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GRAPHITE FUEL STUDIES

PART 2 – FINE GRINDING OF ARTIFICIAL GRAPHITE

by

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Sydney, March, 1959.

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Abstract

Homogeneous dispersions of fissile and fertile material in graphite are being currently considered as fuel for high–temperature, gas–cooled nuclear reactors. A possible method of fuel preparation involves compaction of finely ground artificial graphite with U and Th metal powders. Size reduction of artificial graphite is therefore of interest in the preparation of graphite–fuel compacts.

Reactor graphite has been successively size reduced in a jaw crusher, pin disc mill and ball mill to give a material 50% finer than 9 microns, and with a B.E.T. nitrogen adsorption surface area ranging up to 18.4 m^2/g. The variables in ball milling have been studied, and in particular it was found that Ni–hard balls gave a faster grinding rate, but a higher contamination than was obtained with steel balls. Leaching with boiling HCl was effective in removing contamination introduced by these grinding media.

The Rosin–Rammler function, $R = 100\,e^{-\left(\frac{x}{\bar{x}}\right)^n}$ applied to the size distributions of the ground graphite. In the size range 70 – 1000 microns the distribution constant n equalled 1, which is consistent with published data for other materials. However, above and below this size range, the distribution constant approximated 2. This high value of n for the sub–sieve range has not been previously reported, but is not peculiar to artificial graphite as other materials were found to have a similar value of n.

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1. INTRODUCTION

Nuclear reactor fuel elements consisting of a uniform dispersion of fissile and fertile materials in a moderator have a number of advantages compared with pure fissile–fertile fuel. The fission fragment damage to the fuel element should be reduced by appropriate choice of fissile particle size, and also higher heat ratings are possible because of the extended surface area of the fuel. The physical properties of graphite make this an attractive matrix material for high–temperature, gas–cooled reactor fuel elements (1, 2). Graphite containing uranium and thorium may be produced by solution impregnation techniques, and by compaction or extrusion of carbonaceous filler and binder materials with U and Th metals, oxides or carbides (3). Another method of fabrication involves compaction of finely ground, artificial graphite with the metal powders, followed by heat treatment to convert the U and Th to their carbides (4).

Frechette et al (5) have studied the compaction of natural and artificial graphite. With minus 200 mesh material it was found that bonding of artificial graphite was only achieved after wet oxidation with fuming HNO_3–$KClO_3$ mixtures. However, Livey and co–workers (4) have shown that artificial graphite of sufficiently fine particle size may be cold pressed into compacts of density 1.95 – 2.00 g/cc without any oxidation treatment or the use of binders. Before commencing work on the compaction of graphite with U and Th metal powders it was necessary to study the fine grinding of artificial graphite. The results of this investigation are reported in the present paper.

Graphite is used extensively in the paint and lubricant industries, but very little information is available on the preparation of fine graphite powder. Perry (6), Berry (7) and Burford (8) discuss equipment and performance data for the grinding of natural and artificial graphite. High–speed hammer mills, colloid mills, ball and tube mills, and fluid energy mills have been used with some success on this grinding problem. Berry (7) states that Ceylon graphite has been reduced from 3 mesh to an average size of 2 microns in a Micronizer fluid energy mill. No indication was given how this average size was determined. From the capacity and power data given by the above authors it appears that graphite is more difficult to grind than such materials as bituminous coal and barytes.

Schofield (9) has studied the ball milling of various ceramic materials. Graphite was ground in a carbon mill with carbon balls from 20% minus 200 mesh to 80% minus 325 mesh in 24 hours. The inefficient grinding obtained with carbon balls was offset by the elimination of contamination. This worker found by grinding with steel balls, followed by leaching and washing to remove the iron contaminant, loss of ultrafines generally occurred. In most cases this is undesirable, since these particles constitute a very large part of the surface area of the ground material.

2. EXPERIMENTAL

2.1 Equipment

A 6″ x 3″ open door, roll jaw crusher (Blake Type), manufactured by Sturtevant Engineering Co. Ltd., U.K. was used for the primary crushing. The graphite was then ground in a high–speed, pin disc mill (4″ dia. discs), made by Kek Ltd., U.K. The product from this mill was collected in a bag filter. A Pascall Engineering Co., U.K., three–tier laboratory ball mill with hard rubber pots of nominal capacity 1.0 imperial gallon, and internal dimensions of 6″ dia. x 10–½″ long was used for the fine grinding studies.

British Standard sieves in a Rotap tester were used to determine size distributions of the coarse graphite, and a Sharples Micromerograph was used in the sub–sieve range.

2.2 Materials

The raw material was offcuts from standard 8″ x 8″ x 30″ blocks of grade A reactor graphite of U.K. origin. The offcuts were drilled with a tungsten carbide tipped drill to give a sample for analysis of the original graphite, and then cut with a band saw into 1″ x 1″ x 4″ blocks for feeding to the primary crusher.

The grinding media used were porcelain balls, steel balls with about 5% carbide, and Ni–Hard balls having the approximate composition: 3.3 – 3.6% total carbon, 0.5% Si, 1.7% Ni, and 1.8% Cr. The Rockwell C hardness of the steel balls was 45, and that of the Ni–Hard balls was 58.

The total weight and sizes of the balls used in a 1 gall. mill are given in Table I.

TABLE I

BALL CHARGES IN ONE GALLON MILL

Dia. of Balls inches	Porcelain	Steel	Ni–Hard
1	3.0 lb	9.75 lb	9.75 lb
¾	2.5	7.5	7.5
5/8	–	–	3.75
½	1.5	3.75	–
Total Weight:	7.0 lb	21.0 lb	21.0 lb

2.3 Size Reduction

The 1" x 1" x 4" graphite blocks were crushed by two passes through the jaw crusher with closed jaw settings of 3/4" and 1/16" respectively. After the second pass the product was nominally 50% minus 14 mesh. Two passes of this material through the pin–disc mill gave a material nominally all finer than 72 mesh, and 75% finer than 300 mesh.

Using this material as feed for the ball mill an investigation of the effect of various operating conditions was made on the rate of grinding, size distribution of product and the degree of contamination. These variables were time of grinding, type and size of grinding media, mill speed, graphite charge in the mill, and the effect of grinding aids.

A preliminary study has also been made of the grinding of graphite in a Micronizer fluid energy mill.

2.4 Sampling

Riffling of ground graphite caused loss of fines, and was not used as the sampling procedure. The graphite was carefully spread on a large tray and 30–40 random sections were removed with a spatula. Two samples were taken, one of 50 g. for sieve analysis and the other of 20 g. for contamination analysis. Approximately 0.5 g. was removed from the latter sample for the Micromerograph analysis.

