chondrites containing tetrataenite grains have a highly stable NRM component against alternating field (AF) demagnetization up to 1800 Oe. 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 existance of tetrataenite in the plessite field by the Mössbauer spectrum analyses, the magnetic properties

of plessite may be similar to that of the tetrataenite. From the viewpoint of elucidating the intrinsic properties of tetrataenite, magnetic studies of pure tetrataenite have been performed.

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 materials 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 mechanism of tetrataenite formation in meteorite. The most popular one is tetrataenite lamella resulting from the pattern from the Widmanstätten pattern formation; it is formed as rims on high Ni taenite grains, which are characterized by the M shaped Ni distribution profile in taenite. Another one is discrete trataenite grains which may be formed in shock melted meteorites including high nickel Fe-Ni grains such as Ym-74160 (LL7) (Takeda and Yanai, 1980; Takeda et al. 1984) and St. Séverin (Nagata and Funaki, 1985). In case of Toluca the former origin is identified by the microscopic observations.

2. Characteristics of natural remanent magnetization

Natural remanent magnetization (NRM) of 10 lamellae were measured using the superconducting rock magnetometer. The weights of the lamellae range 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} emu/g respectively. The intensities of 50% occurrence are between 1 and 5×10^{-2} emu/g. Since the bulk sample was 2.24 g in weight, and has 8.727×10^{-4} emu/g of NRM intensity, the intensity of lamellae are 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 1200 Oe as shown in Fig. 2. Although the lamellae samples were not mutually oriented, the directions appeared to be oriented along lamellae planes. This can be seen in the vector orientation along the 90° - 270° meridian. Original intensity of bulk sample (a) is demagnetized by 65% steeply from 0 to 50 Oe and then gradually from 50 to 300 Oe. Although the changes of direction of this sample are relatively small from 50 to 200 Oe, the NRM is unstable on the whole. Compared with the bulk sample, the NRM stability against AF demagnetization for the lamellae is fairly high, not only the intensities but also the directions as shown in Fig. 2; The original NRM is demagnetized gradually having median demagnetization field (MDF) of more than 500 Oe. The NRM directions in this figure suggest that the lamellae have NRM oriented almost parallel in direction to the lamellae plane development.

NRM of the lamellae samples were demagnetized thermally in steps of 50°C from 50 to 650°C . They were embedded completely 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 minutes at 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 break down to 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 3 lamellar tetrataenites, using steady magnetic field $\bar{h}=0.42$ Oe, and maximum alternating field $\tilde{H}=1,200$ Oe. The directions of \bar{h} and \tilde{H} were parallel to each other and were perpendicular to the lamellae plane. These samples were then demagnetized by AF field to 1,200 Oe. After the AF demagnetization tests, the samples were heated up to 650°C repeating the ARM tests to check the differences.

Obtained result are summarized in Fig. 4 curves (1)-(4). Curves (1): ARM acquisition in the original samples takes place gradually to 600 Oe and then steeply increases to 1,200 Oe. It is unsaturated even at $\tilde{H}=1,200$ Oe. Curves (2): These ARMs are demagnetized gradually to 400-600 Oe and then steeply to 1200 Oe. The ARMs disappeared completely at the maximum field. Curves (3):

ARM acquisitions of heated samples were saturated in weak fields, about $\tilde{H}=150$ Oe. Curves (4): Then their ARMs are completely by 200 Oe.

From these results, fairly high coercive forces and small coercive forces can be estimated for the before and after heating lamellae respectively. The increasing rate of ARM acquisition, and the increasing demagnetization rate of ARM change taking place at about 600 Oe suggests that the domain structure in these lamellar tetrataenite have a critical field response in the vicinity of 600 Oe.

4. Thermomagnetic curves (I_S -T curves)

Thermomagnetic curves (I_S -T curves) of a bulk sample were obtained from room temperature to 800°C. An applied external field $H=10$ KOe, 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. 5(a). The sharp magnetic transitions at 735°C in heating curve and 605°C in cooling one correspond to $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \alpha$ phase transition temperature of kamacite respectively, which corresponds to 7% Ni atomic ratio content in the Fe-Ni alloy (Hoselitz and Sucksmith, 1943). Two minor Curie points are observed at 550°C in the heating curve and 240°C in cooling curve. The former is very unstable phase because of no detectable existence of this Curie point can be found in the cooling curve or 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 1st run curve, except over the range between 30 and 550°C in heating curve.

