# MAGNETIC ANISOTROPY OF DISPERSED POWDERS\*

## By F. D. STACEY<sup>†</sup>

It is well known that in most rocks the ferromagnetic fraction occurs as small grains dispersed in a solid, magnetically inert matrix. Recently the magnetic anisotropy of rocks and of chondritic meteorites has been subjected to detailed study by the torque-meter method, and, in an attempt to obtain a physical understanding of the shape and crystal alignments of grains which cause magnetic anisotropy in these natural bodies, a number of artificial specimens have been prepared. Iron, nickel, and magnetite powders were mixed into solidifying media and allowed to set in a cylindrical mould in a 10 kilo-oersted field. The torque curves of the resulting specimens reveal a surprising result. It appears that single crystal magnetic grains tend to string together along lines of force, thus producing strongly anisotropic specimens, only when the first magnetocrystalline anisotropy constant of the ferromagnetic material is positive.

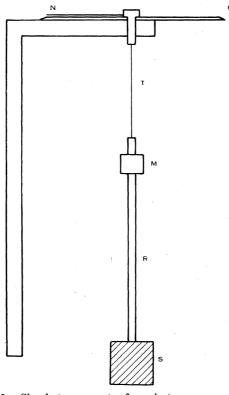
## The Torque-meter Experiment

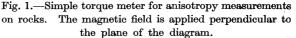
Torque meters are generally used to measure magnetic anisotropy, and Bozorth (1951) has summarized the method and the results which have been obtained with single crystals. For measurements on rocks the simple instrument shown in Figure 1 is adequate. The torsional suspension T is a 7.5 cm length

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of 0.45 mm diameter phosphor-bronze wire, hanging from a needle N, which can be turned by hand round a 360° scale H in a horizontal plane. A brass rod Rwith an adjustable mirror M clipped to it is soldered to the lower end of the suspension; cylindrical specimens are stuck to this rod with wax and hang freely. Only very strongly magnetic, light specimens need to be weighted. The torque meter is placed so that the specimens hang symmetrically in the field gap of an electromagnet (Stacey 1959), which is used in this experiment with 10.8 cm





plane pole faces and normally operated at 10.6 kilo-oersteds over a gap of 4.0 cm. The mirror is used with a galvanometer lamp and scale for sensitive observations of the angle of the specimen.

To make a measurement the needle is set to a predetermined angle  $\theta$  and the mirror adjusted to bring the light spot into the middle of the scale. The field is switched on and the rotation of the specimen is compensated by turning the needle through an angle  $\Delta \theta$  to return the light spot to its original position. Then  $\Delta \theta$  is the angle through which the suspension has been twisted to oppose exactly the magnetic torque on the specimen. This process is repeated at  $\theta=0^{\circ}$ ,  $10^{\circ}$ ,  $20^{\circ}$ , . . ., to  $180^{\circ}$ , and sometimes to  $360^{\circ}$  as a check. Then  $T=\alpha\Delta\theta$  versus  $\theta$ is the torque curve of the specimen, where  $\alpha$  is the torsional constant of the

