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THE EFFECT OF SURFACE TENSION
ON THE ADHESION OF
LAMINAR SOLIDS

by

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THE EFFECT OF PARTICLE SHAPE ON THE
ABRASIVENESS OF LAMELLAR SOLIDS

by

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SUMMARY

The abrasiveness of samples of graphite of controlled shape has been shown to be related to the aspect ratio of the crystallites. A decreasing aspect ratio results in increasing abrasiveness. Whilst the same kind of relationship is valid for MoS_2 , its more complex surface chemistry does not yet permit the direct calculation of crystal aspect ratios. The effect of particle shape on the abrasiveness of graphite and MoS_2 is interpreted mainly in terms of the probability of crystals presenting edge faces to the surfaces in the sliding environment.

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1 INTRODUCTION.

It is well known that graphite can cause considerable wear of even hard metals during machining operations or in certain conditions of sliding. The abrasive nature of graphite appears to be due to its mechanical anisotropy¹, itself a consequence of its lamellar chemical structure, and the hardness in the basal plane direction, in which the carbon atoms are close-packed, has been estimated at 8.5 Mohs¹ or slightly greater than 1500 V.P.N.². Lancaster et al. have shown how the maximum hardness of lamellar solids may be estimated² and how their abrasiveness is related to particle size, impurity content and degree of crystalline perfection³. Under the conditions of testing chosen, increasing particle size and impurity content resulted in increasing abrasiveness. The influence of the degree of order in the crystals was examined by measuring the abrasiveness of samples of graphite having a range of 'p' values, i.e. a range of probabilities of layer displacement. It was found that increasing 'p' values resulted in both increased bulk hardness of the graphite and increased abrasiveness.

With the development of methods both for producing crystals of lamellar solids of controlled shape^{4,5}, and for estimating their shape from adsorption microcalorimetry, it has become possible to assess the effect of particle shape on the abrasiveness of such materials, and this was the object of the present work.

2 EXPERIMENTAL

2.1 Preparation of materials

Samples of graphite and of MoS_2 were ground in a vibratory ball mill to produce crystallites having either a higher or lower ratio of basal plane: edge face areas than the starting material. The method, which has been described in detail elsewhere^{4,5}, relies on the presence of a fluid medium to modify the cleavage of the crystal during the grinding process. Use of air as the medium results in the production of particles with aspect ratios less than approximately 10, whilst the use of a hydrocarbon medium produces thin, plate-like particles with aspect ratios of between 10 and 100. In the present work, the grinding was carried out in liquid n-heptane, and the wear debris was removed magnetically. In the case of graphite, the chosen starting material was a synthetic, nuclear grade, whilst for MoS_2 , a starting material conforming to specification DEF 2304 was chosen. Grinding was carried out as outlined above to produce particles of the desired shape: their shape and abrasive properties were then estimated as described in the following sections.

2.2 Measurement of particle shape

An estimate of the 'mean' particle shape of the crystallites of a lamellar solid may in principle be obtained by examination of a number of photographs or electron micrographs of these crystallites. Such a method would be inordinately tedious. Another method, developed by Groszek, depends on the preference which compounds with long methylene chains exhibit for adsorption on the non-polar, basal plane faces of lamellar solids such as graphite and MoS_2 ⁶. Adsorption of a non-polar, long-chain organic compound, such as a higher paraffin, occurs such that the molecule forms the maximum number of contacts with the surface of the adsorbent. This it achieves when it is adsorbed in the prone position. In contrast, polar organic molecules, such as alcohols, prefer to undergo adsorption on the high energy, polar edge faces of the crystal. Hence the ratio of the basal plane: edge face area of a lamellar solid may be estimated by measuring, in a flow microcalorimeter, the amount of heat evolved when adsorption of n-dotriacontane ($n\text{-C}_{32}\text{H}_{66}$) and n-butanol ($n\text{-C}_4\text{H}_9\text{OH}$) takes place on their respective sites. The amount of heat evolved in each case is a measure of the surface area of that particular type of site and hence the ratio of the heats evolved in non-polar and polar adsorption is related to the ratio of the basal plane:edge face area. This ratio may be calculated if the integral heats of adsorption of the compounds on the particular face of the lamellar solid are known, and hence a mean aspect ratio for the particles may be calculated on the assumption of a particular model crystal shape.

The flow microcalorimeter⁷ used in this work, and its application in measuring the heats of adsorption of long chain compounds on graphite⁸ and of polar organic molecules on oxides and metals⁹ has been described in detail previously.

2.3 Measurement of abrasiveness

The abrasiveness of the lamellar solid was estimated by measuring the rate of wear of a $\frac{1}{4}$ inch diameter phosphor-bronze ball sliding against a PTFE (or polypropylene) disc, in the presence of a dispersion of the lamellar solid. Fig.1 is a photograph of the apparatus, which has been described in detail previously¹⁰. The ball, mounted in a chuck rotating at 240 rev/min was slid, under a load of 1 kg, against a disc rotating at 28 rev/min, whose axis of rotation was offset from that of the chuck. Hence the sliding path of the ball was spread out over the disc into the form of an annular ring, and the wear of the ball was concentrated at its tip. The volume of material removed

from the ball during a known distance of sliding was estimated by superimposing profiles of the ball taken before and after sliding, followed by integration of elemental areas around the axis of the wear scar. Typical results are shown in Fig.2.

The distance of sliding, in given time T , was computed from the relationship

$$D = C[2\pi nd T] , \quad \left(\begin{array}{l} n = \text{speed of rotation of ball} \\ d = \text{half-width of annular ring} \end{array} \right)$$

where the term in brackets is the distance travelled assuming that the disc is stationary and C is a correction factor calculated to account for the relative motions of the ball and disc. Under the conditions used in this work, $C = 0.89$.