Representative I_S - T curves of a lamella sample, (which is shown in Fig. 5(b)), was obtained from $-269(4K)$ to $650^\circ C(933K)$ under the same conditions as the bulk sample, but it was performed in helium gas from 30 to $-269^\circ C$. In addition to this sample, I_S - T curves for 7 lamellae were measured from 30 to $650^\circ C$. Significant characteristic behavior in the heating curves are a very flat decreasing curve from -269 to $550^\circ C$ and then abrupt break down from 550 to $575^\circ C$. In the cooling curve, the magnetization increases gradually from 620 to $-269^\circ C$. 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 cooling curve for 8 lamella samples are in the range of 550 to $585^\circ C$ and 545 to $595^\circ C$ respectively. A small amount of magnetization, less than 5% of original one, is observed at the break down temperature and the main Curie point, but no significant magnetization is observed at a temperature higher than $650^\circ C$. 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 1st run heating. This reason may be explained by degree of saturation at 10 KOe in room temperature; before heating sample does not saturate at 10 KOe for the high coercive force of tetrataenite but after heating it saturates for the small coercive force of disordered taenite under the same conditions.

Figure 5(b) also shows the 1st run I_S - T curves of manmade alloy of 50%Fe 50% Ni. Its $515^\circ C$ Curie point is reasonably consistent with that expected $517^\circ C$ for the same composition, which was reported by Crangle and Hallam (1963). The cooling curves for lamellae resemble to the manmade 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 Curie points after heat treatment of 8 lamellae samples range from 545 to 595°C, the nickel contents are evaluated 52 to 60% atomic percent. We measured the chemical compositions of before 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 a 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. I_S values are estimated by the law of approach to saturation magnetization. In general, the magnetic saturation curve is represented by

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

where I , χ_0 and μ_0 are magnetization, relative susceptibility and permeability of 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 uncertain reason, even though it is isotropic materials. We checked the effects of b/H^2 and a/H in the hysteresis curves ranging of 8 to 14.5 KOe by a method of extrapolating to $H=0$ KOe.

The evaluated I_S values are shown in Table 1 together with

other hysteresis data and rates of after heating values to before ones which are denoted by * on each parameters. Values of $H_C=445-695$ Oe and $H_{RC}=704-877$ Oe decrease to $H_C=12-22$ Oe and $23-30$ Oe by heating at 600°C . These coercivity values in the before heating samples are extremely high compared with chondrites, and the after heating values are almost the same values as for low nickel chondrites (E, H chondrite). The value 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 $H_C=24.5$ Oe 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. Large values of $H_{RC}=1,296$ Oe 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 from Ym-74160 and St. Séverin (Nagata and Funaki, 1985). Ym-74160 is an extremely recrystallized LL chondrite, classified 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 -15 to 15 KOe, the results show the superposed values of kamacite, taenite, tetrataenite, plessite and other ferromagnetic materials. If chondrites include small amounts of high coercive force materials, I_S value are affected by magnetizations 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 their values decrease by heating to 550-580°C (Nagata and Funaki, 1982). For example Ym-74160, these respective values change from 225 and 406 Oe to 8 and 240 Oe by heating to greater than 550°C. As summarized in Table 1, lamellae of Toluca have very high magnetic coercivity, $H_C=445-695$ Oe, before heating and small coercivities, $H_C=12-22$ Oe after heating to 650 °C. It is consistent with the results from tetrataenite grains. Rates of $H_C^*=0.03-0.05$ and $H_{RC}^*=0.03-0.04$ suggest that decreasing rates of coercive force and remanent coercive force are very similar within each lamellae. Néel et al. (1964) observed the I_S value of ordered FeNi. It 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 their results.

Albertsen et al. (1978a) studied Toluca by X-ray techniques 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 $H_C=4,900$ Oe. 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 1200 Oe. From these viewpoints, observed values $H_C=445-695$ Oe may show the coercive force of multidomain structure of single crystal of tetrataenite lamellae.

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 suggests only the existence of kamacite and tetrataenite. As the plessite phase is estimated to have very similar I_S-T curves (ie. 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 both of plessite and tetrataenite phases. The chemical compositions of taenite phase obtained by EPMA analyses are from 20.3 to 27.7 wt% in nickel contents. If sufficient amount of this phase exists in Toluca, it is no detection of any magnetization (paramagnetics) due to lower Curie point than room temperature. Otherwise, if these taenite grains have martensite structure, some magnetizations resulting bcc phase should be observed in I_S-T curves. From the observation results, we can not check whether the taenite grains have some magnetization from the I_S-T curve due to small amount of taenite phase compared with kamacite. Consequently only kamacite and tetrataenite phases are recognized clearly in I_S-T curve magnetically.

The characterized flat 1st run lamellae heating curve shows the typical I_S-T curve of a single component of magnetically homogeneous material. The abrupt break down of magnetization

between 550 to 570°C is consistent with the transformation of an ordered phase (tetrataenite) to a disordered one (taenite) reported by Wasilewski (1982, 85) and Nagata and Funaki (1982) using Bjurbole (L₄), Yamato-74160 (LL₇), ALH-77260 (L₃) and St. Séverin (LL₆) chondrites and Estherville mesosiderite. For the Toluca lamellae, the reason for the break down of magnetization between 550 to 570 °C appears to be influenced by a phase transition from order to disorder.

