Ferromagnetic resonance of lunar samples

FUN-DOW TSAY and SUNNEY I. CHAN

Arthur Amos Noyes Laboratory of Chemical Physics,* California Institute of Technology
Pasadena, California 91109

and

STANLEY L. MANATT

Space Sciences Division, Jet Propulsion Laboratory, California Institute of Technology
Pasadena, California 91103

Abstract—Evidence is presented to support that the electron spin resonance (ESR) spectra observed for a selection of Apollo 11 lunar samples (10087-10, 11; 10046-29, 30; 10062-26, 27; 10017-35, 36) arise from the ferromagnetic centers consisting of metallic Fe. A model study to simulate the polycrystalline spectra has been carried out, from which it was possible to ascertain with some degree of certainty the size and shape of the ferromagnetic centers as well as the metallic iron content. Some variations in the metallic Fe content have been noted in these samples, for example, between rocks and fine soil.

INTRODUCTION

IN OUR previous report (MANATT *et al.*, 1970) on the electron spin resonance studies of the lunar samples, we suggested that the broad, asymmetric signals ($g = 2\cdot12 \pm 0\cdot05$) detected in both rock chip (62-27) and fine (87-10) samples are principally ferromagnetic in nature arising from particles of metallic iron of the order of 1 μm dia. Our previous interpretations were based on the evidence that no significant temperature dependence of the absorption intensity is noted. In addition, both the effective g-value and the first-order anisotropy energy were found to be close to those characterized for metallic Fe.

In spite of the fact that no appreciable amount of ferric ion was detected in Mössbauer experiments (HERZENBERG and RILEY, 1970; MUIR *et al.*, 1970), the strong ESR signals centered at $g = 2\cdot1$ have been interpreted by WEEKS *et al.* (1970a, b) and GEAKE *et al.* (1970a, b) as arising from the paramagnetic Fe^{3+} ion. Their interpretations are hardly acceptable simply because the observed g-value is too high to be accounted for by high-spin Fe^{3+}, which is known to have a $^6S_{5/2}$ ground state. For low-spin Fe^{3+} and for paramagnetic Fe^{2+} as well, no room temperature spectra are expected on the basis of the rapid spin-lattice relaxation rates for these systems. Furthermore, as discussed in more detail below, the signal intensities observed for the lunar samples are at least three orders of magnitude greater than those expected for possible paramagnetic species.

To fully substantiate our claims that these resonances arise from ferromagnetism rather than from paramagnetism, we have carried out a detailed lineshape analysis of the resonance signals observed for the lunar samples. Lineshape analysis is particularly important in this work, not only because the spectra are

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taken on a polycrystalline sample, but also because absorption intensity measurements obtained by integrating the area under the absorption curves are necessary to distinguish ferromagnetism from paramagnetism. In order to do this, we have developed a computer program to simulate the ferromagnetic spectrum of a polycrystalline sample consisting of randomly oriented ferromagnetic centers such as those found in the lunar samples. Our method is similar in substance to that previously described by STANDLEY and STEVENS (1956), but the details of the calculations are quite different.

EXPERIMENTAL

Electron spin resonance spectra were obtained at both X-band (9·50 GHz) and K-band (34·83 GHz) frequencies on a Varian 4500 spectrometer using 100 kHz field modulation. The lunar samples were sealed in 2–3 mm o.d. thin-walled quartz tubes. Blank tests were carried out on some of the tubes to assure that no detectable resonance signals originated from them.

The g-values were determined both from direct measurements of the cavity frequency and the resonance magnetic field, and by comparison with the resonance position of 0·1% DPPH ($g = 2·0037$) dispersed in solid KCl. The low temperature measurements were carried out by the immersion of the cavity in liquid nitrogen in the K-band studies, whereas at X-band, precooled nitrogen gas was blown through the cavity.

The number of the unpaired electrons in the lunar samples was calculated by comparison of the area under the absorption curves with that measured for a 0·102% DPPH sample (1·560 × 10^{18} spins/g). To minimize the experimental error, the tubes used for both the lunar fines and the standard sample were chosen to have the same i.d. and were filled with almost the same volume of the samples. In addition, spectra were recorded with both the lunar sample and the standard in the cavity simultaneously.