The lower limit of detection of wear in this method of measuring abrasion was defined by the wear occurring when no dispersion was present, i.e. when the wear was solely due to the disc-ball contacts, and corresponded to a wear rate for the ball of $\sim 1 \times 10^{-12}$ cm³/cm kg for PTFE discs and to $\sim 3 \times 10^{-12}$ cm³/cm kg for polypropylene discs.

In the case of MoS₂, dispersions of 20% by weight of powder in a silicone fluid of viscosity 20 cs (MS 200) were found to be suitable, whilst for graphite, 10% dispersions were preferable because of the greater thickening activity of the graphite. The high speed of rotation of the ball and chuck served to stir the dispersion efficiently during sliding, whilst the slow speed of the disc assembly avoided separation of the solid particles by centrifugal effects.

3 RESULTS AND DISCUSSION

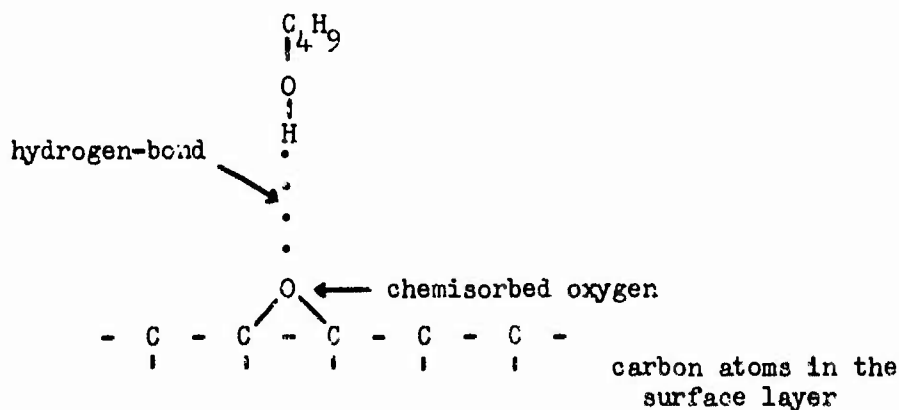
3.1 Graphite

Table 1 lists the samples used and their mode of preparation, their total surface areas, calculated from a B.E.T. analysis of nitrogen adsorption isotherms, the amount of heat evolved when 1 gm of each sample adsorbs n-dotriacontane and n-butanol from n-heptane solution, the ratio of these heats, and the rate of abrasion of a phosphor-bronze ball sliding against PTFE in the presence of a dispersion of these samples. Fig.3 is a plot of the rate of abrasion of the ball as a function of the ratio of the heat evolved on adsorption of the non-polar material to that evolved on adsorption of the polar material, $\frac{\Delta H_{n.p.}}{\Delta H_p}$. It is clear that the rate of abrasion increases with

decreasing $\frac{\Delta H_{n.p.}}{\Delta H_p}$. The latter ratio is a measure of the ratio of non polar: polar surface area in the crystal and hence of the ratio basal plane: edge face area. Thus the rate of abrasion increases as the ratio of basal plane: edge face area decreases, i.e. as the crystal becomes less plate-like and as the aspect ratio decreases.

Recently relationships have been established between the heat of adsorption of n-dotriacontane and the basal surface area of graphites, and between the heat of adsorption of n-butanol and the edge face area of graphites¹¹. Evolution of 1 cal/gm on adsorption of n-dotriacontane corresponds to 89 m² of basal plane area, whilst evolution of 1 cal/gm on adsorption of n-butanol corresponds to an edge-face area of 28 m². Hence from the data in columns three and four of Table 1, it is possible to calculate the basal plane area and edge-face area of each sample of graphite, and therefore to determine a crystallite aspect ratio on the basis of a chosen model crystallite shape. For disc-like particles, or square plates, the aspect ratio is twice the ratio of basal plane-edge face area, whilst for regular hexagonal plates the factor becomes $4/\sqrt{3}$. Hence no significant difference results from varying the choice of particle shape. Table 2 lists the results of calculating the polar and non-polar areas, and hence the aspect ratios, for the graphites in Table 1 by the method outlined above, and the rate of abrasion is plotted as a function of the calculated aspect ratio in Fig.5.

An alternative method of calculating crystal aspect ratios relies on the assumption that the forces binding n-butanol molecules to the edge sites of graphite are due to hydrogen bonding. This is not unreasonable, since these sites are likely to be covered with a layer of chemisorbed oxygen and water^{12,13}, leading to the possibility of hydrogen-bonding as shown below:-



Under this condition, and if the n-butanol forms a close-packed 'monolayer' on the available edge sites, then the integral heat of preferential adsorption of the butanol on graphite is the same as that for n-butanol adsorbed on metal oxides, where hydrogen-bonding controls adsorption¹⁴. The area available for butanol adsorption, that is the edge-face area, may then be derived from previously recorded data⁹, which relates the heat of preferential adsorption of n-butanol on a series of oxides to their known surface area. Having thus derived the polar area of the graphite, and knowing the total B.E.T. nitrogen area, the basal plane area may be obtained by difference and hence the ratio basal plane: edge face area may be calculated. Finally, a crystal aspect ratio may be determined on the assumption of a model crystal shape. The results of such a method are included in Fig.5 as a dotted line, and may be compared with the more direct method which utilises empirically established relationships. The two methods agree only at the higher values of crystal aspect ratio, i.e. when the proportion of edge sites is very small. The reasons for this are twofold: first, the hydrogen-bond model over-estimates the edge face areas by a factor of almost two, and second, the great affinity of n-dotriacontane for the basal planes of graphite allows it to adsorb on basal areas which are inaccessible to nitrogen¹¹. Both phenomena result in increasing divergence in calculated aspect ratio as the aspect ratio decreases. The ability of n-dotriacontane to adsorb on basal surface which is inaccessible to nitrogen leads to the fact that the total surface areas calculated as the sum of the basal and edge face areas in Table 2 are greater than the total surface areas as measured by nitrogen adsorption isotherms (Table 1).