The Mössbauer spectrum of Toluca lamellae (Albertsen et al., 1978) indicated a superposition of two spectra of a paramagnetic \uparrow -phase and an asymmetric six-line spectrum from the ordered phase. The Ni content of the paramagnetic-phase is lower than about 30%Ni with a Curie point below room temperature. We obtained the results of Mössbauer spectrums of our Toluca lamellae at room temperature, as shown in Fig. 6, and find 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 $\epsilon=0.245$ 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 some gradually decay magnetizations exceeded the Curie points; (2) The magnetization increases steeply in lower than about -240 °C in temperature as reported by Nagata et al., 1972. However, we find no magnetization more than 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; I_S values of three lamellae in the range of 133.5 ± 21.4 - 152.5 ± 19.8 emu/g, as shown in Table 1, are similar to the values 150.5 emu/g 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} emu/g) about 1/100 times the lamellar tetrataenite ($2.58-37.42 \times 10^{-2}$ emu/g) 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 each lamellae. It is important to decide the orientation of the lamellae samples in Toluca for evaluation of tetrataenite formation and for paleomagnetic studies of meteorites.

NRM directions of lamellar tetrataenite are fairly stable up to 550°C but unstable to 600°C against thermal demagnetization. The thermal demagnetization curves of these samples essentially resemble those of Ym 74160 (Nagata and Funaki, 1982); the temperature at which unstable NRM is developed is 530°C; it has very flat curves up to about 500°C and then an abrupt break down to 600°C. These break down temperatures are in the same range as the phase transition temperature at 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 break down of NRM is caused by phase transition from order to disorder rather than conventional NRM thermal blocking.

6. Conclusion

NRM intensity of lamellar tetrataenite in Toluca is in the range of 2.58 to 37.42×10^{-2} emu/g. 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 the phase transition from order to disorder takes place during heating to about 550 °C. During this transition, the original lamellae with fairly high H_C values of 445-695 Oe decreases 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 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, 85) and Nagata and Funaki (1982, 85).

Existence of the paramagnetic component in Toluca, estimates 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 it magnetically.

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Table 1. Magnetic properties of Toluca iron meteorite.

Sample	heating	I_S emu/g	I_R emu/g	H_C Oe	H_{RC} Oe	I_S^*	I_R^*	H_C^*	H_{RC}^*
block	before	208.4	1.0	24.5	1296	1	0.02	0.16	0.05
	after	208.4	0.015	4	60				
lamella 1	before	143.5±8.6	28.0±1.68	450	729	0.09	0.18	0.03	0.04
	after	141.8±8.5	5.0±0.30	12	30				
lamella 2	before	152.5±19.8	70.8±9.2	695	877	1.07	0.68	0.05	0.03
	after	163.5±21.3	19.0±2.3	22	30				
lamella 3	before	133.5±21.4	27.0±4.3	445	704	0.98	0.57	0.04	0.03
	after	130.4±20.9	15.5±2.5	18	28				

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.

Figure of captions

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.