2.5 Contamination Removal

The efficacy of acid leaching was studied for the removal of contamination introduced by milling with steel and Ni–hard balls. The graphite was refluxed for different times with hydrochloric acid of various concentrations. Several leaching tests were done with nitric acid. The leached graphite was filtered on a Buchner filter, and washed free of acid in the filter. The loss of fines during the treatment was practically eliminated.

The washed graphite was dried at 120°C in an air oven, then ball milled for 10–15 minutes to break down agglomerates before being sampled.

3. RESULTS

3.1 General

The 1" x 1" x 4" blocks of graphite were readily crushed in the jaw crusher, despite the lubricating effect of graphite. The jaw crusher gave reproducible results, provided the closed set of the jaws was carefully adjusted before each run. The pin disc mill operated satisfactorily providing the feed rate was not too fast. After ball milling the graphite was nominally 50% finer than 9 microns as measured by the Micromerograph.

The size distribution of ground materials may in many cases be correlated by the Rosin–Rammler function (10), (11) given in Equation 1

$$R = 100e^{-\left(\frac{x}{\bar{x}}\right)^n} \qquad (1)$$

where R = percentage weight retained on size x
x = size in microns at which R is retained
$\bar{x}$ = absolute size constant in microns
n = distribution constant

The Rosin–Rammler function has been used to plot the size distributions of the graphite produced by the above grinding procedure. The co–ordinates of the graph paper used are due to Bennett (11); $\bar{x}$ is given by the size at which 63.21% passes size x, and n is the slope of the line. These two constants completely describe any one distribution.

3.2 Size Reduction

The size distributions obtained from the preliminary crushing in the jaw crusher and pin disc mill are shown in Figure 1. In the first pass through the jaw crusher only about 20% passed a 5 mesh screen. After the second pass the distribution has an inflexion at about 800 microns, and the slope changes from 1.7 to 0.9. The screen analyses of the pin disc mill products also have a slope of approximately 0.9. However, the Micromerograph analyses of the pin disc mill products in the sub–sieve range give slopes of the order of 1.8. It was necessary to screen out the +85 mesh material from the pin disc mill products before analysing them in the Micromerograph and this has been allowed for in calculating the resulting distribution. Figure 1 shows that the upper part of the Micromerograph distributions tend towards a slope of 1.

Figures 2 and 3 show the effect on the distribution of time of grinding. With the Ni–hard balls grinding ceases after 40 hours and further grinding does not change the distribution. With the steel balls the same effect was found after 100 hours. Size distributions of the graphite ground with the three different types of grinding media are shown in Figure 4. From Figures 2, 3 and 4 it is seen that the Ni–hard balls give the most rapid reduction, but the final size distribution reached is much the same with each type of grinding media.

The critical speed of a ball mill is given in Equation 2

$$N = \frac{54.19}{\sqrt{R}} \qquad (2)$$

where N = critical speed, R.P.M.
R = inside radius of mill, ft.

An increase in mill speed from the usual speed of 60% critical to 80% critical gives a more rapid reduction as shown in Figure 5 for grinding with Ni–hard balls. Grinding is more rapid the smaller the charge of graphite in the mill as seen in Figures 6 and 7. A 500 g. charge of graphite in a 1 gallon mill just filled the space between the balls and following Coghill and Devaney's recommendation (12), this was used as an optimum charge, being a compromise between fast grinding and high contamination, and slow grinding with practically no contamination. A mill speed of 60% critical was chosen as optimum on similar considerations, and was used in all runs except the run with 80% critical speed as given in Figure 5.

The size distributions of the ball milled graphite, determined with the Micromerograph, when plotted as Rosin–Rammler functions in Figures 2 – 7 inclusive, have a range of slopes from 1.8 – 2.8, with a mean value of 2.2 for the fifteen different runs.

Attempts to lower the limit of grinding by using ¼" steel balls with a 500 g. charge, and grinding for 140 hours were unsuccessful. Grinding aids such as urea and an aryl alkyl sulphonic acid, in quantities up to 0.2% w/w of the graphite were also used, but the results were inconclusive.

Preliminary work on micronizing of graphite was not encouraging. Pin disc milled graphite was reduced to a material with an absolute size constant of 15 microns and distribution constant of 2.5 in a 24–inch mild Steel Micronizer fed at 75 lb/hr. However, the micronized graphite had a 10% coarse fraction with an absolute size constant of nearly 150 microns. Also, the contamination was greater than expected, showing an increase of 0.04% iron. Further detailed investigation would be required to establish whether micronizing is a satisfactory method for producing fine artificial graphite for compaction studies, but this was not warranted in the present work.

3.3 Surface Area

Surface area has been measured by the B.E.T. nitrogen adsorption method. Figure 8 shows the increase of surface area with grinding time for the grinding of 500 g. of graphite with steel balls at 60% critical speed. The maximum specific surface obtained was 18.4m^2/g. for a 500 g. charge ground with Ni–hard balls for 80 hours. It is of interest that the specific surface of this material, calculated from the Micromerograph size distribution assuming spherical particles, is only 0.4m^2/g.

The specific surface changes substantially on acid leaching. Thus, the contaminated material, with a surface area of 18.4m^2/g, after refluxing for 3 hours with 2N HCl gave a surface area of 11.2m^2/g. The surface area did not change appreciably on further refluxing with 3N HNO_3.

3.4 Contamination Studies

The impurities of interest in the original graphite blocks were:–

0.001% Fe, 0.004% Si, Cr, Ni, Al nil (spectrographic) and 0.016% total ash. The jaw crusher gave no detectable pickup, and after pin disc milling the iron content was 0.002%, with no change in silicon or aluminium. In the grinding studies with steel balls only the change in iron impurity was investigated, while with the Ni–hard balls changes in iron, nickel and chromium were determined.

Figure 9 shows the effect of time of grinding and graphite charge to the mill on the rate of iron pickup with steel balls. The smaller charge gives the greater rate of contamination increase. The same effect is shown in Table II for grinding with Ni–hard balls.

TABLE II

CONTAMINATION BY GRINDING WITH NI–HARD BALLS

Impurity	500 g. graphite 80 hr. grinding 60% critical speed	750 g. graphite 100 hr. grinding 60% critical speed
Fe	0.62%	0.14%
Cr	0.016%	0.003%
Ni	0.024%	0.005%

The contamination produced in 500 g. charges of graphite with the three types of grinding media is given in Table III.