It is clear from Fig.5 that the more plate-like the crystallites become, the less abrasive they are under these conditions of sliding. Two factors, the orientation of the crystallites at the sliding interface, and their degree of crystalline disorder, are probably responsible for this effect. Crystals having a high aspect ratio are very likely to lie flat at the sliding interface and hence present their basal planes to the sliding surfaces. They therefore shear easily in the direction of sliding and show little inclination to embed in the plastic or metal surface. Further, stresses normal to the sliding interface will result, as observed by Savage¹⁵, in deformation of the crystal, which is relatively weak in the 'C' direction, rather than in abrasion of the metal. In contrast, crystals having an aspect ratio not far from unity are almost as likely to present edge-faces as basal planes to the sliding surfaces, and thus are more likely to scratch the metal surface. Because they are

also more likely to become embedded in the plastic when the aspect ratio is low, the abrasion effect becomes more pronounced. Hence crystallites of low aspect ratio present the possibility of both three-body abrasion and effectively two-body abrasion.

The second factor, the degree of disorder in the crystals, affects the intrinsic abrasiveness of the material³. The grinding process introduces some turbostraticity (i.e. the degree of misorientation of basal planes by rotation about the 'C' axis) into the crystals, particularly those of low aspect ratio, and the rapid rise in abrasiveness as the aspect ratio falls may in part be due to the effect of turbostraticity itself on abrasiveness, though to what extent it is not possible to judge.

3.2 Molybdenum disulphide

Table 3 lists the abrasiveness, the amount of heat evolved on adsorption of non-polar and polar compounds on their respective sites, and the ratio of these heats on eleven samples of MoS_2 . The starting material, No.1, conforming to specification DEF 2304, was ground in a hydrocarbon and in air to produce respectively crystals of higher (Nos.4, 5 and 6) and lower (Nos.2 and 3) aspect ratios than the original material. Samples 7-11 inclusive are commercial materials from four different sources; 10 and 11 are reference samples provided for research purposes by the Climax Molybdenum Co., having the properties detailed in Table 4.

The influence of $\frac{\Delta H_{n.p.}}{\Delta H_p}$, and thus of particle shape, on the abrasiveness of MoS_2 is shown in Fig.6. The lines are drawn to establish the relationships, both on PTFE and polypropylene, for materials 1-6 inclusive, since these were prepared from common stock. It is clear that, as with graphite, increasing aspect ratio leads to particles of decreasing abrasiveness. The points numbered 7-11 inclusive refer to the commercial materials listed in Table 3. Their scatter from the full line is entirely towards a higher abrasiveness than their measured aspect ratio would suggest: this is probably a consequence of their varying particle size, degree of crystalline perfection and impurity content.

The use of polypropylene discs in place of PTFE causes the wear scars on the phosphor bronze balls to become flatter, since the disc does not deform so readily and the wear is concentrated at the tip of the ball. Further, the wear rates on polypropylene are higher than on PTFE discs. It seems likely that this is related to the ease with which particles become embedded in the plastic

and whether, on becoming embedded, they continue to support any part of the applied load.

It is difficult to differentiate between effects due to particle size and particle shape when considering crystals which are not defined by a single dimension, such as cubes or spheres. However, comparison of samples 10 and 11 shows that their abrasiveness differs by an order of magnitude, despite their similar impurity contents. The 'size' of the crystals, which is probably a measure of their basal dimension, controls how easily they become embedded in the plastic disc and thus how rapidly they are able to wear the phosphor-bronze ball.

Comparison of Figs. 3 and 6 shows that the values of $\frac{\Delta H_{n.p.}}{\Delta H_p}$ for MoS_2 are approximately one order of magnitude lower than the corresponding values for graphite. Therefore, either the ratio of basal plane: edge face areas are very different in the MoS_2 samples, i.e. they have aspect ratios an order of magnitude lower than the graphite samples, or the integral heat of adsorption of n-butanol is greater on MoS_2 than on graphite. The former possibility seems unlikely, since the maximum aspect ratio would then be approximately 10 and the minimum 0.3: the latter particles would be much more likely to present edge faces than basal planes to the sliding surfaces and hence their abrasiveness would be expected to be higher than is observed.

The postulate that the heat of adsorption of n-butanol is greater on MoS_2 than on graphite is much more tenable, in view of the more complex chemical nature of MoS_2 . Thermogravimetric analysis¹⁰ suggests that the surface of MoS_2 is covered by a layer of MoO_3 , which promotes adsorption of water¹², and that even at low temperatures chemisorption of water leads to the formation of MoOS_2 ¹⁷. The situation is further complicated by the fact that comminution of MoS_2 in air leads to the formation of water-soluble sulphate (SO_4^{2-}) and hexavalent molybdenum^{13,16}. It is therefore probable that the presence of ionic surface contaminants results in a considerable increase in the heat of adsorption of polar molecules, such as n-butanol, on the surface of MoS_2 . This is substantiated by experiment (e.g. the heats of adsorption on air-ground materials are 0.0048 cal/m^2 for graphite sample No. 4, and 0.145 cal/m^2 for MoS_2 sample No. 3), and by the fact that, if edge face areas are calculated on the basis of the original assumptions, then in the case of air-ground samples, the edge face areas appear to be greater than the total surface area as measured by nitrogen adsorption. In the absence of an

empirical relationship between the heats of adsorption of n-dotriacontane and n-butanol on the non-polar and polar sites of the molybdenum disulphide surface, it remains impossible to calculate mean aspect ratios for the various samples, but the trend shown in Fig. 6 remains valid.

4 CONCLUSIONS

The abrasiveness of samples of graphite has been shown to be related to their shape. A decreasing aspect ratio results in increasing abrasiveness of the crystallites.

