Reprints — Low Temp

Printed Matter

Prof. K. S. Krishnan

Indian Association for the Cultivation of Science

MSS

Calcutta

Graphite

India

Ox[illegible]

Clarendon Lab
Oxford

8 O'clock

It is an Augean task

Wt of block ·2081

C ·0094

carbon per gm of mix = ·0452

Field = 5177

$C = \bar{2}\cdot 1724$

Rotation 293 – 45

Paraffin 4071 Field

wt of block ·2457

Rotation 268 – 45°

$C = \bar{2}\cdot 6757 / 4\cdot 3^2$

$\Delta\chi$ per ·2081 gm of mixture

$$= \frac{11}{90}\left[248 \times \frac{\bar{2}\cdot 1724}{\cdot 2081 \times 5177^2} - \frac{223 \times \bar{2}\cdot 6757 \times \cdot 1987}{4\cdot 3^2 \times \cdot 2457 \times 4071^2}\right]$$

$= \cdot 41$ ✓

and ~~except~~ $\chi_\perp - \chi_\parallel$ ~~i.e.~~
~~[illegible]~~
In view of the importance of this ~~magn~~ quantity ~~constant~~

New para

~~to we repeated~~ the measurements were

repeated by us with a large number of

~~well~~ specially ~~selected~~ chosen crystals. The value
obtained in these measurements = ~~for~~ ~~for different crystals~~

of $\chi_\perp - \chi_\parallel$ ~~were found to ranged from~~
were ~~slightly~~ different for different crystals and ranged from

~~21 × 10~~ to 21 to 22 $\times 10^{-6}$ per gm. ×

+ ~~Such~~ They are in agreement with the

values obtained by Guha and Roy.

3. Susceptibility for Directions in the

Basal Plane

The ~~absolute~~ susceptibility ~~of graphite~~ ~~along any directions~~ ~~of~~ 5 b.1 b

~~Since~~ $\chi_\perp - \chi_\parallel$ ~~is~~ being now known, if we ~~know~~ can measure

in addition ~~[illegible]~~
the ~~absolute~~ absolute value of either $\chi_\perp$ or $\chi_\parallel$, ~~we~~ both
~~of them are known. Since~~ 5
~~the principal susceptibilities are known.~~
~~$\chi_\parallel$~~ ~~$\chi_\perp$~~ however being ~~$\chi_\perp$~~ ~~numerically very much smaller than~~ ~~becomes at almost negligible~~ ~~being one of~~ to measure

$\chi_\parallel$, ~~it~~ ~~being one of~~ to measure the

$\chi_\perp$ rather than $\chi_\perp$ i.e. ~~≠~~

susceptibility of the crystal for directions in the basal plane. ~~An accurate~~

Calculations:-

Let m_1 be the mass of the Block.

m_2 = the mass of Paraffin.

Then $m_1 - m_2$ = mass of carbon present in the block,

K_1 = Volume susceptibility of Block. = $\cdot 6521$

K_2 = of Paraffin = $\cdot 6462$

Then Mass susceptibility of Carbon $= \frac{1}{m_1 - m_2}\left\{\frac{K_1\, m_1}{\rho_1} - \frac{K_2\, m_2}{\rho_2}\right\}$.

$$= \frac{1}{\cdot 2081 - \cdot 1987}\left\{-\frac{\cdot 6521 \times \cdot 2081}{\cdot 913} + \frac{\cdot 6462 \times \cdot 1987}{\cdot 892}\right\}$$

$$= \frac{1}{\cdot 0094}\left\{-\cdot 1486 + \cdot 1440\right\}$$

$$= -\frac{\cdot 0046}{\cdot 0094} = -\cdot 5008.$$

Weight of the Carbon-Paraffin Block = ·2081 gm.

Weight of Carbon Present in the Block = ·0094 gm.

$\chi_{\perp} - \chi_{\parallel}$ for Pure Paraffin = $\cdot 005 \times 10^{-6}$ per gm.

$\chi_{\perp} - \chi_{\parallel}$ for the Carbon-Paraffin Block (taking the wt. of Carbon into Consideration) = $\cdot 51 \times 10^{-6}$ per gm.