ANALYSIS OF THE FERROMAGNETIC RESONANCE SPECTRA

For a single crystal containing ferromagnetic centers, KITTEL (1948) has shown that the resonance condition for ferromagnetic resonance absorption is

$$h\nu = g\beta\{[H_z + (N_y + N_y^e - N_z)M_z][H_z + (N_x + N_x^e - N_z)M_z]\}^{1/2}, \tag{1}$$

where ν is the resonance frequency; g, the electron g-value; H_z is the applied external magnetic field; and M_z is the magnetization. The constants h and β are Planck's constant and the Bohr magneton, respectively. $N_{x,y,z}$ and $N_{x,y}^e$ denote the demagnetizing factors. The terms $N_{x,y,z}$ reflect the effect of the shape of the specimen, and the terms $N_{x,y}^e$ arise from the crystalline anisotropy energy. In a crystal, the amount of energy required to magnetize the domain structure varies with the relative orientation between the magnetization and the crystal axes of the sample. As a result, one not only expects the resonance condition to be dependent on the shape of the ferromagnetic specimen, but the resonance field is also strongly angularly dependent because of the crystalline anisotropy energy. In deriving the above resonance condition, it was assumed that the size of the ferromagnetic center is small compared to the skin depth of the sample such that eddy current effects are negligible. If this assumption is not valid, there will be mixing of the real and imaginary components of the frequency dependent magnetic susceptibility and the resonance signal will be highly distorted.

It can be seen from equation (1) that, only in the case of a spherical specimen with a small crystalline anisotropy energy, where $N_x = N_y = N_z = 4\pi/3$ and $N_{x,y}^e < H_z$, does the resonance field occur at a position close to $g = 2·0$, the free electron g-value. For such a spherical specimen and if the crystal structure is cubic, as in the case of metallic Fe, the angular dependence of the resonance field has been shown (STANDLEY and STEVENS, 1956; SCHLÖMAN, 1958) to be

$$H(\theta, \phi) = H_0 - \frac{2K_1}{M_z}[1 - 5(\cos^2\theta\sin^2\theta + \sin^4\theta\sin^2\phi\cos^2\phi)], \tag{2}$$

where $H_0 = h\nu_0/g\beta$, and $2K_1/M_z$ is the first-order anisotropy energy. θ, ϕ denote respectively the polar and azimuthal angles of the crystal axes as defined by the edges of the cube relative to the applied external field. The expression in equation (2) was derived on the assumption that the crystalline anisotropy energy is much smaller than the Zeeman energy so that the effective magnetization can still be taken parallel to the external magnetic field. In situations, where the crystalline anisotropy energy is large, we note that it may be necessary to carry the perturbation calculation up to second order.

In the case of polycrystalline or powder samples, the situation is further complicated by the fact that the crystal axes of the microcrystallites are randomly oriented. Consequently, the observed spectrum of a polycrystalline sample is a superposition of the individual, angularly dependent resonances corresponding to the different orientations of all the microcrystallites. Since, for a given azimuthal angle ϕ, the number of microcrystallites whose crystal axes with polar angles between θ and $\theta + \mathrm{d}\theta$ with respect to the external magnetic field is known (SANDS, 1955) to be proportional to $\frac{1}{4\pi}\sin\theta\,\mathrm{d}\theta$, the absorption intensity due to these microcrystallites at their resonance field $H(\theta, \phi)$ is proportional to

$$\frac{\mathrm{d}N}{\mathrm{d}H} = \frac{\mathrm{d}N}{\mathrm{d}\theta}\frac{\mathrm{d}\theta}{\mathrm{d}H} = \frac{N_0\bar{n}\sin\theta}{4\pi(\mathrm{d}H/\mathrm{d}\theta)}, \tag{3}$$

where N_0 is the total number of microcrystallites and $\bar{n}$ is the average number of spins per microcrystallite. The quantity $\mathrm{d}H/\mathrm{d}\theta$ can be obtained by differentiation of the resonance field expression given by equation (2) with respect to the polar angle. Equation (3) then becomes

$$\frac{\mathrm{d}N}{\mathrm{d}H} = \left(\frac{M_z}{2K_1}\right)\left(\frac{N_0\bar{n}}{40\pi}\right)\{\cos\theta[\cos 2\theta + 2\sin^2\theta\sin^2\theta\cos^2\phi]\}^{-1}. \tag{4}$$

From this expression, we see that the absorption intensity approaches infinity at $\theta = 90°$, and at angles where the quantity in the brackets [ ] becomes zero. In the latter situation, the so-called angular anomalies occur (NEIMAN and KIVELSON, 1961). Because of the crystalline anisotropy energy and the resultant angular dependence of the resonance field, we therefore expect angular anomalies to contribute substantially to the overall lineshapes of the ESR spectra of a ferromagnetic polycrystalline material.