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

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

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

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

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

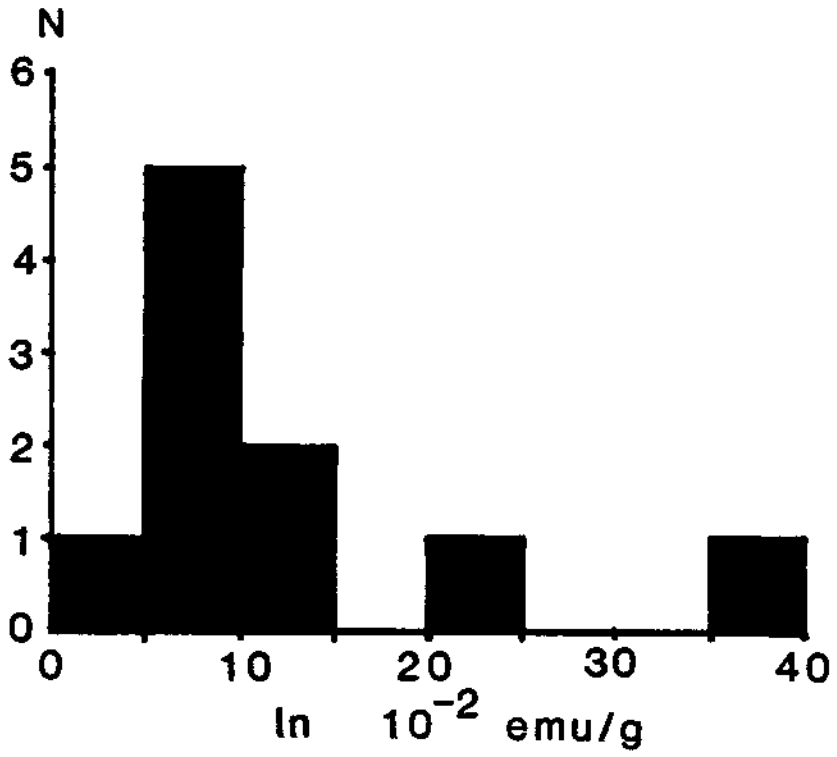


Fig. 1

AF DEMAG. OF NRM

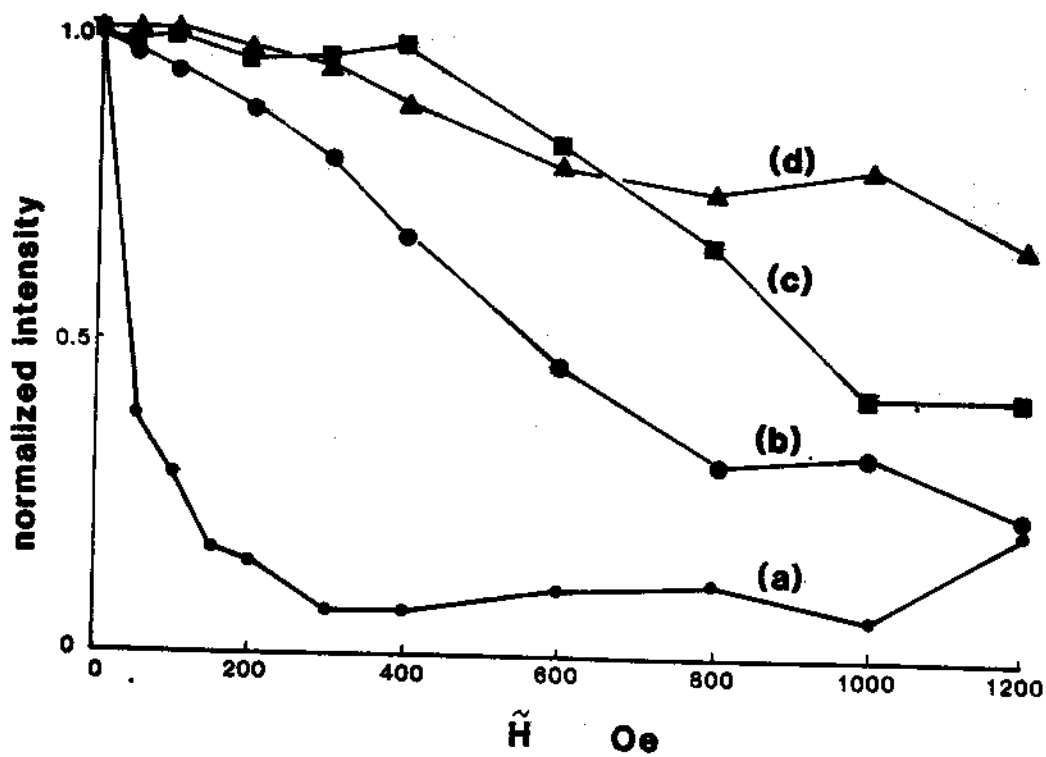
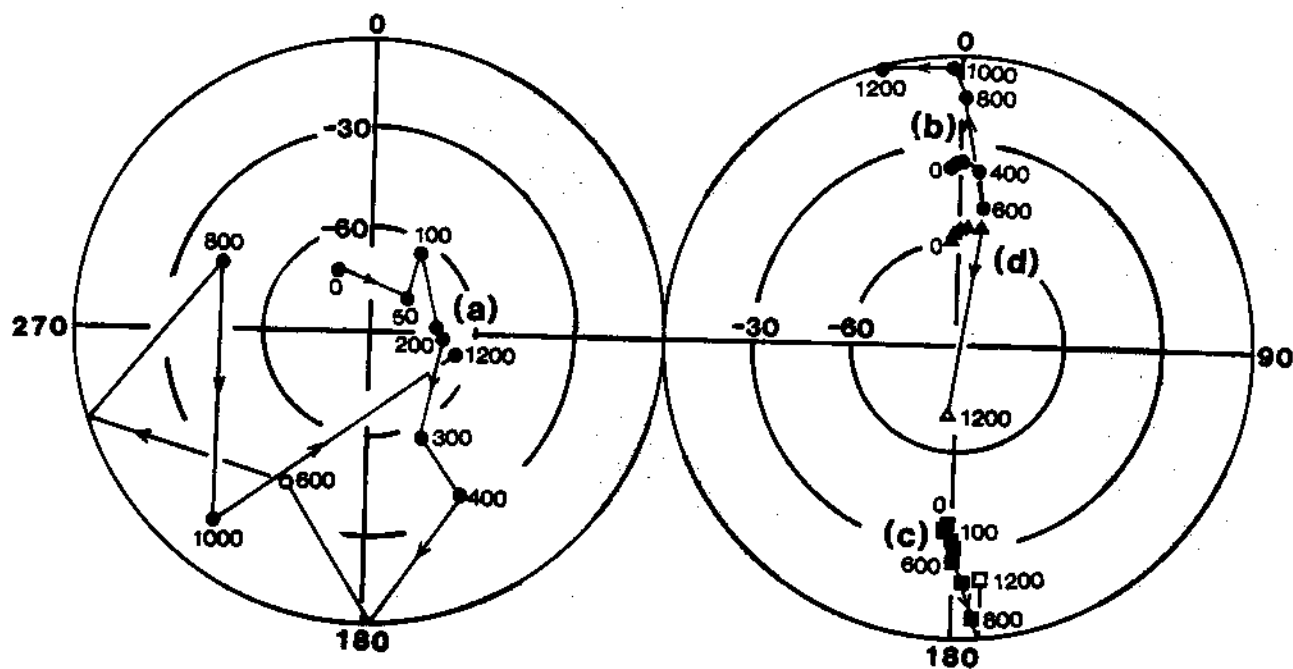