Established relationships between the heat of adsorption of non-polar compounds and the basal surface area of graphites, and between the heat of adsorption of polar compounds and the edge face areas allow direct calculation of mean crystallite aspect ratios. Calculations based on the assumption of a specific surface chemistry of adsorption of polar compounds on edge face sites of graphite yield similar, but less reliable, aspect ratios.

A similar relationship exists between the particle shape and abrasiveness of MoS_2 , although the more complex surface chemistry of MoS_2 does not permit the calculation of aspect ratios.

The differences in abrasiveness shown by particles of varying shape has been interpreted mainly in terms of the probability of the crystal presenting edge faces to the sliding surfaces; differences in intrinsic abrasiveness, due to turbostraticity, are probably of secondary importance.

ACKNOWLEDGEMENTS

The authors would like to thank Mr. G.H. Kinner for his continued support of this work and Dr. J.K. Lancaster for helpful discussions.

Table 1
ADSORPTIVE AND ABRASIVE PROPERTIES OF GRAPHITE MATERIALS

Sample	Surface area m^2/g	Heat evolved on adsorption of $\text{n-C}_{32}\text{H}_{66}$ ΔH n.p. cal/g	Heat evolved on adsorption of $\text{n-C}_4\text{H}_9\text{OH}$ ΔH p. cal/g	$\frac{\Delta H \text{ n.p.}}{\Delta H \text{ p.}}$	Rate of abrasion of phosphor- bronze ball $\text{cm}^3/\text{cm kg}$
1. Starting material Synthetic graphite nuclear grade	5	0.117	0.059	1.98	3.2×10^{-11} 2.3×10^{-11} 2.9×10^{-11}
2. Air-ground graphite	410	2.90	5.54	0.52	5×10^{-10} 3.2×10^{-10} 3×10^{-9}
3. Repeat of 2					
4. Air-ground graphite	58	0.65	0.28	2.32	1.3×10^{-11} 1.6×10^{-11}
5. Hydrocarbon- ground graphite	120	1.61	0.20	8.05	5×10^{-12} 6.4×10^{-12}
6. Hydrocarbon- ground graphite	95	1.27	0.079	16.1	3×10^{-12}

Table 2
ASPECT RATIOS OF GRAPHITE SAMPLES, CALCULATED FOR
SIMPLE DISC-LIKE CRYSTALS

Sample	Basal plane area m ²	Edge face area m ²	Aspect ratio of crystal
1	10	1.65	12
2 & 3	258	155	3.3
4	58	7.8	15
5	143	5.6	51
6	113	2.2	103

Table 3
ADSORPTIVE AND ABRASIVE PROPERTIES OF SAMPLES OF MOLYBDENUM DISULPHIDE

Sample	Heat evolved on adsorption of $n-C_{32}H_{66}$ ΔH n.p. cal/g	Heat evolved on adsorption of $n-C_4H_9OH$ ΔH p. cal/g	$\frac{\Delta H \text{ n.p.}}{\Delta H \text{ p.}}$	Rate of abrasion of phosphor-bronze ball $\frac{cm^3}{cm^2 \cdot cm \cdot kg}$
1. Commercial material, specification DEF 2304	0.0028	0.0167	0.17	$\left. \begin{array}{l} 3 \times 10^{-11} \\ 2 \times 10^{-11} \\ 2 \times 10^{-11} \end{array} \right\} \text{PTFE disc}$ 1.1×10^{-10} , polypropylene disc
2. As No.1, air-ground	0.029	0.86	0.034	$\left. \begin{array}{l} 7.5 \times 10^{-11} \\ 1.2 \times 10^{-10} \end{array} \right\} \text{PTFE disc}$ 3.7×10^{-10} , polypropylene disc
3. As No.1, air-ground	0.0566	1.89	0.03	1.3×10^{-10} , polypropylene disc
4. As No.1, hydrocarbon-ground	0.197	0.58	0.34	1.8×10^{-12} , polypropylene disc 1.1×10^{-12} , PTFE disc
5. As No.1, hydrocarbon-ground	0.047	0.143	0.33	3×10^{-12} , PTFE disc
6. As No.1, hydrocarbon-ground	0.132	0.170	0.78	4.5×10^{-12} , "
7. Commercial, micronated grade	0.016	0.008	2.0	3×10^{-12} , "
8. Commercial, micronated grade	0.036	0.126	0.285	5×10^{-12} , "
9. Commercial	0.0048	0.0118	0.41	6×10^{-11} , "
10. Commercial, micronated	0.036	0.114	0.31	3×10^{-11} , "
11. Commercial, as No.10, unmicronated	0.010	0.054	0.18	5×10^{-10} , "

Table 4
THE CHEMICAL COMPOSITION OF MoS₂
SAMPLES NUMBERS 10 AND 11

	Sample No.10	Sample No.11
Mo	58.55	58.60
S	39.15	39.44
MoO ₃	0.18	0.03
Fe	0.14	0.12
H ₂ O	0.035	0.0
Oil	0.17	0.047
C	1.34	1.22
Total insolubles	0.58	0.51
Acid No.	0.11	0.014
Particle size	0.7 μm	7.0 μm
$\Delta\text{H n.p.}/\Delta\text{H p.}$	0.31	0.18