RESULTS

In the ESR experiments, rather broad resonances with g-values greater than that for the free electron were detected in the Apollo 11 samples (87-10, 11; 46-29, 30; 62-26, 27; 17-35, 36). For the lunar fines (87-10, 11) and the chips (46-29, 30), the signal intensities were found to be roughly an order of magnitude greater than those observed for the rock chip samples (62-26, 27). These signals were, in turn, an order of magnitude stronger than those observed for the rock samples (17-35, 36), even though a much larger amount of the rock samples was used in the latter measurements. These results tend to agree with the results of the magnetic susceptibility measurements where the strongest magnetization was also found in the lunar fines (LSPET, 1969; NAGATA et al., 1970).

The X-band and K-band spectra recorded for the fine sample (87-11) over the temperature range from 80°K to 298°K are presented in Fig. 1 and Fig. 3, respectively. In these experiments, the resonance field was searched from 8000 gauss to 50 gauss at X-band frequency (9·501 GHz), and from 18,000 gauss to 1750 gauss at K-band frequency (34·825 GHz). No other resonances were observed in these regions, except for the one centered at $g = 2·09 \pm 0·03$. The overall lineshape of the observed spectra is asymmetric, and as we shall later show, this asymmetry
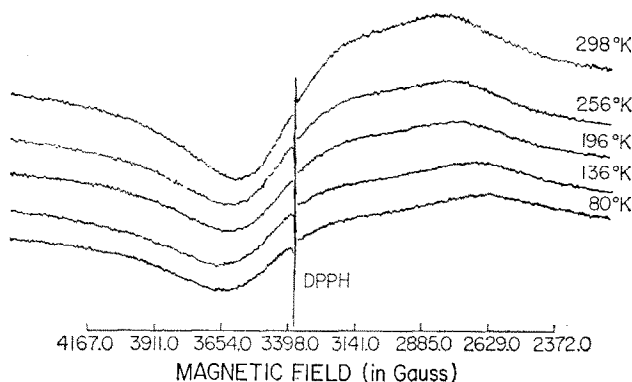
Fig. 1. Temperature dependence of the X-band ESR spectra of the lunar fines (10087-11).
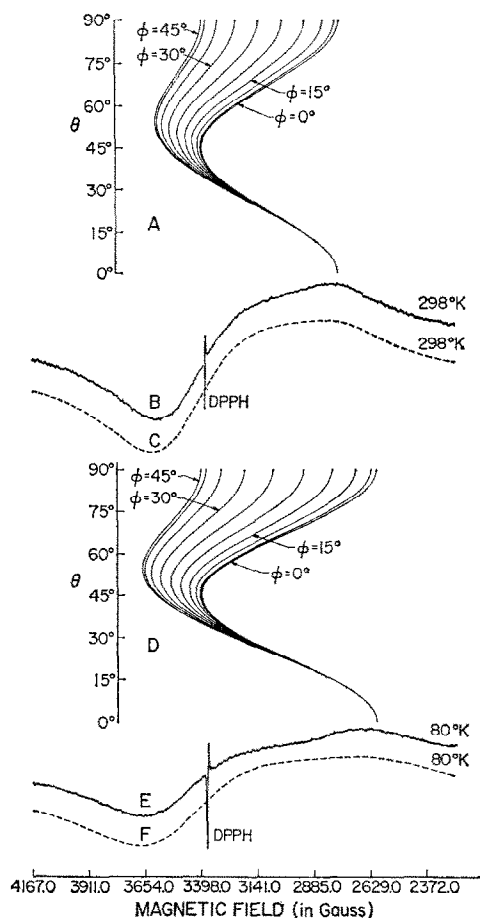