TABLE III

CONTAMINATION FROM DIFFERENT GRINDING MEDIA

Impurity	Porcelain Balls 100 hr. grinding 60% critical speed	Steel Balls 100 hr. grinding 60% critical speed	Ni–hard Balls 80 hr. grinding 60% critical speed
Fe	0.005%	0.034%	0.62%
Si	0.007%	–	–
Al	0.003%	–	–
Cr	–	–	0.016%
Ni	–	–	0.024%
Total Ash	0.048%	0.075%	1.02%

Porcelain balls gave the least total contamination, but it was considered that contamination produced by the steel or Ni–hard balls would be more readily removed than that from the porcelain balls. Since this work was connected with the preparation of nuclear fuel irradiation specimens, the purity of the graphite was of paramount importance. For this reason only preliminary runs were made with porcelain balls, and the detailed grinding study was made with steel and Ni–hard balls. Table IV shows that refluxing the contaminated graphite with HCl readily removes Fe, Cr and Ni, but this was not achieved by refluxing with 3N HNO_3.

TABLE IV

CONTAMINATION REMOVAL BY ACID LEACHING

Material	Acid	Time of Leaching (hrs.)	Impurity Content (%)
Graphite ground 100 hours with steel balls	–	Nil	0.034 Fe
	0.5N HCl	8	0.002 Fe
	2N HCl	1	0.003 Fe
	2N HCl	3	< 0.001 Fe
	3N HNO_3	13	0.012 Fe
Graphite ground 80 hours with Ni–hard balls	–	Nil	0.62 Fe, 0.016 Cr, 0.024 Ni
	3N HCl	2.5	< 0.001 Fe, < 0.001 Cr, < 0.001 Ni

4. DISCUSSION

4.1 Size Reduction

The ball milling procedure used in this work was not capable of producing graphite finer than 50% minus 9 microns as measured by the Micromerograph. Prolonged grinding times will not produce finer material, as seen in Figure 2. This is probably due to coating of the grinding media with graphite, thereby cushioning any grinding effect. The use of finer balls and also grinding aids produced no significant improvement.

The grinding rate may be increased by

(a) decreasing the graphite charge in the mill (Fig. 6),

(b) increasing the mill speed (Fig. 5), and

(c) using Ni–hard balls (Figs. 2, 3 and 4).

However, an increase in the grinding rate is generally accompanied by an increase in contamination rate, although the work on contamination obtained with an increase in mill speed was not conclusive. Coghill and De Yaney state that the ball wear/unit power is independent of the ball size and of the mill speed (12). In this work, however, it was impracticable to measure the power input to the mill, and hence no conclusions can be drawn on the ball wear/unit power, or the rate of grinding/unit power.

Ni–hard balls caused a marked increase in contamination and this was probably related to the rough surface of these balls. The contamination results were not as consistent as may be expected from the precautions taken in sampling. It is believed that the contamination is not only present as a fine coating on the graphite, but also as discrete particles unevenly distributed through the material. This makes sampling of representative small weights of material very difficult.

4.2 Size Distribution

The Micromerograph uses an air sedimentation method of size analysis, and hence it measures a Stokes diameter. According to Hawksley (13), a Stokes diameter has no real significance in grinding and possibly a better method to use would be a microscopic analysis which measures some function of the projected area of the particles. However, it is considered doubtful whether the projected area of a particle has a direct connection with grinding of the particle. Microscopic analyses are tedious, and apparatus was not readily available for performing these analyses. Micromerograph analyses are rapid and reproducible, and hence have been used for size measurement in the sub–sieve range. Other methods may have been used, and although possibly giving different values of absolute size constant and distribution constant, they would probably have shown the grinding trends equally as well as the Micromerograph. It must be realised, however, that there is no adequate measure of absolute particle size, and the present data only indicate the direction of changes in the particle size distributions.

Size reduction of brittle metals satisfies the Rosin–Rammler function, giving a slope of 1 (14). Coal in the sieve sizes also gives a slope of 1 or less when the size distribution data are plotted according to Equation 1 (10), (11). In the present work it was found that petroleum coke and coal tar pitch, the raw materials in graphite manufacture, did not satisfy the Rosin–Rammler function when crushed in the jaw crusher. However, it has been shown that graphite does obey the function, giving a distribution constant of approximately 1 in the size range 70 – 1000 microns, and approximately 2 above and below this size range.

The value of 2 for graphite could be due to a physical characteristic of the material, the method of size reduction, or the method of size measurement. Some work has been done in an endeavour to elucidate this matter.

Coarse crystalline limestone crushed in the jaw crusher gave the same inflexion as did graphite. This inflexion is almost certainly due to the screening action of the jaw crusher. Bennett (11) mentions this effect for crushing of coal in a hammer mill with closely spaced screen bars. It is well known that a high value of the distribution constant is often associated with some form of classifying process (10), (11). Hence, it is suggested that the slope of 2 in the coarse range is due to the screening action of the jaw crusher, and is not due to a physical property of the graphite.

There remains the question of the high slope in the sub–sieve range, as this has not been previously reported for fine materials. A reduction was performed omitting the pin disc milling stage by passing directly from jaw crushing to ball milling. This gave substantially similar curves, and shows that the pin disc milling stage does not control the distribution. Since both the pin disc mill and the ball mill gave essentially the same distribution constant, the method of reduction does not control the slope in the sub–sieve range.

The slope of 2 in this range may have been related to the method of size analysis, i.e., the Micromerograph. In order to clarify this matter size analyses were carried out by several other methods on graphite that had been ground in the pin disc mill. The size distribution determined by the Andreasen method gave a slope of 1.2, the Haultain Infrasizer gave 1.4, and a microscopic count gave 2.1. These compare with $n = 1.7$ found with the Micromerograph. Spherical particles were assumed in calculating the weight distribution from the microscopic number count. This is a simplification, and is not valid if the particle shape varies with size. However, if the shape does not vary with size, the value of n found from the Rosin–Rammler plot will not depend on the assumption of spherical particles.

The size reduction procedure and the method of size analysis can influence the distribution constant found from Equation 1, but it is believed that neither are responsible for the unusually high value of n found for graphite in the sub–sieve range. The size distributions of other fine materials have been measured by various methods, and Table V shows that these also have high values of the distribution constant.