$\chi_{\perp} - \chi_{\parallel}$ for the Carbon particles =

Density (ρ_1) of the pure Paraffin Block = ·89

Density (ρ_2) of the Carb.-Para. Block = ·91

Carbon Content (g) per gm. of the Block = ·04517

Volume Susceptibility (κ_1) of the pure Paraffin Block = $-\cdot 6462 \times 10^{-6}$

Mass Susceptibility (χ_1) of the Same Block = κ_1/ρ_1 = $-\cdot 7265 \times 10^{-6}$

Volume Susceptibility (κ_2) of the Car-Par. Block = $-\cdot 6521 \times 10^{-6}$

Mass Susceptibility (χ_2) of the . . . . . . = $-\cancel{\cdot 7269}$

= $-\cdot 7166 \times 10^{-6}$

Mass Susceptibility (χ) for Carbon = $\dfrac{\chi_2 - \chi_1(1-g)}{g} = -\cdot 52 \times 10^{-6}$

Table I.

No. of Crystal	Weight of the Crystal Before treatment	After treatment	$(\chi_\perp - \chi_\parallel) \times 10^{-6}$ per gm. of Carbon	Period of oxidation	$\chi_\perp - \chi_\parallel \times 10^{-6}$ per gm.	$\chi_\perp \times 10^{-6}$ per gm.	Method of Treatment.
1.	·0011 gm.	·0015 gm	21·3	18 hours	1·26	·50	Treated with a mixture of Conc. HNO_3 and H_2SO_4
2.	·0052 "	·0071 "	21·0	7 "	1·27	·50	
3.	·0025 "	·0034 "	20·7	12 "	1·24	·498	"
~~4.~~	~~·0067 "~~	~~·0091 "~~		~~12 "~~	~~1·39~~	~~·49~~	~~"~~
5.	·0020 "		21·2	10 "	1·26	·50	Treated with a mixture of HNO_3, H_2SO_4 + $KClO_3$.
6	·0040 "		21·5	14 "	1·25	·48	

Susceptibility of Graphitic Acid.

Force ~~due~~ on ~~to~~ the empty tube in the field = ·0022 gm.

Wt. of Graphitic Acid taken = 1·1919.

~~Force on the tube + Graphitic Acid~~ =

~~Wt. of~~

Force on the tube + Graphitic Acid taken = ·0048

Force on the tube filled with water = ·0086.

Density of the column of Graphitic Acid in the tube = ·542.

Mean Susceptibility of Graphitic Acid = ·54.

1. Preparation of graphite-paraffin mixture

0.417 gms of graphite was mixed with 33.983 gms of paraffin.

and the blocks were prepared out of this mix by the usual method in magnetic field.

2. Density of the block

A wt. of cryst. in air = 0.2430 gms.

B. wt. of sinker + suspension in air = .2961 gms.

sinker alone = .2869

C. (wt of cryst. + sinker + susp) in air = .5391

D same as C except that sinker + part of its suspension was under water = .4475 gms.

.0916

loss in wt due to sinker.

E same C - both cryst. + sinker with remaining suspension = .1822 gms

.2653

Density = $\frac{.2430}{.2653}$ = 0.9160

.916

Redetermined

3. Volume suscep. of block

A soln of KCl of density 1.1255 balanced $K_\perp$

of standard KCl soln
concentn = ·2832 gms of KCl per c.c. of soln.

~~balanced the block for $K_\perp$.~~

~~suscep = −(·2832 × ·516 + ·720 × ·8423)~~

=

Balancing soln was prepared by ~~addi~~ mix of 20 c.c. of KCl with 6 c.c. of water.

∴ contains 5.664 gms of KCl

28°·6

1.13072
1.12852
·00220 for 5%.
× 3.6
5

1.13072
·00158
20% = 1.12914

·00792
5

·00158 for 3.6

772

1272
636
7632

18%. 1.11494
·01420 per 2%.

1.11647
153

1.11647
1.11435
·00212 × 3.6
5

19.5%.

1.12914
1.1255
0036

·0036
·00718

χ for the paraffin block containing Graphite

powder ——— = ·837 ~~p~~ gm (·766 p.c.c.)

density of block = ·916.

Vol. Sus = (·195 × 1·1255 × ·516 + ·805 × 1·1255 × ·720)

Density of bal. soln = 1·1255 at 28°·6 C.

corresp. concen = 15·5%.

·1006
·5795
·680 × 1·1255 = ·765 per c.c.