Fig. 2. Angular dependence of the resonance field positions at X-band for the lunar sample (10087-11) at 298°K (curve A) and at 80°K (curve D); and comparison of the observed and computer-simulated spectra at 298°K (spectra B, C) and at 80°K (spectra E, F). Simulated spectra are indicated by broken curves.
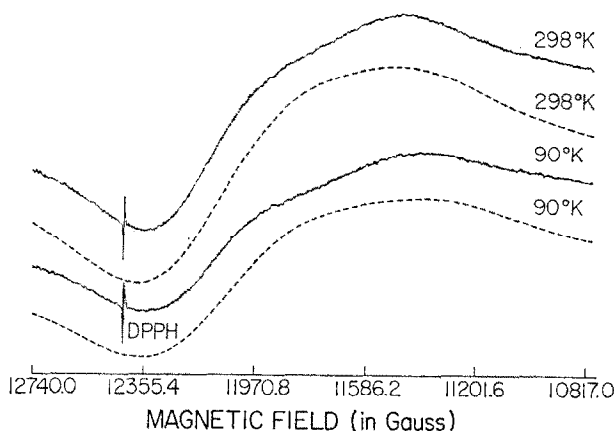
Fig. 3. Observed and computer-simulated K-band spectra for the lunar fines (10087-11) at 298°K and at 90°K. Simulated spectra are indicated by broken curves.

arises from the crystalline anisotropy energy and the resultant angular dependence of the resonance field rather than from eddy current effects.

A comparison of both the X-band and K-band spectra at the two extreme temperatures (80°K, 298°K) indicates insignificant changes in the intensity of the spectra with temperature. The peak-to-peak height was found to decrease slightly at the lower temperature (80°K), but this was accompanied by a concomitant increase in the peak-to-peak separation between the resonance maximum and minimum. For the lunar fines (87-11), the area under the absorption curves was shown to remain constant within the experimental error of 5 per cent over the temperature range of 80°–298°K. A careful measurement was carried out to determine the absorption intensity of the lunar fines (10·25 mg) relative to that measured for a 0·102% DPPH sample (9·55 mg, $1·560 \times 10^{18}$ spins/g). This intensity ratio was found to be $1·01 \times 10^5$: 1 at $T = 298°K$ and $\nu = 9·501$ GHz.

The lack of any noticeable temperature variation in the ESR intensities led us to conclude that the spectrum arises from ferromagnetic centers with particle size smaller than the skin depth. Since the observed g-value is very close to the free electron g-value, and is essentially the same at both X-band and K-band, we further concluded that the shape of these ferromagnetic centers is essentially spherical. In our analysis of the observed ESR spectra, we have assumed that the crystal structure of the ferromagnetic centers is cubic, since there is already ample evidence to indicate the existence of high concentrations of metallic Fe in the lunar samples. On the basis of this model and the expressions which we have developed for the analysis of ferromagnetic resonance spectra of polycrystalline materials, a Fortran IV program was written for the IBM 360/75 computer to simulate the first derivative ESR lineshapes. The derivative spectrum was computed from the following expression:

$$\sum_{\theta=0}^{\pi} \sum_{\phi=0}^{2\pi} \frac{K[H - H(\theta, \phi)]W \sin \theta}{[3W^2 + (H - H(\theta, \phi))^2]^2}, \tag{5}$$

where $H(\theta, \phi)$ is the resonance field calculated from equation (2), W is half the peak-to-peak linewidth of the derivative Lorentzian line-shape function, and $\sin \theta$ is the weighting factor which takes into consideration the number of microcrystallites with different polar angles between the crystal axes and the external magnetic field. The factor K was used to normalize the amplitude so that $K \times$ (peak-to-peak height) $\times (2W)^2 = 1$, but was usually omitted in the calculations where the same lineshape function was employed for all the individual resonance absorptions. The sum in equation (5) is taken over all the individual resonance absorptions that occur at the angles (θ, ϕ), from $\theta = 0$, $\phi = 0$ to $\theta = \pi$, $\phi = 2\pi$. However, since the resonance field $H(\theta, \phi)$ is symmetric about $\theta = \pi/2$ and has fourfold symmetry about the z-axis with planes of symmetry at $\phi = \pi/4$ and $\phi = 3\pi/4$, it was only necessary to include in the sum contributions from $\theta = 0$, $\phi = 0$ to $\theta = \pi/2$, $\phi = \pi/4$ in the computation of the composite spectrum.