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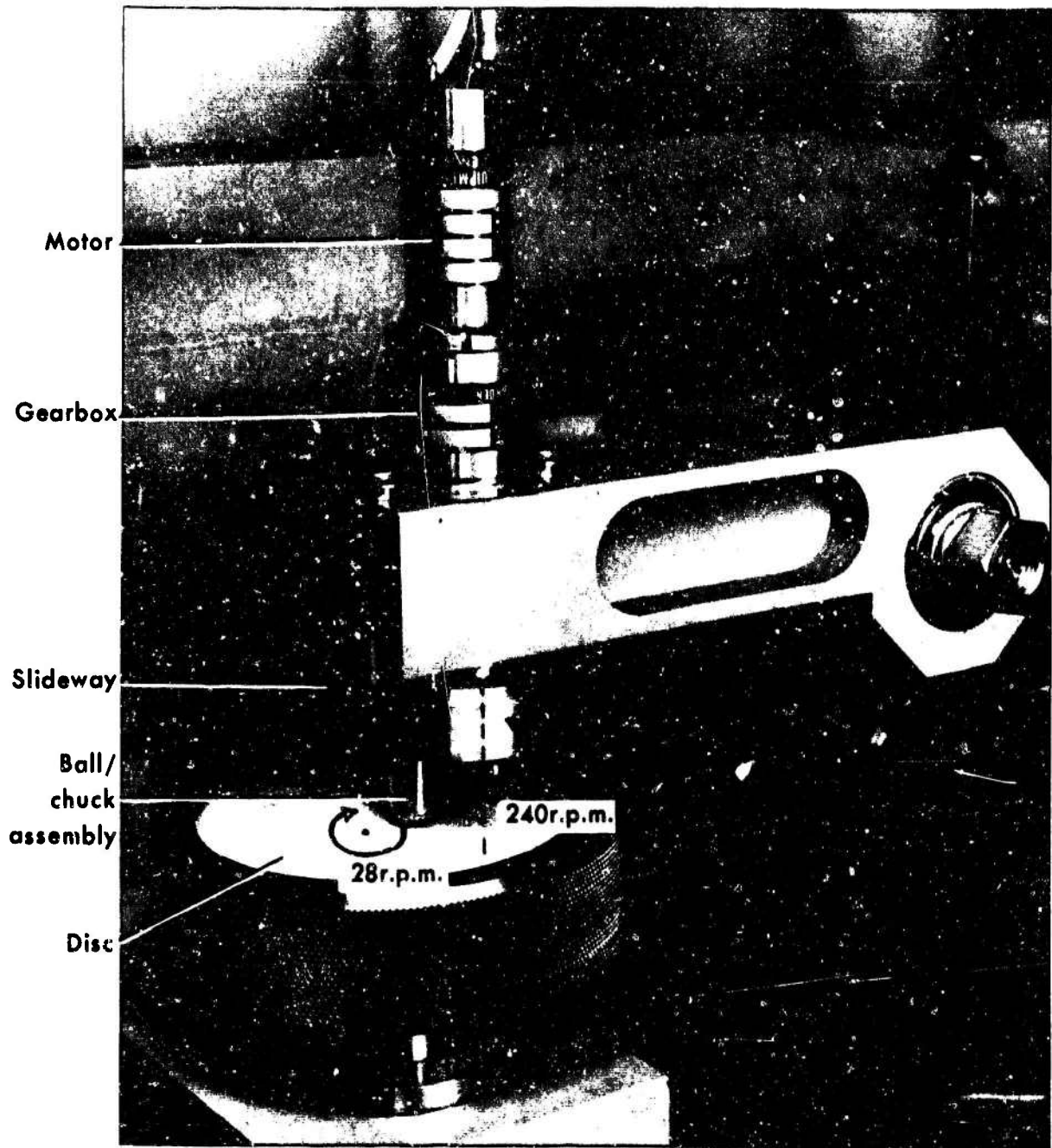
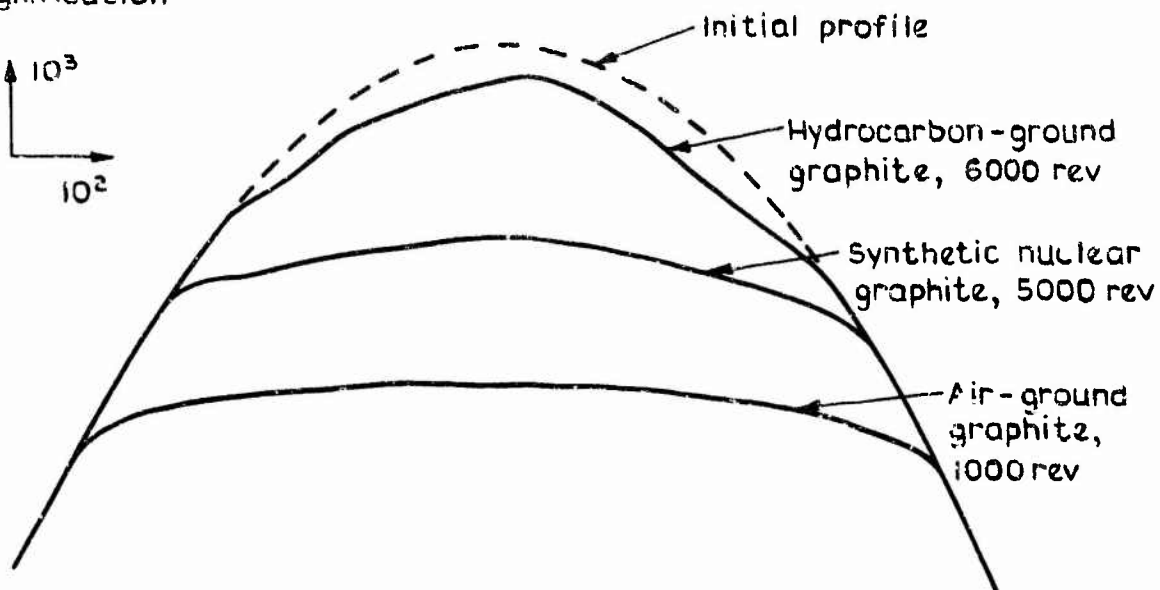
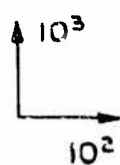


