

# THE $g$ -FACTOR IN PURE POLYCRYSTALLINE IRON

Z. FRAIT

*Institute of Physics, Czechosl. Acad. Sci., Prague\**)

In order to obtain exact values of the spectroscopic splitting factor ( $g$ -factor) in pure polycrystalline iron, measurements of ferromagnetic resonance and antiresonance in thin foils have been performed in parallel configuration at frequencies 26, 36 and 70 GHz, at room temperature. The results confirm the independence of the  $g$ -factor on frequency in the given range and yield the value  $g = 2.088 \pm 0.008$ , in good agreement with measurements of single crystals and with corrected data of other authors.

## 1. INTRODUCTION

The spectroscopic splitting factor ( $g$ -factor) of ferromagnets is related to the ratio of the orbital and spin contributions to the total magnetization and may be evaluated from measurements of ferromagnetic resonance (FMR). In such experiments use is made of the methods of microwave spectroscopy which are, in principle, very exact. However, the existing values of the  $g$ -factor in pure iron at room temperature obtained on polycrystalline samples [1–8] vary from 2.00 to 2.16, i.e. in a range much broader than the reported limits of experimental accuracy (usually  $\pm 1\%$ ). In order to obtain more exact data we have performed measurements of ferromagnetic resonance and antiresonance on samples in the form of thin discs at room temperature, using microwave frequencies in the interval from 26 to 70 GHz. The parallel configuration of FMR experiment has been used, i.e. the external static magnetic field is parallel to the sample plane. From these measurements we have computed the values of  $g$ , considering as far as possible all effects which influence the evaluation of experimental data, mainly the shift of the resonance field caused by the finite penetration depth of the microwave field into the ferromagnetic metal, and the shift due to the average value of effective field of magnetocrystalline anisotropy of individual grains in the polycrystal. The method used by us in the process of evaluation of the resonance and antiresonance experiments is confirmed by the results obtained from FMR measurements on nearly perfect iron single crystals. Finally, our results are compared with the gyro-magnetic ratio measurements and with the FMR measurements of other authors and the existing discrepancies are discussed.

## 2. MEASUREMENTS AND THEIR EVALUATION

The iron samples were in the form of discs with diameter of approx. 20 mm, which were cut out of a foil 6  $\mu\text{m}$  thick. The foil has been prepared by cold rolling from an ingot of polycrystalline iron in several steps, and contained up to tens of ppm of impurity atoms (Mn, K, Na, Si, Ca, Mg, C) [9]. The samples were finally annealed in pure hydrogen at 600 °C during 24 hours, after annealing the surface was not treated mechanically nor chemically. The samples were covered by a copper foil with a central hole of 2 mm diameter, in order to limit the microwave field only to the

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\*) Na Slovance 2, 180 40 Praha 8, Czechoslovakia.

central part of the sample plane. This insures that at all microwave frequencies used for measurements the same part of the sample is examined; moreover, it eliminates the possible influence of demagnetizing field inhomogeneity, which may disturb FMR measurements in samples of non-ellipsoidal shape [10].

The FMR measurements were performed at 22 °C on three microwave spectrometers working in the  $K_a$  (approximately 26 GHz),  $Q$  ( $\approx 36$  GHz) and  $V$  ( $\approx 70$  GHz) bands. The microwave radiation was generated by low power (5–100 mW) klystron oscillators, and its frequency was measured by using resonant cavity wavemeters. The cavities were calibrated with an accuracy of  $1 \times 10^{-4}$  by means of harmonic frequencies of frequency-stabilized microwave source working in the X band (8–12 GHz), the frequency of which was measured exactly (with accuracy of  $1 \times 10^{-7}$ ) by means of a microwave frequency counter. The samples formed the termination (short) of a waveguide and their surface plane was directed along the dc magnetic field direction (so called parallel configuration of the experiment). The static magnetic field was modulated with a small amplitude (approx. 0.1 G, 0.01 mT) auxiliary high-frequency magnetic field (at 115 kHz); the microwave energy reflected by the sample was decoupled by a directional coupler and was detected by a silicon diode. The diode output ac voltage was amplified by a narrow band amplifier and led to a lock-in detector. The derivative of the real component of the surface impedance (in arbitrary units) was observed as a function of the applied static field, the measured curves were registered by means of an X–Y recorder. The static field was generated by a 15" electromagnet, and was stabilized by means of a thermostatically controlled Hall probe (Varian Mark-II probe) with a stability of  $1 \times 10^{-6}$ . The controls of the field stabilizer were calibrated by a NMR gaussmeter using  $H_2O$  and  $D_2O$  probes doped with paramagnetic relaxators. The accuracy of the determination of resonance field values  $B_{res}$  is limited to  $\pm 5$  G (0.5 mT) by the relatively large FMR linewidth of the samples (around 400 G, 0.04 T).