Attempts to fit the observed spectra were made by varying the g-value, the crystalline anisotropy energy $2K_1/M_z$, and $2W$, the linewidth of the individual resonances. As expected, the overall spectral width of the composite spectrum was extremely sensitive to the crystalline anisotropy energy. Once the best fit was obtained for the observed derivative lineshape, a second program was generated to convert the derivative spectrum into an absorption curve, from which the absorption intensity was calculated. A comparison of computer simulated spectra with the experimental spectra at X-band and K-band is presented in Fig. 2 and 3. The agreement can be seen to be satisfactory.

The results of these spectral analyses are summarized in Table 1. It is noteworthy that at given temperature, similar values of the g-value, $2K_1/M_z$, and W were extracted from the X-band and K-band spectra. We feel that these results lend support to the model which we are proposing. If our model is correct, the g-value should also be isotropic, independent of magnetic field, as observed, and we would also expect the crystalline anisotropy energy to decrease with increasing temperature as has been found for other ferromagnetic materials.

Table 1. Ferromagnetic resonance data for the lunar fines (10087-11)

	X-band frequency ($\nu = 9\cdot501$ GHz)		K-band frequency ($\nu = 34\cdot825$ GHz)	
	298°K	80°K	298°K	90°K
g-value	$2\cdot08 \pm 0\cdot03$	$2\cdot09 \pm 0\cdot03$	$2\cdot08 \pm 0\cdot03$	$2\cdot09 \pm 0\cdot03$
$2K_1/M_z$	$+500 \pm 50$ G	$+640 \pm 50$ G	$+500 \pm 50$ G	$+610 \pm 50$ G
$2W*$	370 ± 40 G	440 ± 40 G	410 ± 40 G	450 ± 40 G

* Linewidth of Lorentzian lineshape function.

As we had anticipated, angular anomalies contribute substantially to the overall lineshape of the observed ESR spectra. In Fig. 2 we have plotted the angular dependence of the resonance field $H(\theta, \phi)$ at X-band frequency. It is evident that the high field portion of the spectra arises principally from the superposition of resonance absorptions due to angular anomalies. In particular, the spectral minimum at the high field end of the spectrum arises from the angular anomaly that

occurs at $\theta = 54°44'$ and $\phi = 45°$, or at the angle where the external magnetic field is aligned along the [111] direction of a cubic crystal. The spectral maximum at the low field part of the spectrum can be seen to originate from those microcrystallites whose cubic edges are parallel to the external magnetic field. Since the peak-to-peak separation between the spectral maximum and minimum is proportional to the crystalline anisotropy energy, as is readily ascertained from our expression for the resonance field, the observed increase in this separation upon decreasing the temperature simply reflects the greater crystalline anisotropy energy at the lower temperature. We note that at a given temperature, the overall width of the ESR spectrum is independent of the frequency of observation; for example, at room temperature, the peak-to-peak separation between the spectral maximum and minimum is 840 ± 50 gauss at both X-band and K-band. This observation is also borne out by the theory, as the angular anomalies which arise from the crystalline anisotropy energy are independent of the magnetic field in contrast to those due to anisotropies in the electron Zeeman interaction or the nuclear hyperfine interaction (ROLLMANN and CHAN, 1969).

DISCUSSION

It is well known that the absorption intensity in ESR experiments is proportional to the population difference between the lower ($M_s = -1/2$) and upper ($M_s = 1/2$) Zeeman states. When the experiments are undertaken at a constant frequency with field sweep as they are in this work, this difference is given by

$$\Delta N = N_-(-1/2) - N_+(1/2) = N \left(\frac{1 - e^{-h\nu/kT}}{1 + e^{-h\nu/kT}} \right) \simeq Nh\nu/2kT, \qquad (6)$$

where N is the total number of unpaired electrons and we have assumed that the splitting between the Zeeman levels is much smaller than kT, i.e. $h\nu/kT \ll 1$. The absorption intensity for paramagnetic absorptions is thus temperature dependent, increasing with decreasing temperature, and dependent on the frequency of observation. Since the Zeeman splitting is of the order of 1 cm^{-1} or less, we expect this expression to hold, except at extremely low temperatures, where the more exact expression must then be used. In this limit, $\Delta N \simeq N$, as all the unpaired electrons are occupying the lower ($M_s = -1/2$) state.