Suscep. of paraffin = ~~·2832 × 6·5 × ·516 × 26·5~~

density of balancing soln. 1·042

(·0716 × ·516 + ·9284 × ·720) × 1·042

= ·705 ~~× 10~~ per gm × 1·042

= ·735

Density of paraffin = ·905.

new values

·909
·909
·910

·909

Vol. suscep. of block $\qquad$ s = sp. sus. of graphite

$$\frac{\cdot 417\, s + \dfrac{33\cdot 983 \times \cdot 735}{\cdot 905}}{} = \cdot 765$$

$$\frac{34\cdot 400}{\cdot 916}$$

$$s = \frac{\dfrac{\cdot 765 \times 34\cdot 40}{\cdot 916} - \dfrac{33\cdot 98 \times \cdot 735}{\cdot 905}}{\cdot 417}$$

$$\frac{\begin{array}{r}28.74\\27.60\end{array}}{1\cdot 14} = 2\cdot 7$$

$$\frac{\dfrac{\begin{array}{r}28.74\\27.48\end{array}}{1\cdot 26}}{\cdot 417}$$

3·0

8·73
8·57

11·7
6·0
17·7
5·9

Vol. Susc. of graphite + paraffin block

Density of the balancing soln of KCl = 1.12 ~~08~~ gms/cc

∴ Concn. .2225 gms of KCl } per c.c. of solution.
.9043 gms of H_2O

$$\therefore \chi = -\left(\overset{.1148}{.2225 \times .516} + \overset{.7658}{.9043 \times .720}\right) \times 10^{-6} \text{ per c.c.}$$

$$= -0.766 \times 10^{-6} \text{ per c.c.}$$

Vol. Susc. of the paraffin ~~block~~ oriented in the magnetic field.

Density of balancing KCl soln = 1.0396

concn .0713 gms of KCl
.9683 gms of water

$$\text{vol susc.} = -\left(\overset{.0368}{.0713 \times .516} + \overset{.7339}{.9683 \times .720}\right)$$

$$= -0.734 \times 10^{-6} \text{ per c.c}$$

$$\text{Specific susc. of graphite} = \frac{\dfrac{\overset{\text{vol. sus. of block}}{.766} \times \overset{\text{wt. of paraffin + graphite}}{34.40}}{\underset{\text{density of block}}{.916}} - \dfrac{\overset{\text{paraffin}}{33.98} \times \overset{\text{vol. susc of paraffin}}{.734}}{\underset{\text{density of paraffin}}{.905}}}{.417}$$

9

Calculation of the density of the block

In 1 c.c. of block wt. 0.9160 gms →

~~...~~ wt. of graphite = $\dfrac{.9160 \times .417}{\begin{array}{r}33.983\\ .417\\ \hline 34.400\end{array}}$; vol = $\times \dfrac{1}{2.15}$ = .0052

~~...~~ vol. of paraffin = $\dfrac{.9160 \times 33.98}{34.40 \times .905}$ = $\dfrac{.9983}{1.0035}$

.9086

If we adopt .909 as density

.005
.994
.999

36
x.4
396

99

I.

	Weight of the Crystals (in gms.)	Field Strength (in Gauss.)	Rotation (in degrees).	δx	Mass Susceptibility $\times 10^{-6}$.
1.	·0223	492·7	381·5	21·2 21·21 ~~22·5~~ 21·04	·4940 $\times 10^{-6}$.
2.	·0177	492·7	313·7	21·3 21·29 ~~21·16~~ $\times 10^{-6}$	·4931 $\times 10^{-6}$
3.	·0315	492·7	525	21·4 21·42 ~~21·24~~ $\times 10^{-6}$.	·4944 $\times 10^{-6}$.
4	·0517	492·7	600·	21·1 21·06 ~~22·66~~ $\times 10^{-6}$	·5064 $\times 10^{-6}$
5.	·0190	436·6	272·5	21·3 21·26 $\times 10^{-6}$	·4955 $\times 10^{-6}$
6.	·0284	436·6	350.	15·1 19·07 $\times 10^{-6}$.	·5023 $\times 10^{-6}$
7.	·3563	385·5	238·9.	20·89 $\times 10^{-6}$.	·4981 $\times 10^{-6}$
8.	·0212	596·6	250.	21·5 $\times 10^{-6}$	·4955 $\times 10^{-6}$
9.	·0425	596·6	489	20·99 $\times 10^{-6}$	·4997 $\times 10^{-6}$
(10)	·0748	〃	865·5	22·03 $\times 10^{-6}$	·4943 $\times 10^{-6}$
(11)	·1040	〃	1108	20·53 $\times 10^{-6}$	·4977 $\times 10^{-6}$.
(12)	·0788	〃	790	21·02 $\times 10^{-6}$	·5001 $\times 10^{-6}$
(13)	·0611	〃	600	20·19 $\times 10^{-6}$	·4992 $\times 10^{-6}$
(14)	·0549	〃	531.	19·68 $\times 10^{-6}$	·4957 $\times 10^{-6}$
(15)	·0495	〃	490	20·00 $\times 10^{-6}$	·4927 $\times 10^{-6}$.

mass

5

Susceptibility of ~~the~~ Powder Carbon. (Spectroscopic Carbon).