Fig. 2

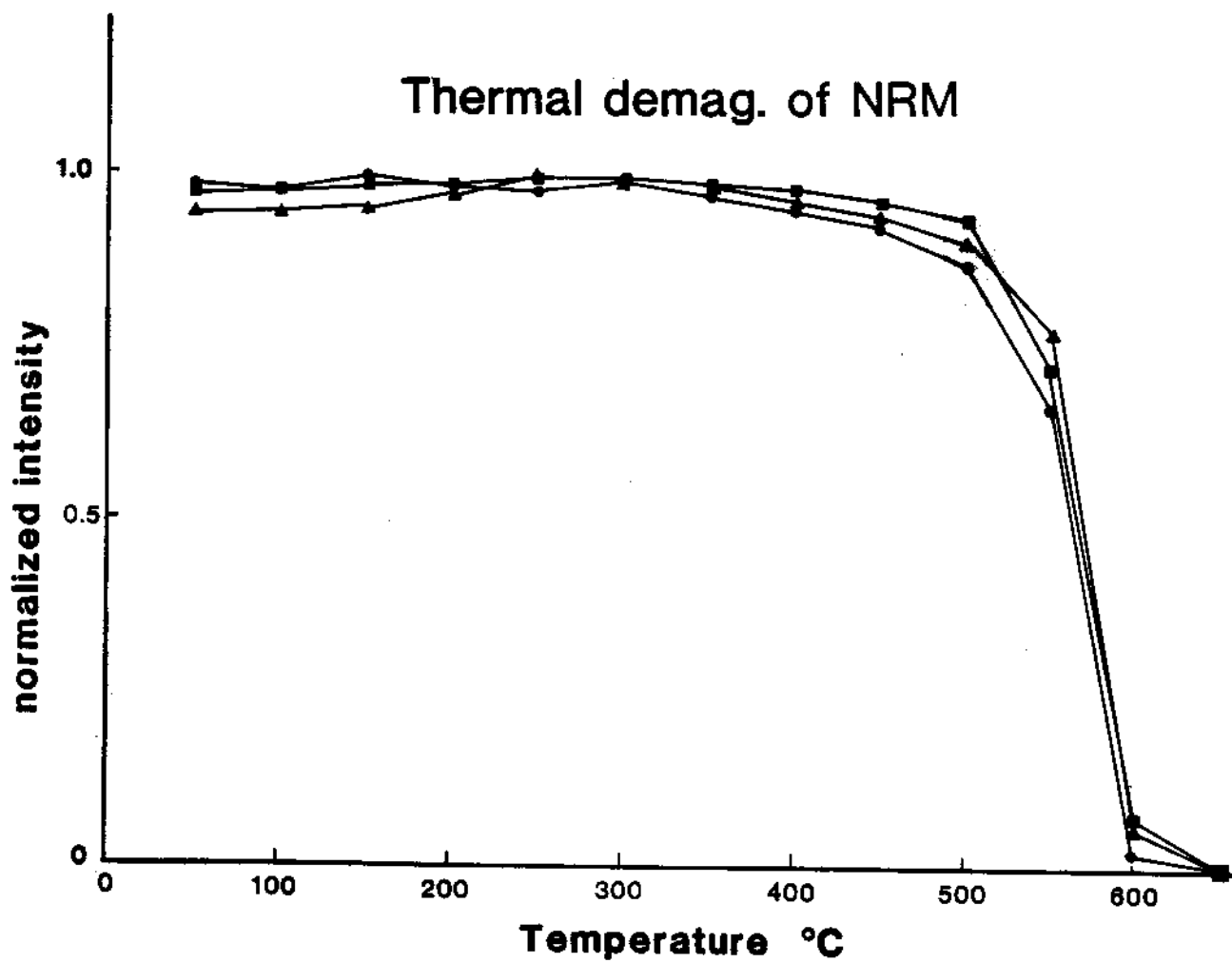


Fig. 3

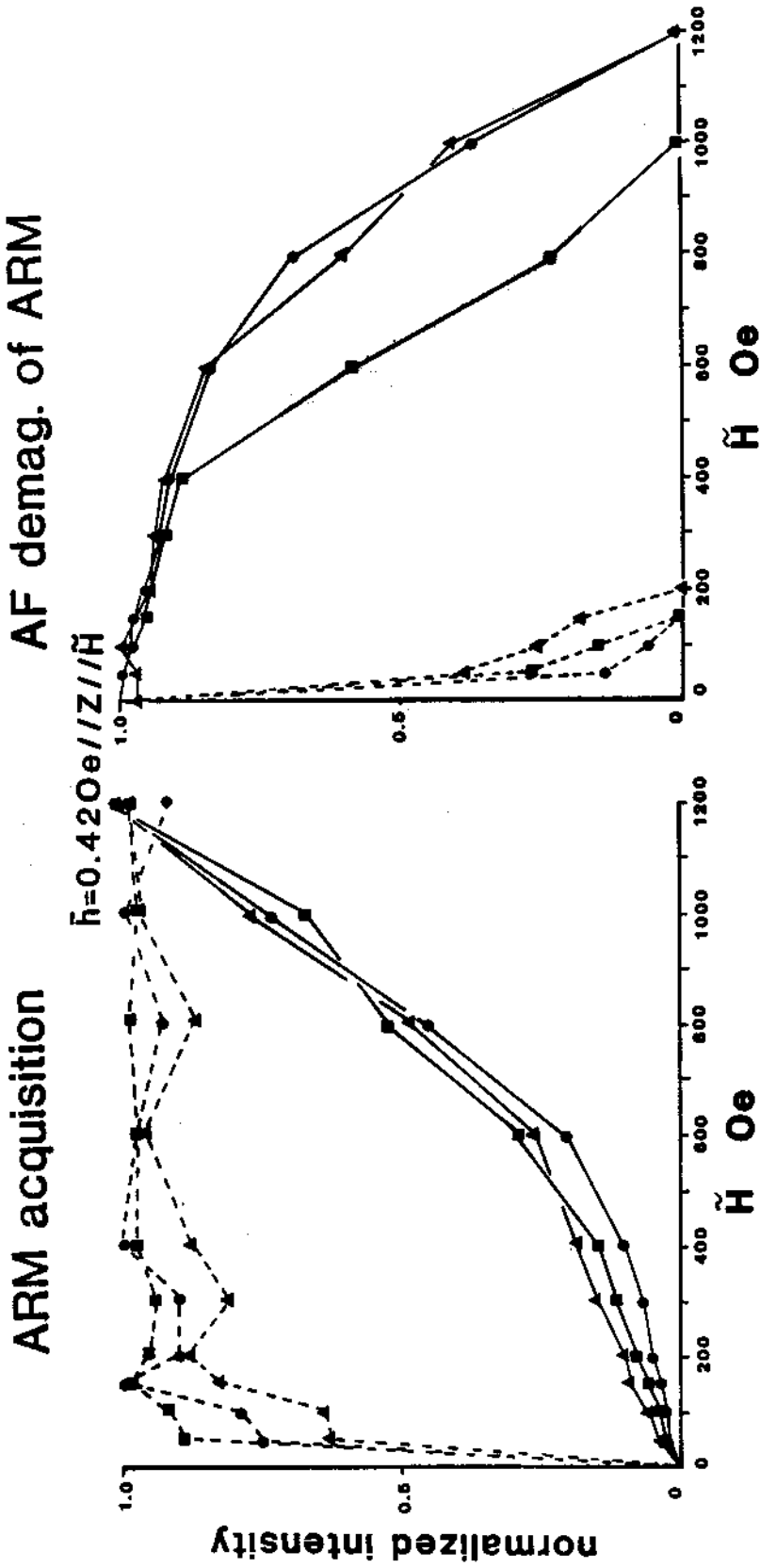