In a ferromagnetic system the electron spins within a ferromagnetic domain are strongly exchange coupled to each other, resulting in a total magnetic moment which tends to align itself along the direction of an external polarizing magnetic field. Hence the ferromagnetic resonance absorption intensity is expected to be proportional to the total number of electron spins only and independent of the temperature at temperature sufficiently below the Curie point as well as the magnetic field at sufficiently high field strengths. Since $h\nu/2kT \simeq 0.75 \times 10^{-3}$ for an ESR experiment at room temperature and X-band frequency, the ferromagnetic resonance absorption intensity of a ferromagnetic material is roughly three orders of magnitude greater than the ESR absorption intensity of a paramagnetic system for the same number of electron spins.

If we assume that the ESR spectrum which we have observed for the lunar samples is paramagnetic in origin, then the number of unpaired electrons calculated

for the lunar fines (87-11) is $2 \cdot 5 \times 10^{-3}$ mole/g based on the absorption intensity ratio of $1 \cdot 01 \times 10^5 : 1$ determined for the lunar fines (87-11) relative to the $0 \cdot 102\%$ DPPH sample at $T = 298°\text{K}$ and $\nu = 9 \cdot 501$ GHz. However, the number of unpaired electrons would become $2 \cdot 5 \times 10^{-3} \times (h\nu/2kT) = 1 \cdot 91 \times 10^{-6}$ mole/g if the absorption signal is essentially ferromagnetic in nature. Thus if the ESR signals observed here were due to paramagnetic Fe^{3+}, as has been suggested by Weeks et al. (1970a, b) and Geake et al. (1970a, b), our intensity measurements would indicate that the lunar fines contain at least 270 per cent Fe^{3+} by weight, which is apparently not the case. Runcorn and his coworkers (1970a) have suggested that there is roughly $1 \cdot 25\%$ Fe^{3+} ions present in the lunar fines (10084-13). On the other hand, if these same results were interpreted in terms of ferromagnetic centers of metallic Fe, our calculations would show that there is about $0 \cdot 50 \pm 0 \cdot 05$ wt. % metallic Fe in the lunar fines (87-11), which is about the same order of magnitude as that determined by other methods (Nagata et al., 1970; Strangway et al., 1970; Runcorn et al., 1970a; Herzenberg and Riley, 1970).

The above conclusions are confirmed by the lack of any noticeable temperature dependence of the ESR absorption intensity of the lunar samples. Computer simulation of the lineshapes employing a model consisting of small sphere of ferromagnetic centers with cubic symmetry appears to fit the observed signals. Our calculations show that the broad, asymmetric spectrum is the result of overlapping resonance absorptions due to microcrystallites with different orientations of the crystal axes relative to the externally applied magnetic field. Our model indicates that the peak-to-peak separation between the spectral maximum and minimum is proportional to the crystalline anisotropy energy and as such should increase with decreasing temperature, since the crystalline anisotropy energy is known to increase with decreasing temperature at temperatures below the Curie point. Experimentally, the linewidths do become somewhat broader and more asymmetric at lower temperatures in agreement with the prediction of the model. The determined g-value of $2 \cdot 09 \pm 0 \cdot 03$ and the crystalline anisotropy energy of $+500$ oersted (at $298°\text{K}$) and $+640$ oersted (at $80°\text{K}$) further suggest that the ferromagnetic centers in the lunar sample consist mainly of metallic Fe (Kittel, 1948). On the basis of the known skin depth of metallic Fe (Kittel, 1948) and our failure to detect eddy current effects in our ESR experiments at both X-band and K-band, we set an upper limit of 1 μm dia. for the size of the metallic Fe particles.