TABLE V

ROSIN–RAMMLER CONSTANTS FOR VARIOUS MATERIALS

Material	Method of Preparation	Method of Size Analysis	Absolute Size Constant $\bar{x}$ microns	Distribution Constant n
Graphite	Ground in pin disc mill (no classification)	Micromerograph Andreasen Infrasizer Microscopic	40 95 40 75	1.7 1.2 1.4 2.1
Graphite	Ground in ball mill (no class–ification)	Micromerograph Microscopic	11.5 15	2.0 2.5
Tungsten	Hydrogen reduction of WO_3	Micromerograph Microscopic	5.5 2.0	2.7 2.1
Blanc Fix	Precipitation	Micromerograph	4.5	2.6
Talc	Ground in Raymond mill with internal classifier	Micromerograph Andreasen	16 30	2.7 2.0
Bituminous Coal	Ground in ball mill (no class–ification)	Micromerograph	16	2.5
Coal	Ground in sieveless tube mill (Rosin–Rammler) (10)	Sieve	Variable	1.5

It is significant that the values obtained are higher than any previously reported, and that reasonable agreement was found by independent methods of size analysis for materials prepared by widely different processes. Rosin and Rammler found no values of n greater than 1.5 (10), and Hüttig and Sales claim that n = 1 for grinding of brittle metals (14).

4.3 Surface Area

As may be expected, the surface area of ball milled graphite increases with grinding time. However, it is not clear why the B.E.T. surface area of graphite ground with Ni-hard balls (18.4 m^2/g for 500 g. charge ground 80 hr.) is greater than that of material ground with steel balls (10.3 m^2/g. for 500 g. charge ground 120 hr.) when they have substantially the same size distribution measured by the Micromerograph. This would be explained if the Ni-hard ground material contained a much greater proportion of minus 2 micron particles than the graphite ground with steel balls, as the Micromerograph does not measure graphite particles finer than 2 microns.

The B.E.T. surface area is very considerably higher than that calculated from the size distribution assuming spherical particles. Although the particles are very angular, as seen in Figure 10 for -200 + 300 mesh pin disc milled graphite, and allowing for the fact that the Micromerograph does not record particles smaller than 2 microns, this large difference in surface area must be related to internal surface remaining in the fine particles.

The marked decrease in surface area obtained after leaching with HCl is exemplified by the decrease in number of very fine particles seen by comparing Figures 11 and 12. Micromerograph measurements of the size distribution of leached and unleached material support this observation although the size distribution change is only small. Fine material was not physically lost during the leaching process, and it is difficult to explain why the surface area and size distribution change after this treatment.

5. CONCLUSION

Successive size reduction of artificial graphite in a jaw crusher and pin disc mill, followed by grinding in a ball mill for up to 120 hr. has produced material approximately 50% finer than 9 microns as measured with the Micromerograph. The surface area of the product, measured by B.E.T. nitrogen adsorption, ranges up to 18.4 m^2/g.

Ni-hard grinding balls are preferred to steel balls having a high iron carbide content, as the former grinding medium gives the greater rate of grinding. The contamination produced by Ni-hard balls is higher than that obtained with steel balls, but leaching with boiling 2N HCl for 3 hr. effectively removes impurities introduced by either grinding medium. The surface area of the graphite decreases by about 30% on leaching.

The Rosin-Rammler function has been found to apply to the size distributions of the graphite after each stage of size reduction. In the size range 70 - 1000 microns the distribution constant has a value of 1, which is consistent with published data for other materials. Above 1000 microns the distribution constant is about 2, and this is due to the classifying action of the jaw crusher. Below 70 microns, in the sub-sieve range, the distribution constant also approaches 2. Such a high value has not been previously reported for fine materials. Experiments have shown that the high value of n is not due to either the method of comminution or the method of size analysis. Other fine materials have also shown this large value of the distribution constant in the sub-sieve range, and hence it is concluded that it is not a peculiarity of artificial graphite.

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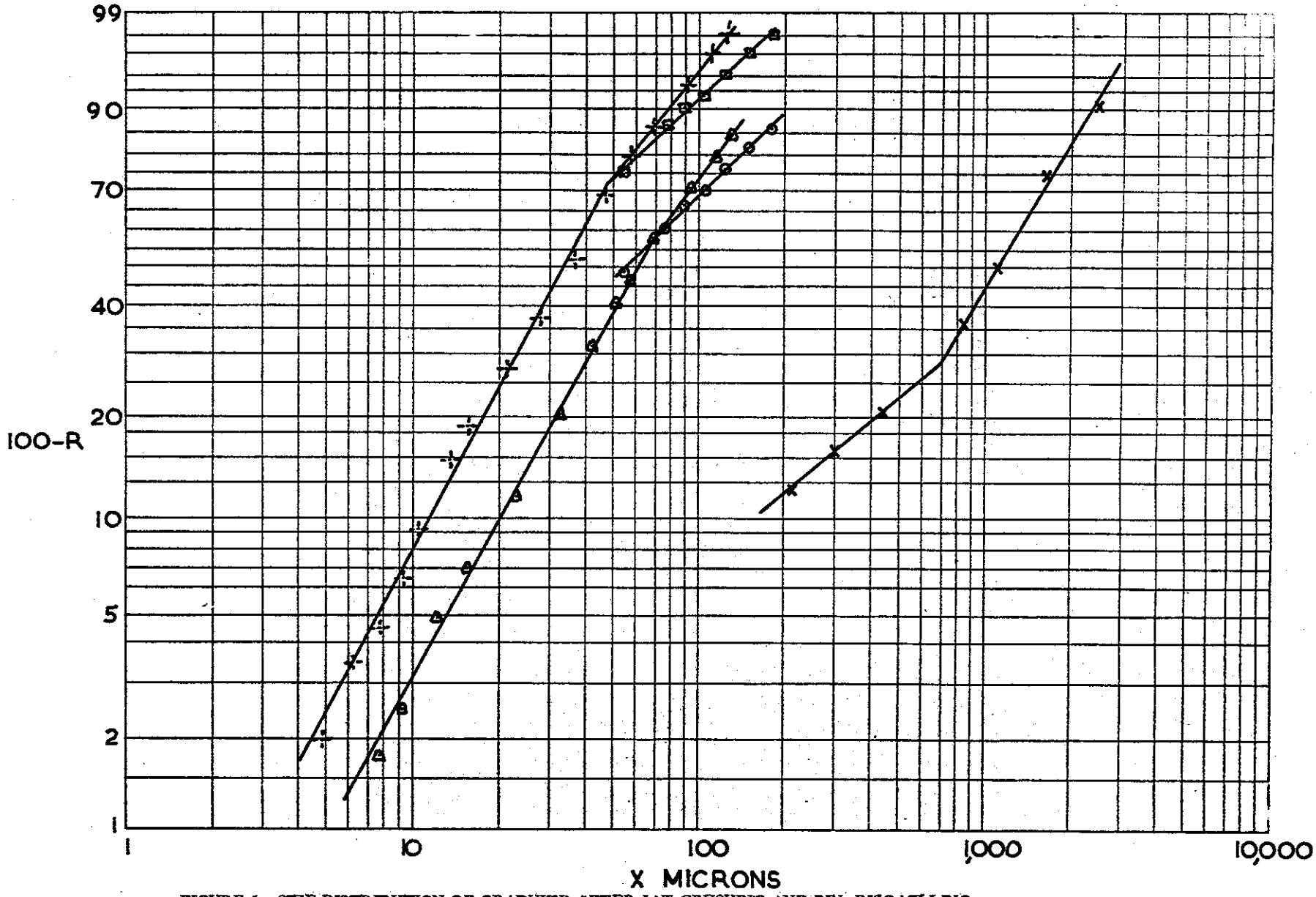

FIGURE 1. SIZE DISTRIBUTION OF GRAPHITE AFTER JAW CRUSHING AND PIN–DISC MILLING.