Fig.1. The abrasion apparatus

Fig.2 a&b

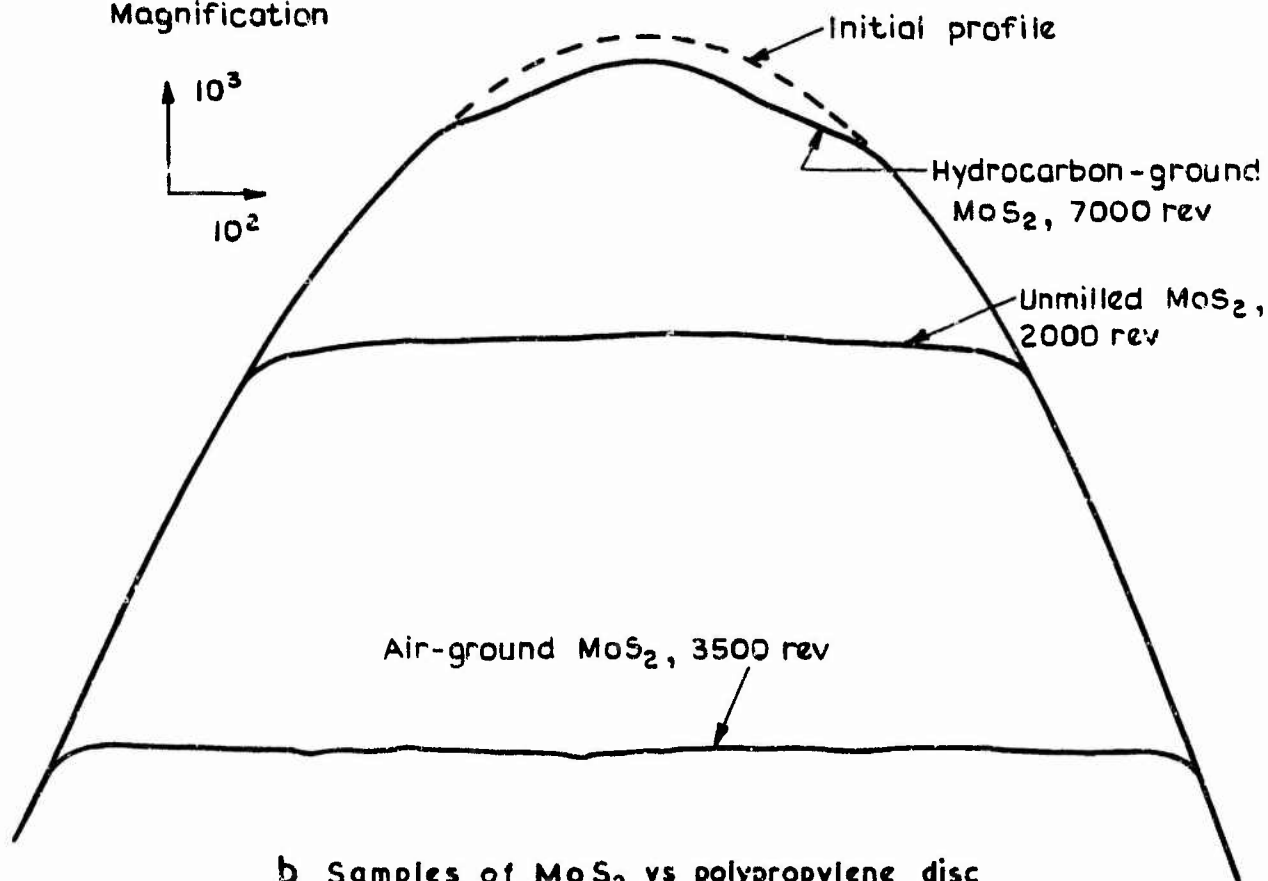
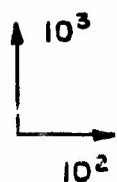
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Magnification



a Graphites vs PTFE disc

Magnification



b Samples of MoS₂ vs polypropylene disc

Fig.2a&b Typical profiles of ball-tips after abrasion in dispersions of lamellar solids