The metallic Fe content in the lunar samples can also be estimated from static magnetic susceptibility measurements by comparing the saturation magnetization of the lunar sample with that of pure iron, provided that the saturation magnetization of $217 \cdot 8$ emu/g for pure iron still holds for the lunar samples (Strangway et al., 1970). To confirm that our particular lunar sample has essentially the same magnetization as those previously examined by others, we have carried out similar static susceptibility experiments using a Princeton Applied Research FM1 vibrating sample magnetometer. The results of our susceptibility measurements are summarized in Fig. 4. Both the field and temperature dependences of the induced magnetization for the fine sample (87-11) were found to be in agreement with those reported for the samples 10084-13, 89, 90 (Runcorn et al., 1970b; Nagata et al., 1970; Strangway et al., 1970), indicating that the magnetic centers in these lunar
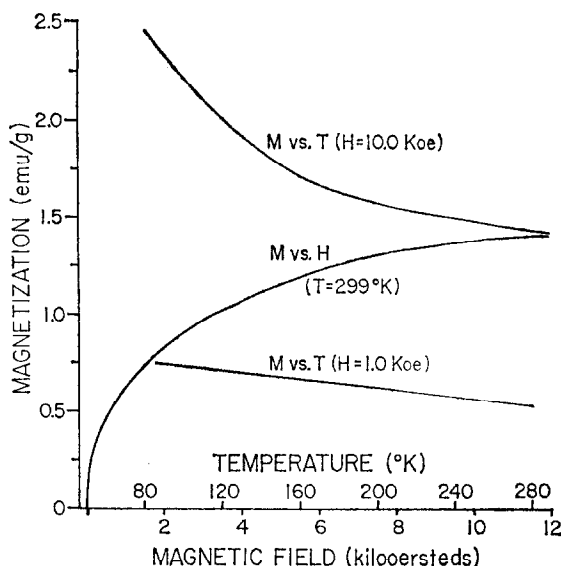
Fig. 4. Induced magnetization for the lunar fines (10087-11). Top curve: temperature dependence at high field (H = 10,000 oersteds); center curve: field dependence at 299°K; and bottom curve: temperature dependence at low field (H = 1000 oersteds).

fines are similar among themselves. It is clear from the field dependence of the magnetization (center curve in Fig. 4) that the metallic Fe does make a major contribution to the magnetization of the lunar fines. The saturation magnetization for sample 87-11 was found to be 1·0 emu/g as estimated from the field dependence of the magnetization (center curve in Fig. 4) and assuming that saturation occurs at about 3000 oersteds. The temperature dependence of the magnetization at high field (top curve in Fig. 4) seems to follow Curie law down to 80°K, but it more likely obeys Curie–Weiss law with the constant θ close to the Néel temperature of the ilmenite (θ = 64°K) (STRANGWAY et al., 1970), which is known to be antiferromagnetic.

The metallic Fe content determined for the lunar fines (10087-11) by our ESR and static magnetic susceptibility measurements are summarized in Table 2.

Table 2. Metallic Fe content in lunar fines

Sample	Metallic Fe (wt. %)	Method of determination	Reference
10087-11	0·50 ± 0·05	a	Present work
	0·45 ± 0·05	b	Present work
10087-4	0·5	c	HERZENBERG and RILEY (1970)
10084-89	0·6	b	NAGATA et al. (1970)
10084-90	0·28–0·53	b	STRANGWAY et al (1970)
10084-13	0·5	b	RUNCORN et al. (1970b)
10084-85	0·71 ± 0·07	c	HOUSLEY et al. (1970)

(a) ESR measurements.
(b) Magnetic susceptibility measurements.
(c) Mössbauer measurements.

For sake of comparison we have also included data previously reported by other investigators for the lunar fines. It is gratifying to note the agreement between our two determinations as well as the general overall accordance between our values with those determined employing similar as well as different methods in other laboratories.

CONCLUSIONS

We have shown that the ESR spectra observed for the lunar samples returned by the Apollo 11 mission are ferromagnetic in origin. Our conclusions are based on the evidence that the ESR absorption intensities observed for these samples are at least three orders of magnitude greater than those expected for paramagnetic Fe^{3+} ions, and that no significant temperature dependence was observed for the ESR spectral intensities over a wide range of temperatures. Moreover, we were able to simulate the observed polycrystalline spectra on the basis of a model which assumes that the spectrum arises from ferromagnetic centers roughly spherical in shape with cubic crystal structure and with diameter less than 1 μm. On the basis of the g-value and the crystalline anisotropy energy obtained from the detailed analysis of these polycrystalline spectra, we concluded that the ferromagnetic centers are principally metallic Fe particles, and if so, are present to the extent to $0.50 \pm 0.05\%$ by weight, in agreement with an abundance of data previously reported by other workers using other methods of analysis. We note that this conclusion is in serious disagreement with that reached by WEEKS et al. (1970a, b) and GEAKE et al. (1970a, b), who have interpreted their ESR data of the lunar fines primarily in terms of paramagnetic Fe^{3+} ions.

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