X Screen analysis; two passes through jaw crusher
0 Screen analysis; one pass through pin–disc–mill
Δ Micromerograph; one pass through pin–disc–mill
□ Screen analysis; two passes through pin–disc–mill
\+ Micromerograph; two passes through pin–disc–mill

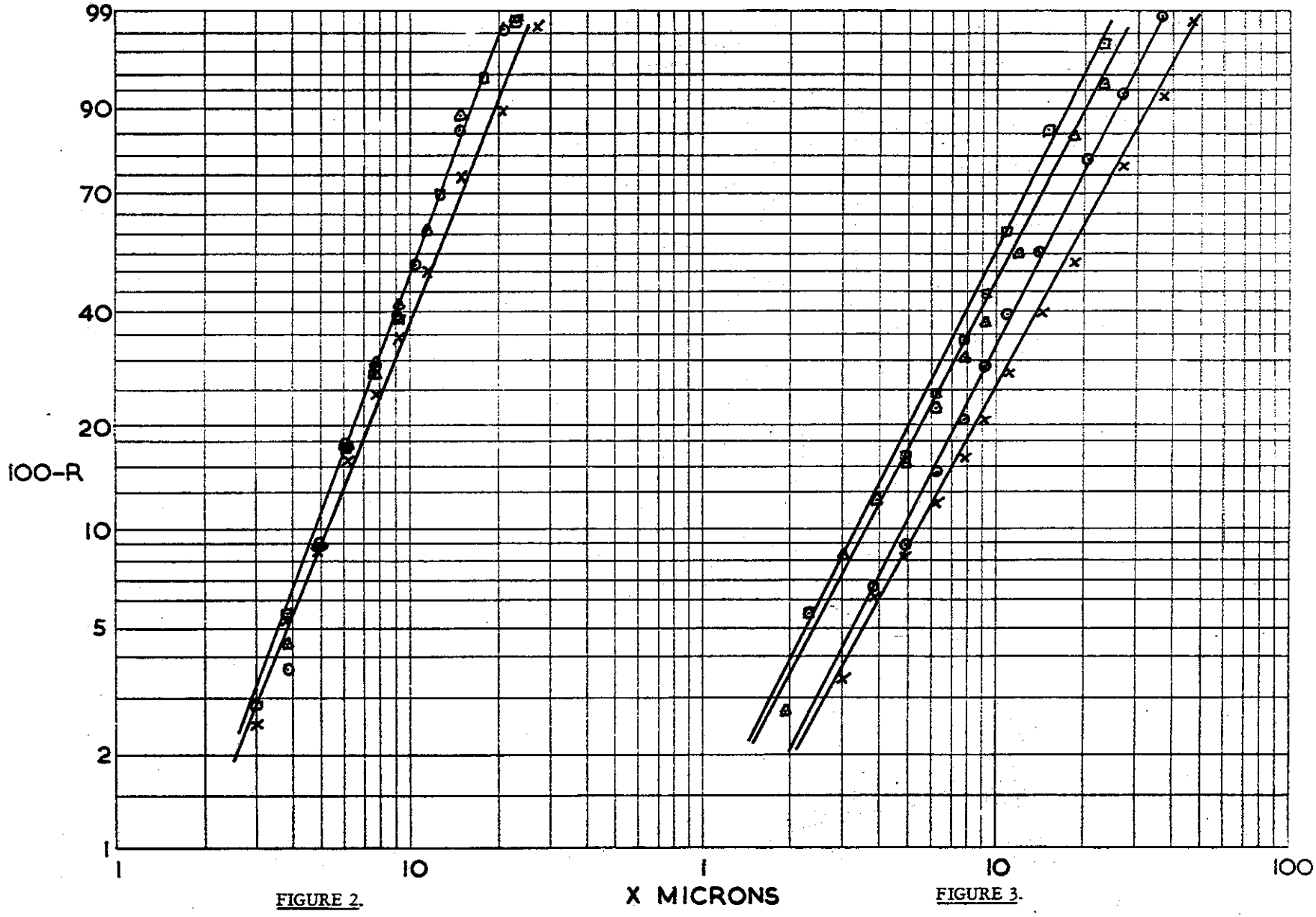

FIGURE 2.

EFFECT OF GRINDING TIME ON BALL MILLING OF GRAPHITE NI HARD BALLS
500 g. graphite charge
Mill speed 60% critical

X 20 hr.
0 40 hr.
Δ 60 hr.
□ 80 hr.

FIGURE 3.

EFFECT OF GRINDING TIME ON BALL MILLING OF GRAPHITE STEEL BALLS
500 g. graphite charge
Mill speed 60% initial

X 20 hr.
0 40 hr.
Δ 60 hr.
□ 100 hr.