1st Crystal. x– 20°

– ·52 → ·50

2nd Crystal – ·5375. ·54

– 14·65 → ·51

·002

·005

·0009 gm.

1) ·009 ~~·50~~

do Spectro

~~Cryst. no. 9~~ ~~$m = \cdot 0044$ gms.~~

~~Treated 12 - 13 hrs. washed 18 hrs~~

~~$\Delta\chi =$~~

No. 9.

treated ~~16~~ 12 hrs
washed 18 hrs.

$m = \cdot 0011$ gm.

first after washing $\cdot 0037$
30
26
20

within 15 mi

$\Delta\chi = 1\cdot 26$

which remained const. even after 9 hrs drying.

$1\cdot 3$

$1\cdot 27$
$1\cdot 24\text{-}6$
$1\cdot 39$

No. 7 $m = \cdot 00\cancel{2}4$

Treated 18 hrs.
washed 18 hrs.

$\Delta\chi = 1\cdot 21$

7 hrs
12
24

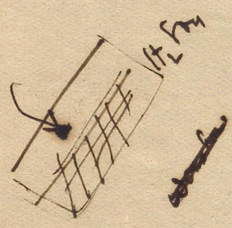
(1.3)

Graphite

3rd cryst m = ·0052 gm. before treatment

Treated with mix of con. H_2SO_4 + HNO_3.

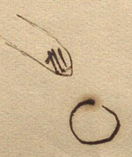
for 7 hrs, & washed in running water for 12 hrs.



$\Delta X = 1{\cdot}27 \times 10^{-6}$ per gm. of carbon

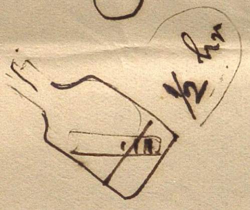
which remains ~~cons~~ the same after repeated drying — 29 hrs

~~4th~~ 4th m = ·0067. gm.



4th m = ·0060 gm.

treated for 19 hrs. washed 1 to 2 hrs.



ΔX = ·6 → effective of drying not tried

after washing wt = ·0246 gm.

5th m = ·0067 gm treated 24 hrs.

washed 12 hrs.

~~wt~~;

after simple drying $\Delta X = 1{\cdot}39$

Even after 8 hrs drying in dessicator ΔX same.

6th m = ·0025 ~~af~~ Treated 12 hrs.

washed > 18 hrs

after washing & drying with filter ·00245 gm.

1st – after washing ΔX = 1·24

& after 8 or 9 hrs drying : 1·24

1^{st} cryst. ·0170 gm

~~kept~~ treated for 4 hrs. — washed in running water

for 1 hr.

$\Delta X =$ ·7

after drying for 4 hrs $L =$ ·3

2^{nd} crystal split into 2 parts in the

acid mix. Hence rejected.

Graphite powder in paraffin

1) $\Delta\chi = 8.7 \times 10^{-6} = \chi_\perp - \chi_\parallel$

als $\chi_\perp = -3.0$ mean $= -5.9$.

.2

whereas mean value for single crystal

$= \frac{22}{3} + .5 = -7.8$.

2) Spectroscopic carbon

$\Delta\chi =$

$=$

(5). Influence of oxidation.

When a single crystal of graphite is kept for some time in a mixture of HNO_3 and H_2SO_4, a remarkable change in the size of the crystal is brought about. The crystal swells up, loses its brilliancy and the colour changes from black to green and finally to blue. It is therefore expected that there will be some change in the crystal structure and in the magnetic properties of the crystal on oxidation. To investigate the matter, ~~a few~~ some small crystals of graphite were selected and their x-ray photographs taken. The susceptibilities in both the directions were then measured. The crystals were then kept in separate dishes containing a mixture of conc. HNO_3 ~~and~~ H_2SO_4 (Proportion 2 parts H_2SO_4 + 1 Part ~~of~~ to HNO_3). The period of oxidation varies from crystal to crystal, ranging from 4 hours for the first to 24 hrs for the last crystals. In addition to these, 3 crystals were kept in a mixture of HNO_3 + H_2SO_4 containing $KClO_3$ in requisite proportion. After taking out a crystal from the mixture it was thoroughly washed in running water for about 12 hours and then dried in a dessicator. Just before placing it in the dessicator it was weighed and studied magnetically. The results obtained with 10 crystals are given in Table II.

Two of the crystals left for oxidations were washed thoroughly as before, and their x-ray photographs were taken. Fig. II & III. From the photographs they appear to be single crystals.