Fig. 4

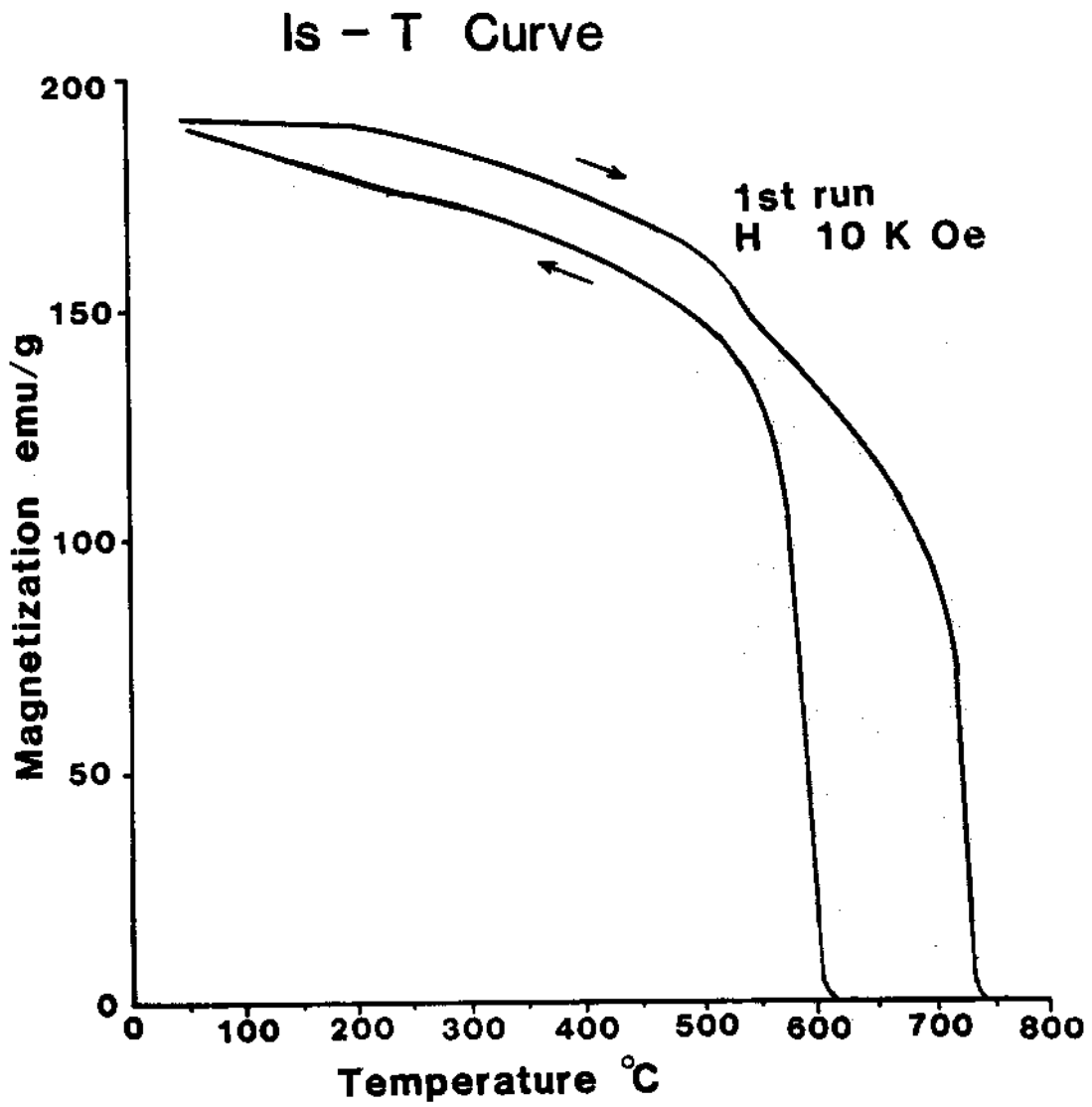


Fig. 5(a)