	$T_4/T_2$		-0.16 $\pm 0.02$	$\pm 0.15$	-0.12 $\pm 0.02$	-0.16 $\pm 0.02$	$\frac{-0.14}{\pm 0.015}$	−0·07 ±0·07		$\pm 0.015$	0.015		
TABLE 1 ANALYSED COMPONENTS OF TORQUE CURVES All measurements at 10.6 kilo-oersteds except where noted for specimen 3. Amplitudes $T_2$ , $T_4$ , $T_6$ are in dyne-cm.	Per cm <sup>3</sup> of Ferromagnetic	$T_{6}$	$0.4 \times 10^{5}$		$0.4 \times 10^{5}$	$0.7 \times 10^{5}$	$0\cdot 2  imes 10^5$	<u> </u>		$0.6 \times 10^{5}$	1		
		$T_4$	$-2.3 \times 10^{5}$	$-2\cdot6\times10^{5}$	$-2.0 \times 10^{5}$	$-2.4 \times 10^{5}$	$-1.0 \times 10^{5}$	$-0.02 \times 10^{5}$	$<0.006 \times 10^{5}$	$-2 \cdot 1 \times 10^{5}$	$-12.9 \times 10^{5}$	$< 0.005 \times 10^{6}$	
		$T_2$	$14 \cdot 4 \times 10^{5}$	$17 \cdot 1 \times 10^{5}$	$17 \cdot 1 \times 10^{5}$	$14.5 \times 10^{5}$	7 · 17 × 105	$0\cdot 24  imes 10^5$	$< 0.006 \times 10^{5}$	$11 \cdot 0 \times 10^{5}$	$57 \cdot 0  imes 10^{5}$	$0\cdot 02_6  imes 10^5$	
	Total	$T_{6}$	$0 \cdot 17 \times 10^{3}$		$0.3  imes 10^3$	$0.4 \times 10^3$	$0\cdot 1_2  imes 10^3$			$1\cdot4 imes10^3$			- 
		$T_4$	$\binom{-1\cdot 0}{\pm 0\cdot 1}  imes 10^3$	$\left(\frac{-3\cdot3}{\pm0\cdot5}\right)\times10^3$	$\left(\frac{-1\cdot 3}{\pm 0\cdot 15}\right)\times 10^3$	$\left(\frac{-1.5}{\pm 0.15}\right) \times 10^3 0.4 \times 10^3$	$\left(\frac{-0.63}{\pm 0.06}\right) \times 10^{3} 0.1_{2} \times 10^{3}$	$-30\pm30$	<10	$\left(\frac{-4\cdot8}{\pm0\cdot4}\right)\times10^3$	$\left( \frac{-7 \cdot 3}{\pm 0 \cdot 5} \right) \times 10^3$	<10	
		$T_2$	$\binom{6 \cdot 1}{\pm 0 \cdot 1} \times 10^{3}$	$\left( {\begin{array}{*{20}c} {21 \cdot 7} \\ \pm 0 \cdot 5 \end{array}  ight)  imes 10^3$	$\left( {\begin{array}{*{20}c} 10.9 \\ \pm 0.15 \end{array}}  ight) \!  imes \! 10^3$	$\left(rac{9\cdot2}{\pm0\cdot15} ight)\! imes\!10^{3}$	$\left(\frac{4\cdot 55}{\pm 0\cdot 06}\right) \times 10^{3}$	$\left( {\begin{array}{*{20}c} 0.40 \\ \pm 0.03 \end{array}}  ight)  imes 10^3$	<10	$\binom{25 \cdot 0}{\pm 0 \cdot 4} \times 10^{3}$	$\left(rac{32\cdot4}{\pm0\cdot5} ight) imes10^3$	$50\pm 20$	
	Matrix		Household cement	Plaster of paris	Plaster of paris		Plaster of paris	Plaster of paris	Plaster of paris	Benzoic acid+ pyrex powder (solidification point 122 °C)	". Armo wax '.+ pyrex powder (solidification point 132 °C)	Plaster of paris	
	Quantity (mg)		32.6	100	50		50	150	150	200	50	100	rsteds.
			Two <sup>1</sup> / <sub>32</sub> in. balls	Mond carbonyl grade MCP	Mond carbonyl grade MCP		Mond carbonyl grade MF	Mond carbonyl grade A	S h e r r i t t- Gordon grade C50	Sherritt- Gordon grade C50	Sherritt- Gordon grade C50	BDH artificial powder	* Same specimen at 6.5 kilo-oer
	Speci- Material		Steel	Iron	Iron		Iron	Nickel	Nickel	Nickel	Nickel	Magnetite	me specime
			1	67	e	3*	4	5	9	2	8	5	* Sa

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suspension.  $\Delta \theta$  can be measured to about  $0.2^{\circ}$ , which is somewhat less than 1 per cent. of the maximum normally usable deflection. For very weakly anisotropic specimens the direct observation of  $\Delta \theta$  is inaccurate and a more sensitive method uses calibrated deflections of the light spot without movements of the needle.