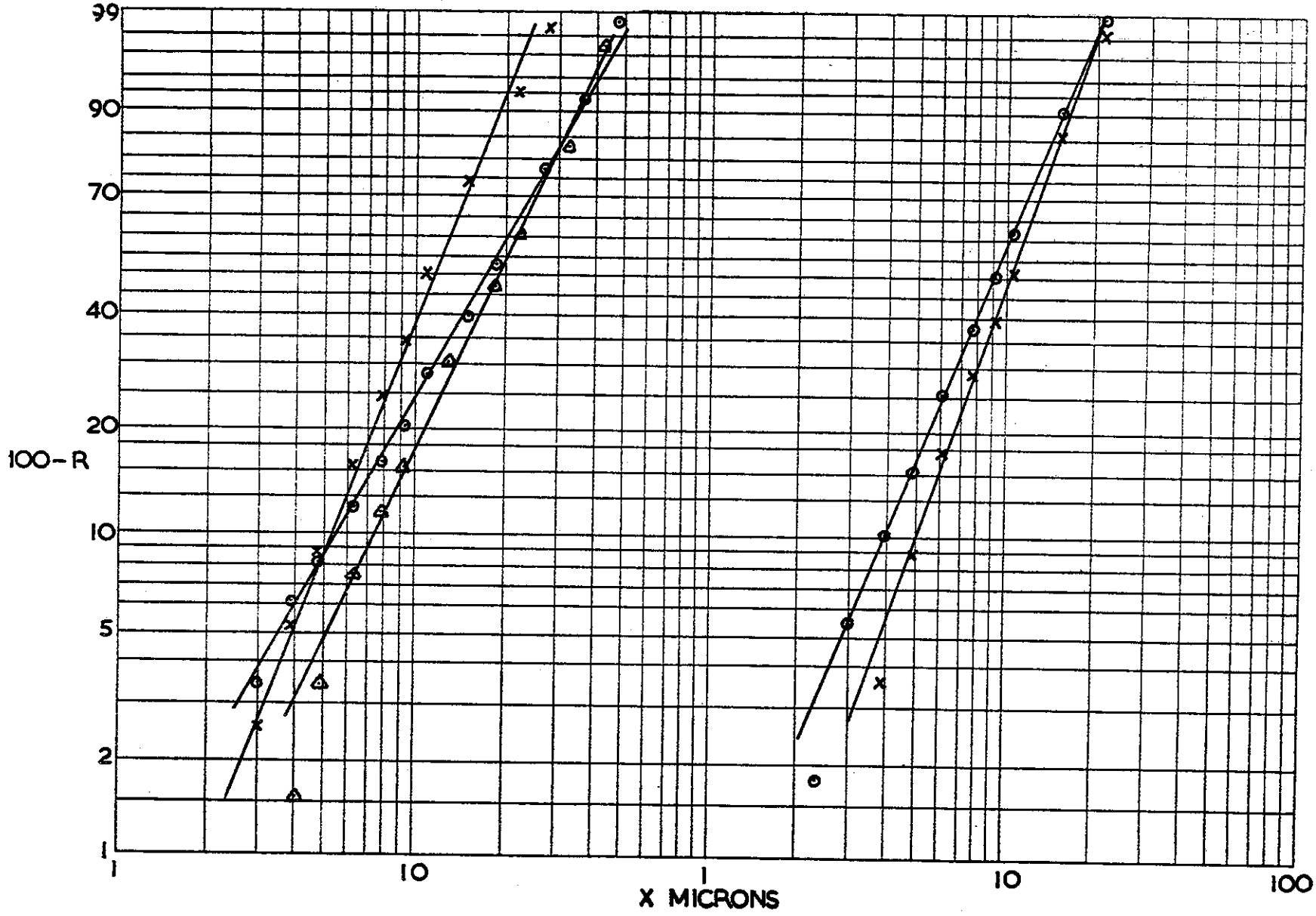

FIGURE 4.

EFFECT OF GRINDING MEDIA ON BALL MILLING OF GRAPHITE
500 g. graphite charge
Mill speed 60% critical
X Ni–hard 20 hr. grinding
0 Steel 20 hr. grinding
Δ Porcelain 30 hr. grinding

FIGURE 5.

EFFECT OF MILL SPEED ON BALL MILLING OF GRAPHITE
500 gm. graphite charge
Ni–hard balls 40 hr. grinding
0 80% critical speed
X 60% critical speed

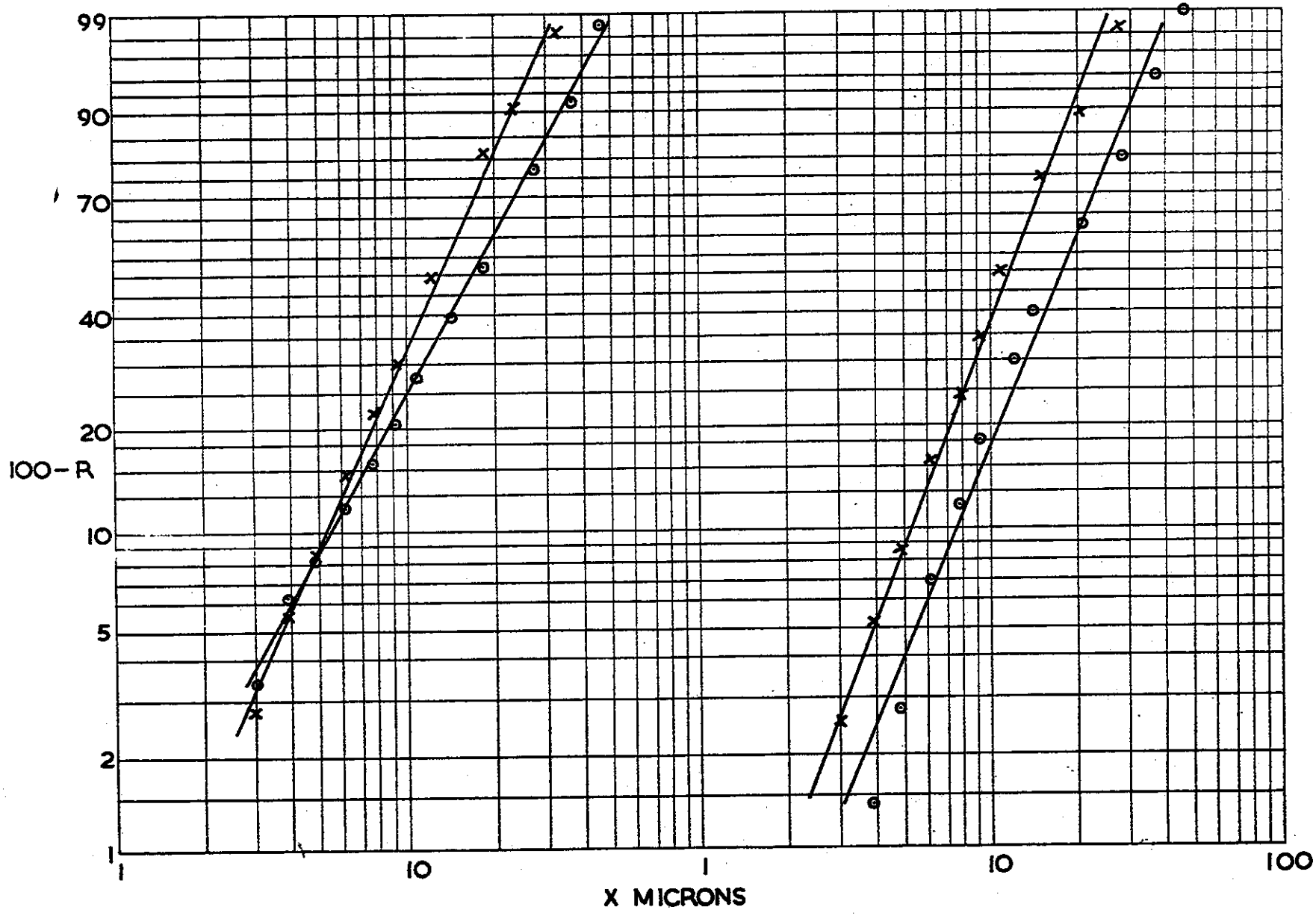

FIGURE 6.