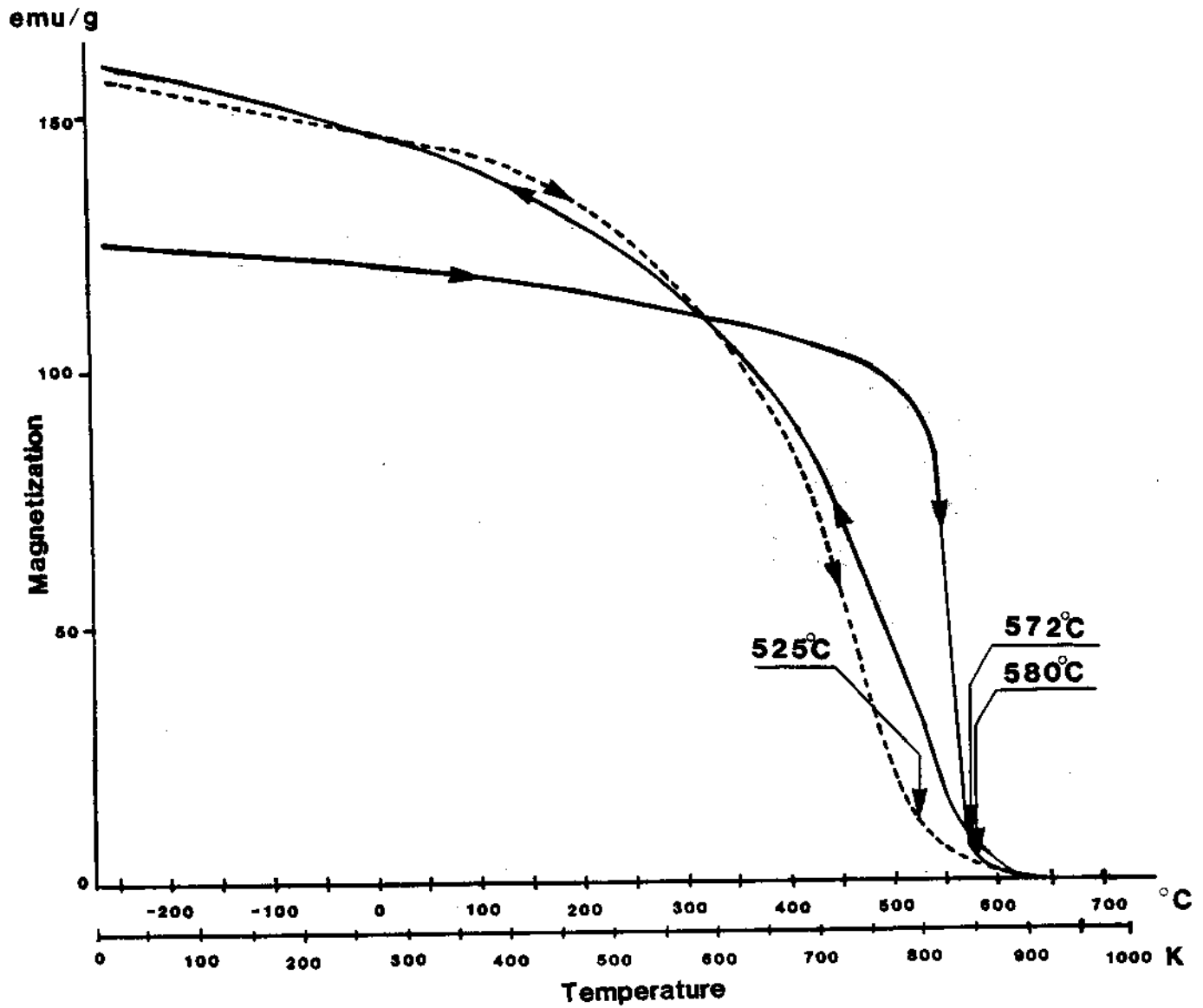


Fig. 5(b)

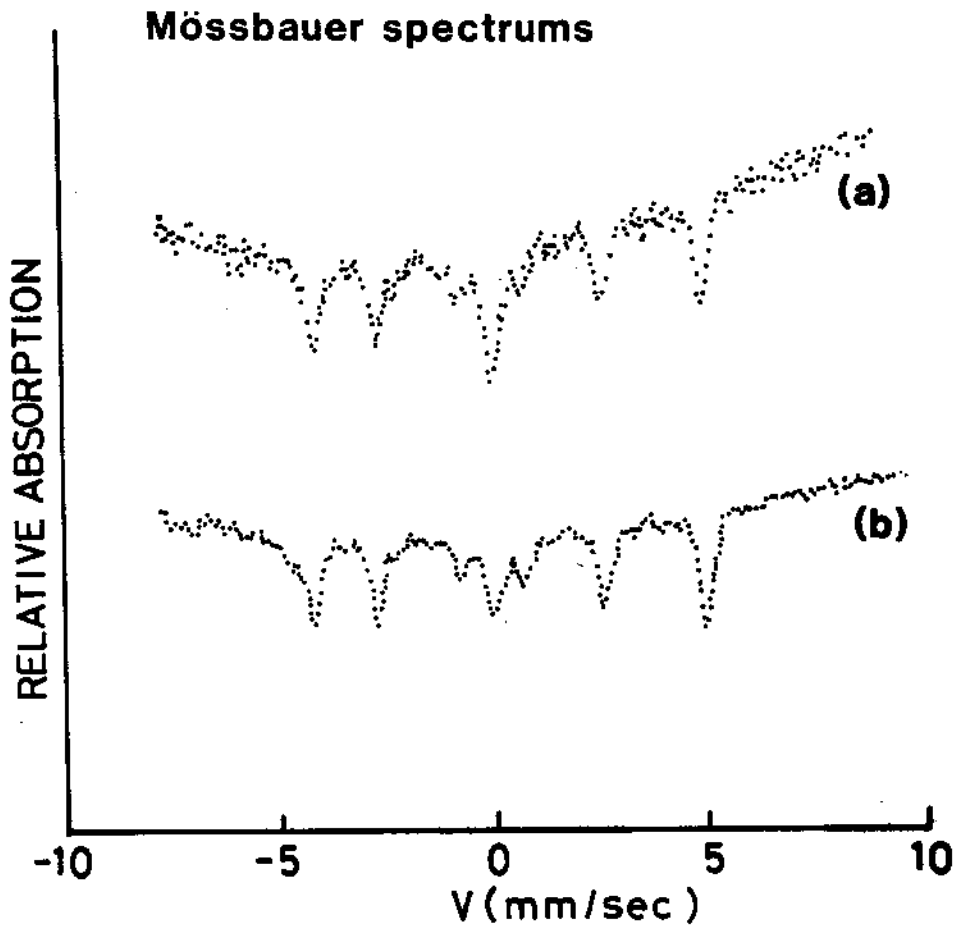


Fig. 6

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