For convenience the artificial specimens are set in the torque meter so that their known axes of anisotropy coincide with the field direction when the scale reading is zero. Their torque curves can then be resolved into even harmonics in  $\theta$ :

$$T(\theta) = T_2 \sin 2\theta + T_4 \sin 4\theta + T_6 \sin 6\theta + \dots \quad \dots \quad (1)$$

Small disturbing terms  $T_0$  and  $T_1 \sin(\theta + \varphi)$ , ( $\varphi$  representing arbitrary phase) may also appear. The  $T_0$  term is due to rotational hysteresis and is generally observable only at low fields (a specimen at  $\theta + 10^\circ$  tends to "remember" the magnetization it acquired at  $\theta$  and thus experience a torque independent of  $\theta$ ). The  $T_1$  term may be observed with specimens having unevenly distributed magnetic material, suspended asymmetrically in the field. It has been noticed with a few rocks, but with none of the artificial specimens.

### **Experimental** Results

It is to be expected that spherical, polycrystalline grains of ferromagnetic material, immersed in a non-magnetic paste and exposed to a strong magnetic field, will tend to align themselves in strings along the lines of force. The specimen which is formed when the paste has set will have a magnetic anisotropy which may be simulated by a pair of individually isotropic ball-bearings stuck together as specimen 1 (see Table 1). The similarity of the torque curves of the other specimens to that obtained with specimen 1 is a strong indication that the observed anisotropies were principally due to the stringing together of magnetic particles.

However, in spite of the similarity of shape of all the torque curves, their amplitudes differ most remarkably. The specimens of nickel and magnetite powder, which were mixed with about 1000 times their own volume of plaster of paris (5, 6, and 9), wetted, and allowed to set in the field for about 5 hr, exhibited very little or no anisotropy, whereas the iron specimens (2, 3, and 4), prepared in the same way, were strongly anisotropic.

We may consider the behaviour of a specimen containing single (cubic) crystal magnetic particles which rotate in the setting field to give [100] crystal alignment with the field and random crystal directions in the perpendicular plane. Neglecting the stringing effect it would have a torque curve given by

where  $K_1$  and  $K_2$  are the first and second magnetocrystalline anisotropy constants of the material. Since for iron  $K_1 \sim +4 \times 10^5 \text{ ergs cm}^{-3}$  and  $K_2$  is negligible for this purpose, it is apparent that the sign of the sin 40 term in the measured

torque curves is opposite to that which would result from crystal alignment. Further, no distinction can be made between the shapes of the torque curves for the specimens containing iron (2, 3, and 4) and the curve for the pair of ball-bearings (specimen 1), so that crystal alignment does not contribute to the anisotropy of the iron specimens. This is expected from the polycrystalline nature of carbonyl iron powder (Pfeil 1947).

The torque exerted on specimen 3 with its axis of anisotropy at a fixed angle  $(30^{\circ})$  to a variable field H was measured over the range  $3 \cdot 9 - 11 \cdot 5$  kilo-oersteds. It showed the normal linear variation in 1/H above about 8 kilo-oersteds and extrapolated to a value at (1/H=0) 18 per cent. higher than at  $H=10\cdot 6$  kilo-oersteds. Comparison of the torque curves of specimen 3 at  $6 \cdot 5$  and  $10 \cdot 6$  kilo-oersteds showed that it is only the sin  $2\theta$  term which increases with increasing field. The sin  $4\theta$  and sin  $6\theta$  terms decrease in magnitude. The decrease in  $T_6$  is so strong that this term extrapolates to zero at (1/H=0) within the limits of experimental error. The extrapolated ratio  $T_4/T_2$  is approximately  $-0 \cdot 07$ . However, the striking, qualitative difference between the torque curves of the iron and nickel specimens is not influenced by the magnitude of the field in which the measurements are made.