EFFECT OF MILL CHARGE ON
BALL MILLING OF GRAPHITE

Steel balls 20 hr. grinding
Mill speed 60% critical
X 250 g. charge
0 500 g. charge

FIGURE 7.

EFFECT OF MILL CHARGE ON
BALL MILLING OF GRAPHITE

Ni-hard balls
Mill speed 60% critical
0 750 g. charge 18 hr. grinding
X 500 g. charge 20 hr. grinding

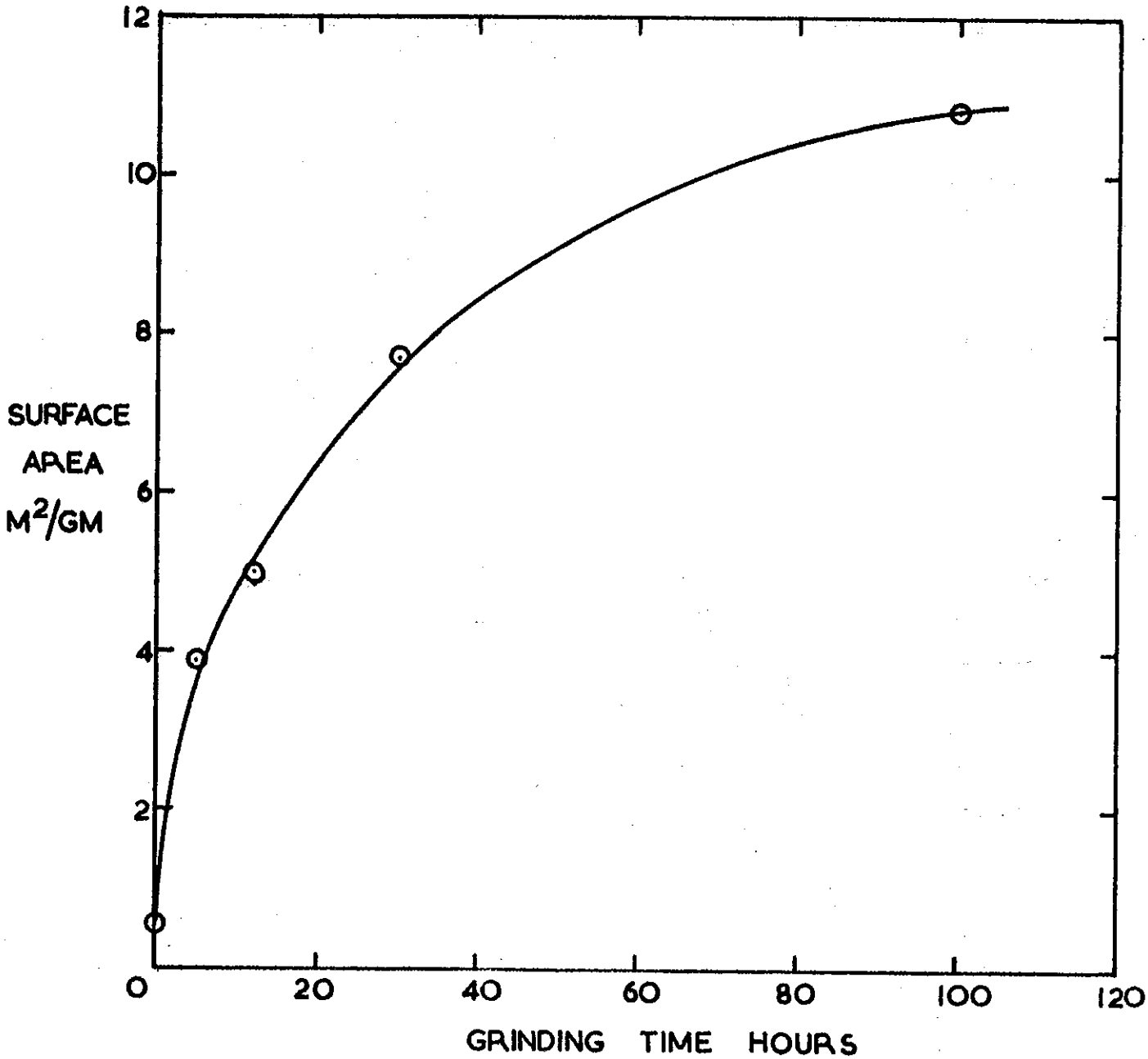

FIGURE 8. EFFECT OF BALL MILLING ON SURFACE AREA OF GRAPHITE
500 g. graphite charge
steel balls
Mill speed 60% critical