In the experiments the resonance (antiresonance) field intensity  $B_{res}(B_{ar})$  was determined at the given frequency of the microwave field  $\omega$  as the maximum (minimum) value of the real component of surface impedance of the sample. The  $g$ -factor was calculated from the experimental data by means of the Kittel's FMR resonance and antiresonance equations, which were used in the form

$$(1) \quad \omega^2 = \gamma^2 (B_{res} - B_d + \delta B - B_a) (B_{res} - B_d + \delta B - B_a + 4\pi M),$$

$$(2) \quad \omega^2 = \gamma^2 (B_{ar} - B_d + \delta B - B_a + 4\pi M)^2.$$

where  $\gamma = g\mu_B/\hbar$  is the gyromagnetic ratio,  $\mu_B$  the Bohr magneton,  $2\pi\hbar$  the Planck constant,  $g$  the spectroscopic splitting factor ( $g$ -factor),  $B_d$  the intensity of the demagnetizing field of the sample,  $\delta B$  the field shift caused by the exchange-conductivity mechanism,  $B_a$  the mean value of effective fields of magnetocrystalline anisotropy,  $M$  is the saturation magnetization of the material. Resonance equation and formulas are quoted in the Gaussian CGS system of units, for detailed definition of the magnetic field intensity  $B$  see e.g. [11].

In our case of very flat disc the  $B_a$  field can be computed by using the approximate formula for flat ellipsoid of rotation [12],  $B_a = M\pi^2 b/a$ ,  $b(a)$  is the thickness (diameter) of the sample; by taking  $M = 1700$  e.m.u. (2.135 T) [13] we obtain  $B_a = 5$  G (0.5 mT). The resonance and antiresonance field shifts  $\delta B$  are functions of frequency  $\omega$ , magnetization  $M$ , resistivity  $\rho$ , exchange constant  $A$ , damping constant  $\lambda$  and surface anisotropy  $K_s$  of the material, and their values were evaluated by means of a computer using the macroscopic theory developed in [14]. For the case of pure iron  $\delta B$  values were obtained in the range from 52 G (5.2 mT) at 26 GHz to 86 G (8.6 mT) at 70 GHz using  $\rho = 9.7 \mu\Omega \text{ cm}$  [15],  $\lambda = 4.2 \times 10^7 \text{ rad/sec}$  [16],  $A = 2 \times 10^{-6} \text{ erg/cm}$  (20 pJ/m) [17],  $K_s = 0.03 \text{ erg/cm}^2$  (3  $\mu\text{J/m}^2$ ) [16]. The  $B_a$  value was calculated by means of the theory presented by STANDLEY and STEVENS [3]. Although this theory was formulated for samples of spherical form, ASCH [18] proved its validity for disc-shaped specimen. The formula for  $B_a$  is given in [3] as  $B_a = k 2M/K_1$ , where  $K_1$  is the constant of magnetocrystalline anisotropy,  $k$  is a function of the FMR linewidth of individual grains (single crystals) of the polycrystalline samples,  $k = 0$  for infinite linewidth,  $k = 1$  for zero-width curve. As the single-crystalline linewidth is a function of frequency,  $B_a$  is also frequency dependent. By using the half-power line-width values measured on single crystals of pure iron [16] and the value of magnetocrystalline anisotropy  $K_1 = 4.86 \times 10^5 \text{ erg/cm}^3$  (4.86 kJ/m<sup>3</sup>) [13], we have computed  $B_a$  values from [3] as 140 G (14 mT) at 26 GHz, 136 G (13.6 mT) at 36 GHz and 127 G (12.7 mT) at 70 GHz.

### 3. RESULTS AND DISCUSSION

The FMR measurements were performed on four samples at the frequency 68.931 and 36.165 GHz, two of the samples were also measured at 26.000 GHz. At the highest frequency the mean resonance field intensity was established as 15251 G (1.5251 T) with standard deviation (SD) 8 G (0.8 mT), the antiresonance field value was measured at this frequency only as 2300 G (0.2300 T) with SD 18 G (1.8 mT). Using these data the  $g$ -factor value was determined by means of the method described above as  $g = 2.088$ , its accuracy was estimated as  $\pm 0.008$  taking into account both the accuracy of measurements and the standard deviations of resonance field. For the magnetization of pure iron we have obtained  $M = 1700 \pm 8$  e.m.u. ( $2.130 \pm 8 \times 10^{-3}$  T). The resonance measurements at 36 GHz (with  $B_{\text{res}} = 5740$  G (0.5740 T), SD 18 G (1.8 mT)) yielded  $g = 2.088$ , and at 26 GHz (with  $B_{\text{res}} = 3224$  G (0.3224 T)) gave  $g = 2.087$ . This confirms well the frequency independence of the  $g$ -factor.