There is another experimentally variable parameter in the particular case of nickel, in which the first magnetocrystalline anisotropy constant is negative at room temperature but becomes positive (iron-like) above about 100 °C. Specimens 7 and 8 were made by mixing weighed samples of nickel powder with pyrex powder and either benzoic acid or "Armo wax" (Armour Chemical Division) to form a thick paste at about 150 °C; the specimens were then allowed to cool in beakers in the 10 ·6 kilo-oersted field, so that they solidified well above 100 °C (see Table 1). The anisotropies of the specimens of nickel powder set in this way were more than 1000 times stronger than for the same grade of powder set in plaster at room temperature.

### Discussion

The shape of the torque curves can be explained almost entirely in terms of a stringing together of grains. It is surprising therefore that of the specimens which had been set at room temperature only those containing iron showed this effect. A correlation with magnetocrystalline anisotropy is indicated by the torque curves of specimens containing nickel powder which had been set above 100 °C (7 and 8) and which showed very strong anisotropy similar to that of the iron specimens. Above about 100 °C the first magnetocrystalline anisotropy constant becomes positive (iron-like) (Honda, Masumoto, and Shirakawa 1935; Bozorth 1951).

A physical difference between the iron and nickel powders must be noted. Carbonyl iron powders are polycrystalline, but when nickel powder is produced "the particles tend to be single crystals and to possess an idiomorphic shape" (Pfeil 1947). Microscopic examination of the magnetite grains used in specimen 9 showed that these also had irregular but characteristic shapes indicative of single crystals. The conclusion may therefore be advanced that polycrystalline magnetic particles (iron, specimens 2, 3, 4) or single crystals with positive magneto-

crystalline anisotropy (nickel set above 100 °C, specimens 7, 8) tend to string together along lines of force when exposed to a magnetic field. Single crystals with negative magnetocrystalline anisotropy (nickel below 100 °C, specimens 5, 6, and magnetite, specimen 9) do not show this effect. No mechanical explanation for this difference in behaviour can be offered at present.

The torque curves can also be examined for details indicative of microcrystalline alignment. For material with positive magnetocrystalline anisotropy this would be most apparent as an increase in the sin 40 term in the torque curve by the addition of a term having the form of equation (2) to the curve due to the stringing effect. This means a decrease in magnitude of the negative coefficient  $T_4$  relative to the magnitude of  $T_2$ . Experimental uncertainty in determining the ratio  $T_4/T_2$  (see Table 1) does not permit any distinction to be made between specimens 2, 3, and 4, so that carbonyl iron powders of grades MCP and MF are evidently both polycrystalline, as mentioned above.

The occurrence of a magnetocrystalline term in the torque curves of specimens 7 and 8 appears possible, particularly in the latter. At high temperatures the nickel powders would experience [100] crystal alignment in the field, so that with this alignment frozen in at room temperature the addition to the torque curve of a term having the form of equation (2) with negative  $K_1$  will increase the magnitude of the negative  $T_4$  term relative to  $T_2$ . The increase in  $(-T_4/T_2)$  which was observed for specimen 8 is well outside experimental error and is consistent with crystal alignment in the nickel powders at high temperatures.

The difference between specimens 5 and 6 is evidently the result of a real physical difference between the nickel powders used. The Sherritt-Gordon grade C50 powder gave no measurable anisotropy at all, whereas exactly the same treatment of a sample of Mond carbonyl grade A powder resulted in a specimen of very small but readily measurable anisotropy. If the foregoing conclusion that anisotropy will only appear if the grains are polycrystalline or have positive magnetocrystalline anisotropy is correct, then it appears that the Sherritt-Gordon powder is entirely composed of single crystal grains, but that the Mond powder contains a small but noticeable fraction of polycrystalline grains.

Powders for these experiments were provided by the Mond Nickel Company, London, and Sherritt-Gordon Mines Ltd., Alberta, Canada.

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