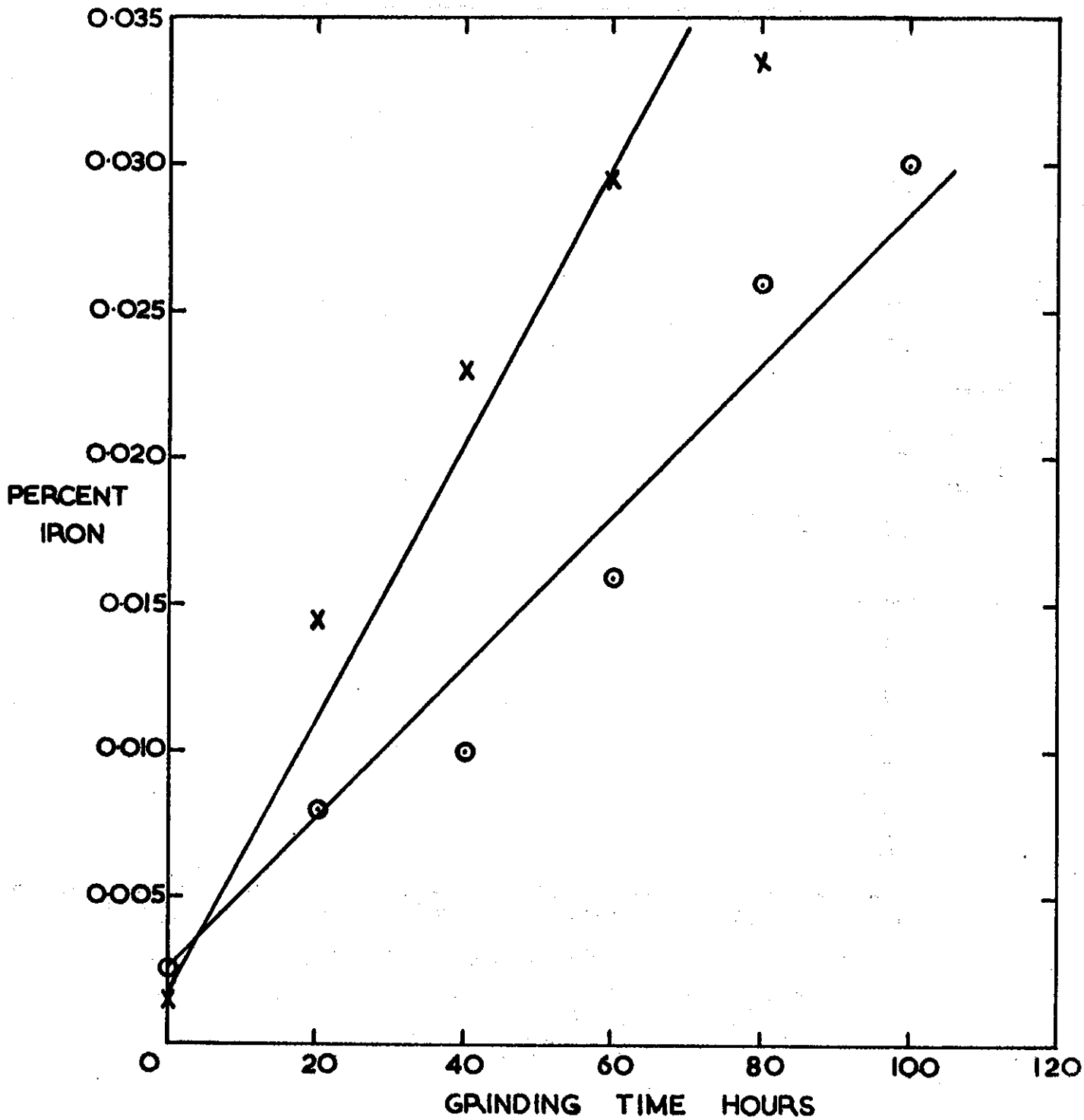

FIGURE 9. EFFECT OF BALL MILLING ON IRON CONTAMINATION OF GRAPHITE
Steel balls
Mill speed 60% critical
X 250 g. charge
0 500 g. charge

FIGURE 10. GRAPHITE PARTICLES

– 200 + 300 mesh pin disc milled
220 x

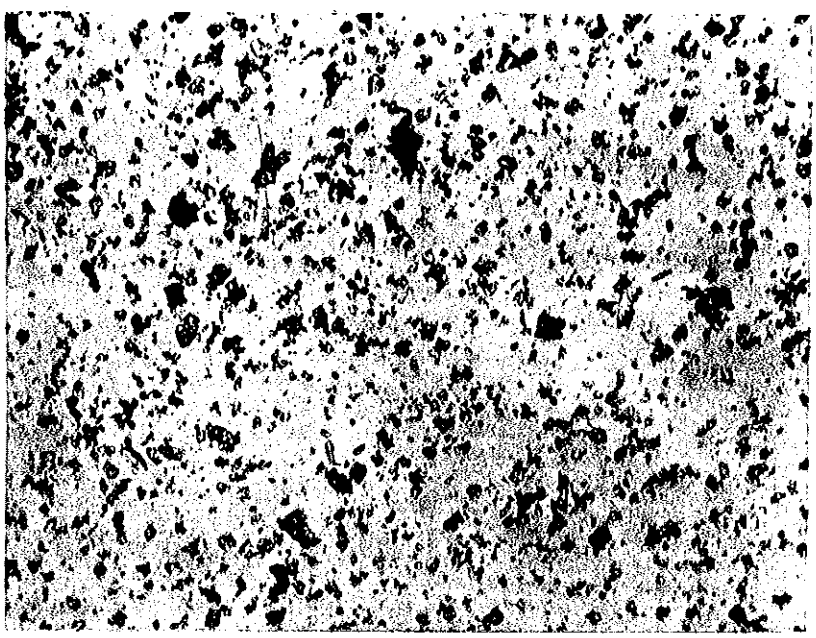

FIGURE 11. GRAPHITE PARTICLES GROUND 80 HR.

Ni–hard balls 118 x

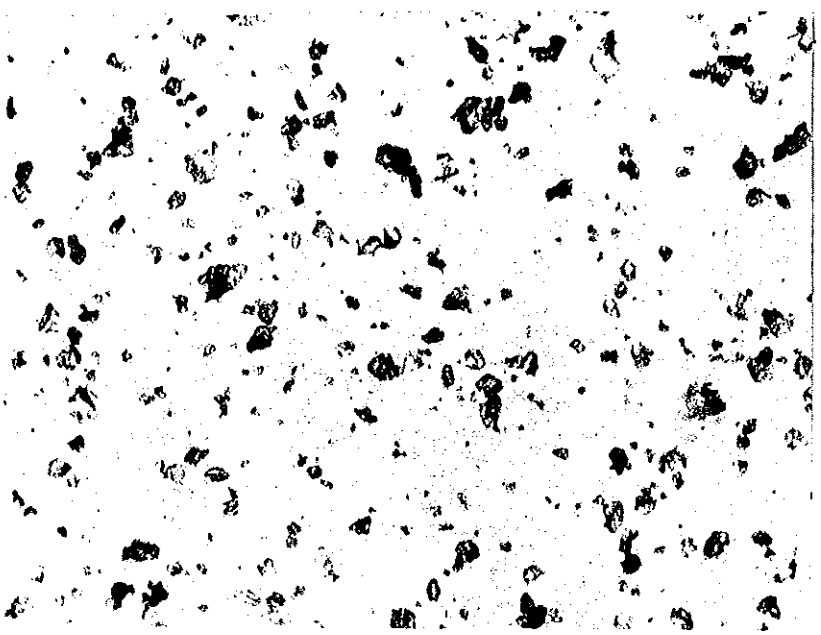

FIGURE 12. GRAPHITE PARTICLES GROUND 80 HR.

Ni–hard balls
Leached 2–1/2 hr. with 3N HCl
118 x