~~From~~ It appears from Table II that the difference in susceptibility in the two directions comes down from 22×10^{-6} to 1.3×10^{-6} whereas the specific susceptibility

IV.

for directions in the basal plain remains constant at $\cdot 5 \times 10^{-6}$.

(6). Susceptibilities of Graphite by Goetz's method.

In a series of notes in Phy Rev. Goetz and others published a some results obtained with graphite powder in a quite different way. Finely powdered and carefully purified small crystals of graphite are dispersed in a solution of gum Damar in Benzene. The solution is then placed in a strong magnetic field, ~~so as to have this~~; due to the high anisotropy of the crystals, they will align themselves under the influence of the field, so as to have their basal planes parallel to the field, the hexagonal axes of the crystallites being distributed ~~in~~ uniformly in the plane normal to the field. If under this condition the benzene is evaporated, a solid cake will remain, having the particles embedded in it and orientated as above. From measurements on this cake they obtained a value 13·2 for the ratio of the two principal susceptibilities of graphite. Later using agar + gelatin instead of gum Damar, they got 18 and 28 respectively.

In view of these uncertain values for the anisotropy of graphite, a series of experiments were performed. ~~Taking~~ In this case pure Paraffin was first ~~melted~~ mixed with pure graphite powder and then the Paraffin ~~in~~ in the mixture was melted, ~~and the molten liquid is shaken thoroughly~~ and then tranferred to a narrow thin walled test tube suspended in a very strong homogeneous magnetic field from a torsion head. The tube was then rotated continuously until it solidified to a hard mass. A block cut out from the solid Paraffin-graphite cylinder was studied. The Paraffin used in the process was then melted + experiment repeated with Paraffin alone. From the values obtained in the two cases, the difference in ~~anisotropy~~ susceptibilities due to graphite alone was found to be 6.7×10^{-6}. The results obtained by substituting fine spectroscopic carbon powder in place of graphite is

$$\delta\chi - \cdot 51 \times 10^{-6}$$

~~above~~ $$\chi_1 - \cdot 5 \times 10^{-6}.$$

To investigate further as to the dependence of susceptibility on particle size a new method was developed. Very fine powder of Purified graphite were dispersed in a Concentrated Solution of Potassium Iodide taken in a narrow tube with a bulb at the top. The particles were kept in suspension due to the high density of the Solution. The diameter of the tube used was about 1–2 mm. The tube was then placed in a very strong inhomogeneous magnetic field produced by bringing the Conical pole pieces of a magnet as near as possible. The movements of the individual particles ~~a~~ ~~[illegible]~~ in the Solution was observed with a high-Power microscope. It was observed that on putting on the Current the individual particles first orientated themselves in a particular direction, ie along the direction of the field and were then moved away from the strongest part of the field. The experiment was repeated with fine ~~and~~ finer particles using different balancing Solutions. It was observed that even with particles of spectroscopic carbon (diameter near about 10μ) $\chi_{\perp}$ remains Constant ~~at~~ ie $\underline{\cdot 5 \times 10^{-6}}$.

Susceptibility of Graphitic Acid.

Graphitic Acid Powder supplied by was first freed completely from oxides of Iron present as impurities, and the mean susceptibility of the pure powder was measured by the modified ~~improved~~ Gouy method.

Mean Susceptibility = $\cdot 54 \times 10^{-6}$.

Table I.

No. of Crystal	Mass in gm.	$(\chi_{\parallel} - \chi_{\perp}) \times 10^6$ per gm.	$\chi_{\perp} \times 10^6$ per gm.	
1.	·0223	−21·2	~~− ·494~~	− ·49~~4~~
2.	·0177	−21·3	~~− ·493~~	− ·49~~3~~
3.	·0315	−21·4	~~− ·506~~	− ·49~~4~~
4.	·0517	−21·1	~~− ·496~~	− ·5~~06~~
5.	·0190	−21·3	~~− ·502~~	− ~~·496~~ ·50
6.	·0284	−19·1	~~− ·498~~	− ·50~~2~~
7.	·3563	−20·9	~~− ·496~~	− ~~·498~~ ·50
8.	·0212	−21·5	~~− ·50~~	− ~~·496~~ ·50
9.	·0425	−21·0	~~− ·49~~	− ·50
10.	·0748	−22·0		− ·49~~4~~
11.	·1040	−20·5		− ~~·498~~ ·50
12.	·0788	−21·0		− ·50
13.	·0611	−20·2		− ~~·499~~ ·50
14.	·0549	−19·7		− ~~·496~~ ·50
15.	·0495	−20·0		− ·49~~3~~