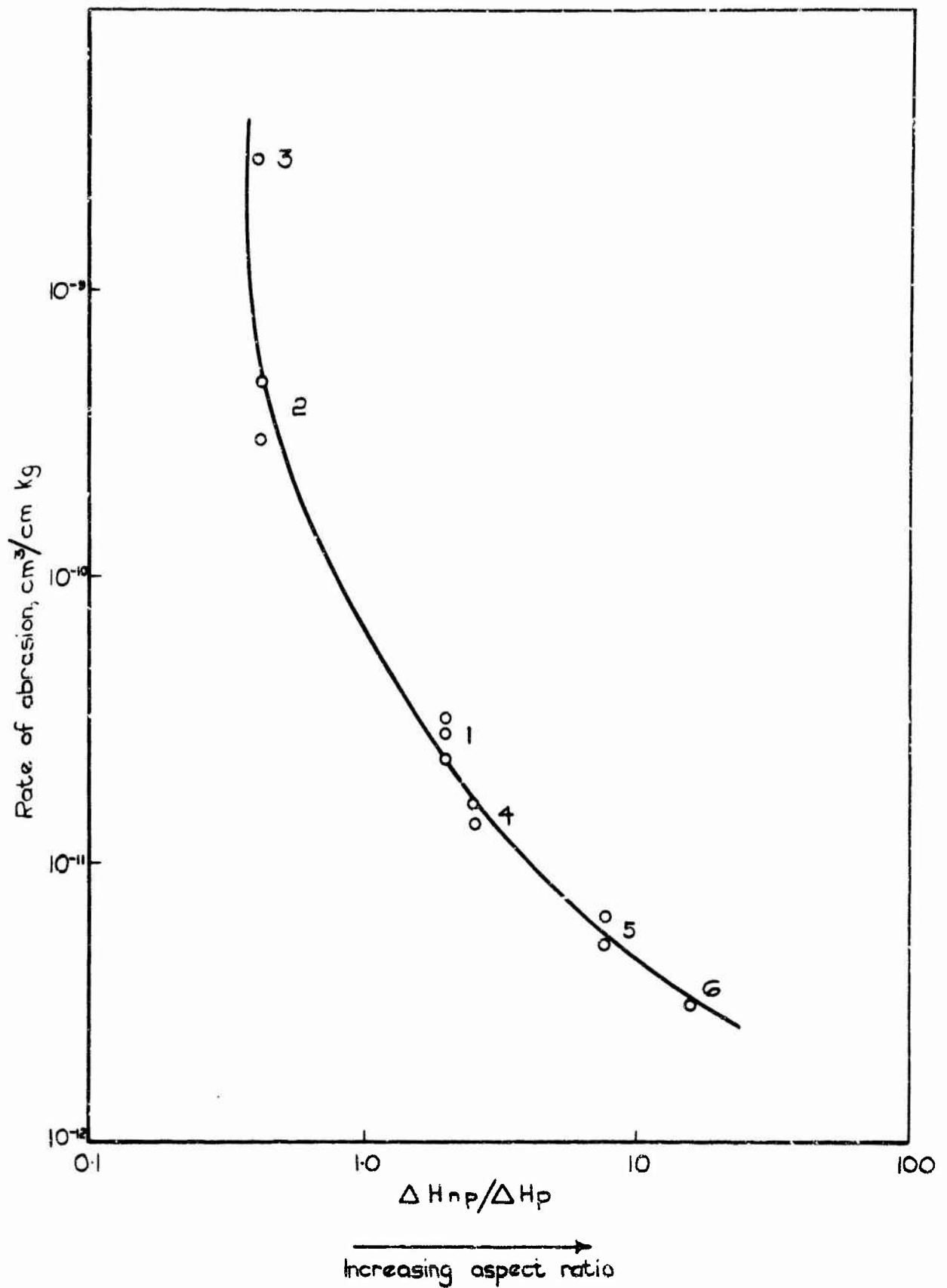


Fig.3 The influence of particle shape on the abrasiveness of graphite. Numbers refer to the samples in Table I

Fig.4a

T.R.69024



Fig.4a. Electron micrograph of air-ground graphite, x20,000



Fig.4b. Electron micrograph of hydrocarbon-ground graphite, x20,000

Fig.5

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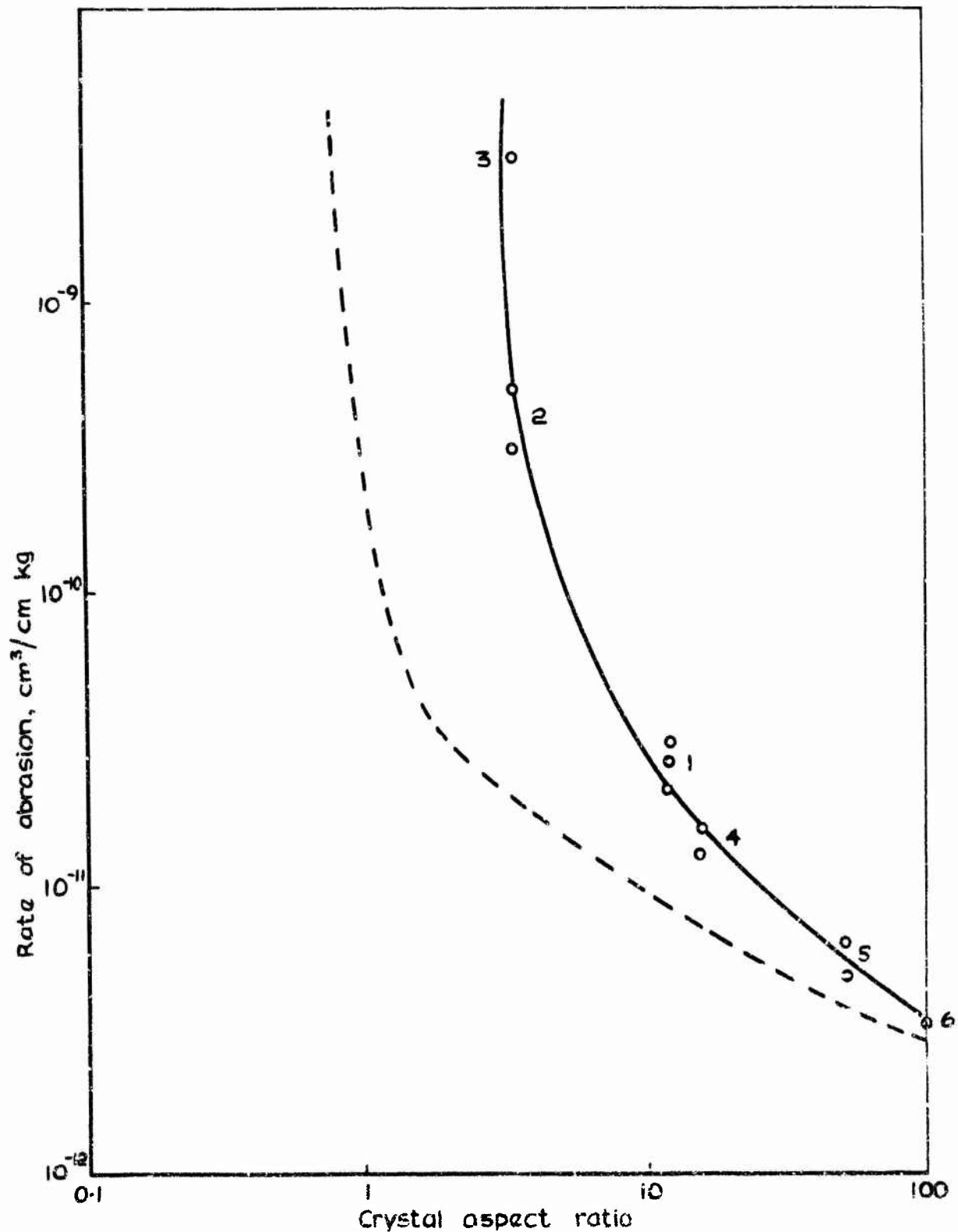


Fig.5 The influence of aspect ratio on the abrasiveness of graphite crystallites. Numbers refer to the samples in Table I. Full line—aspect ratio calculated from established relationships between heats of adsorption and surface areas. Dotted line—aspect ratios calculated on hydrogen-bond mode!