The  $g$ -factor value determined by us on polycrystalline pure iron samples was compared with studies performed on single crystals and was found in a good agreement with  $g = 2.089 \pm 0.007$ , the value of which has been obtained from FMR experiments in a broad frequency interval (12–70 GHz) on whisker type crystals [13]. Also the saturation value  $M = 1700$  e.m.u. (2.120 T) agrees well with the data

found from FMR single crystal measurements ( $M = 1699 \pm 2$  e.m.u.,  $2.130 \pm 20$  T) [13] and is only slightly less than the values determined from static measurements  $M = 1707$  e.m.u. (2.143 T) [7],  $M = 1708$  e.m.u. (2.145 T) [20, 21],  $M = 1714$  e.m.u. (2.152 T) [19]. The agreement of  $g$ -factors for single- and polycrystalline material also shows that we have taken properly into account the effects of demagnetization, magnetocrystalline anisotropy and exchange conductivity mechanism. The difference between the magnetization data is probably caused by planar stress, which may originate in the process of sample preparation.

Concerning FMR measurements performed on polycrystalline iron by other authors, BAGGULEY [1, 2] measured  $g$ -factors in colloidal suspensions at 9 and 24 GHz; he explains the wide spread of the obtained values (2.00–2.16) by defective structure of some of the colloids (partial oxidation of ferromagnetic particles occurred in some cases), and he quotes for good samples  $g = 2.06 \pm 0.02$ . It is difficult to discuss his results in detail mostly because of the complex problem of exact determination of internal static magnetic field intensities acting on a single particle in the colloidal system. On polycrystalline bulk samples STANDLEY and STEVENS [3] obtained  $g = 2.07 \pm 0.02$  BARLOW and STANDLEY [4]  $2.09 \pm 0.02$ , ASCH [5]  $2.09 \pm \pm 0.02$ , MEYER and ASCH [6]  $2.09 \pm 0.016$ . These measurements agree well with our results within the limits of given accuracy; in [3] and [4] an inadequate (too large and frequency independent) value of single crystalline line-width was used in calculations of the  $B_a$  shift.

Another determination of  $g$  in polycrystalline bulk samples has been performed by FISCHER, he quotes  $g = 2.113 \pm 0.009$  at room temperature and at 35 GHz [7, 8]. This result should be, however, slightly corrected because of two effects. In the first place his field shift values  $B_a$  caused by the magnetocrystalline anisotropy were calculated by using too large linewidth values for single crystals, and are too small;  $B_a$  value of approx. 132 G (13.2 mT) should be used. Then, in [7, 8] a very rough approximation for the demagnetizing field  $B_d$  of the samples has been applied, namely the formula for flat ellipsoids [11]. As in Fisher's case the ratio of the thickness and the diameter of the sample is relatively high (0.0287), the approximation of the sample shape by an ellipsoid is not justified [22]. By taking into account the results of experimental and theoretical studies of demagnetizing effects in FMR of disc-shaped specimens [22] we have determined  $B_a = 315$  G (31.5 mT) instead of the value 484 G (48.4 T) used by FISCHER. Introducing these corrected values of  $B_a$  and  $B_d$  into his calculations, one obtains  $g = 2.090 \pm 0.009$  in a very good agreement with our results.

Finally, our result also agrees very well with the  $g$ -factor value, which we have computed from gyromagnetic factor ( $g'$ ) measurements performed by SCOTT and STURNER [23],  $g' = 1.919 \pm 0.002$ . By using the Kittel - Van Vleck formula  $g'^{-1} + + g^{-1} = 1$  we get from [23]  $g = 2.088 \pm 0.002$ .

#### 4. CONCLUSION

The  $g$ -factor (spectroscopic splitting factor) value of pure polycrystalline iron has been obtained by measurements of ferromagnetic resonance and antiresonance on thin polycrystalline foil, which was statically magnetized in the plane of the specimen. The experiments were performed at microwave frequencies ranging from 26 to 70 GHz, at room temperature, and yield  $g = 2.088 \pm 0.008$ . This value of  $g$ -factor is in a very good agreement with the measurements of FMR parameters on single crystals of pure iron, with corrected results of previous  $g$ -factor studies on iron polycrystals, and with gyromagnetic factor measurements on pure polycrystalline iron. This agreement shows, that in our case the influence of demagnetization, of magnetocrystalline anisotropy and of exchange-conductivity mechanism has been properly included into the process of  $g$ -factor evaluation from the experimental data.

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