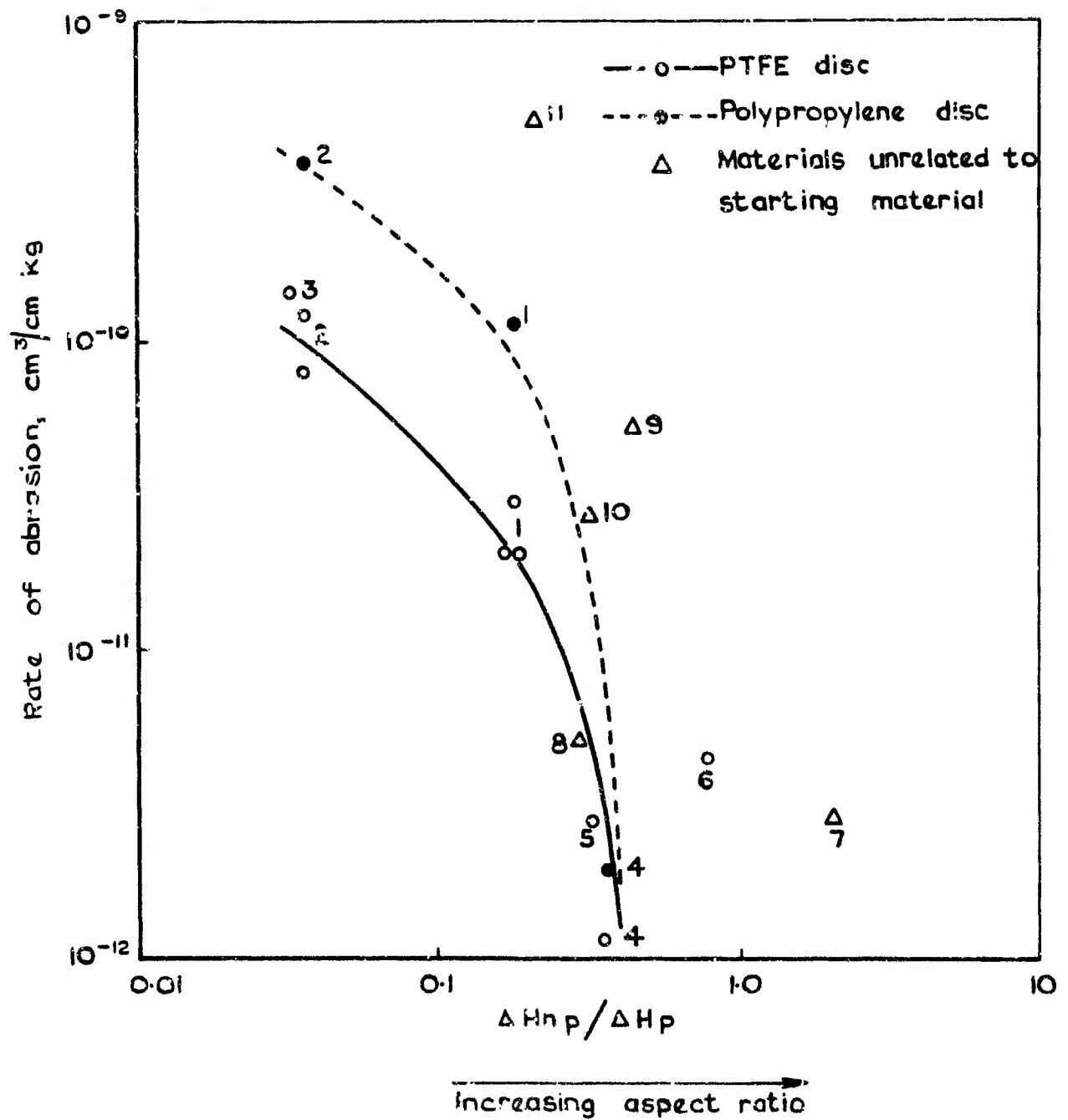


Fig. 6 The influence of particle shape on the abrasiveness of MoS_2 . Numbers refer to samples in Table III

Giltrow, J.P. Groszek, A.J. 539.216 : 539.576 : 539.215.4 THE EFFECT OF PARTICLE SHAPE ON THE ABRASIVENESS OF LAMELLAR SOLIDS Royal Aircraft Establishment Technical Report 69024 February 1969 The abrasiveness of samples of graphite of controlled shape has been shown to be related to the aspect ratio of the crystallites. A decreasing aspect ratio results in increasing abrasiveness. Whilst the same kind of relationship is valid for MoS_2, its more complex surface chemistry does not yet permit the direct calculation of crystal aspect ratios. The effect of particle shape on the abrasiveness of graphite and MoS_2 is interpreted mainly in terms of the probability of crystals presenting edge faces to the surfaces in the sliding environment.	Giltrow, J.P. Groszek, A.J. 539.216 : 539.576 : 539.215.4 THE EFFECT OF PARTICLE SHAPE ON THE ABRASIVENESS OF LAMELLAR SOLIDS Royal Aircraft Establishment Technical Report 69024 February 1969 The abrasiveness of samples of graphite of controlled shape has been shown to be related to the aspect ratio of the crystallites. A decreasing aspect ratio results in increasing abrasiveness. Whilst the same kind of relationship is valid for MoS_2, its more complex surface chemistry does not yet permit the direct calculation of crystal aspect ratios. The effect of particle shape on the abrasiveness of graphite and MoS_2 is interpreted mainly in terms of the probability of crystals presenting edge faces to the surfaces in the sliding environment.
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