MICRONIZED HIGH PURITY ALUMINA

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Submicron alpha alumina is produced by Keramont Corporation in Tucson Arizona. Intended application for this powder is high quality tape casting applications, electronic packages, abrasives, and additive for magnetic media formulations. Several production alternatives based on mechanical fragmentation are discussed. Grinding kinetics are described within the framework of macroscopic population balance models. Properties of this product are compared to precipitated spherical aluminas also produced by Keramont.

INTRODUCTION

Keramont Corporation has developed a process to produce high purity aluminas to be used as an abrasive additive in magnetic tape formulations, and other abrasive and ceramic applications. This process is based on the mechanical fragmentation of α-alumina. Keramont Corporation has also developed through its Italian branch based in Novara, Italy, another process to produce ultra high purity spherical alumina to be used in applications requiring highly smooth surface and high purity. This process is based on the controlled precipitation of alumina. This paper describes some of the features associated with the fragmentation technology and compares these aluminas with those obtained through precipitation.

Sub-micron aluminum oxide additions to magnetic media formulations that are based on ferric oxide are common technology since they have been found to significantly reduce dropouts by providing a gentle cleansing action to the head, improve media durability, and reduce head-media surface friction. Controlled alumina additions are used for both flexible and rigid media, each application imposing different requirements on the size of powder to be used.[1]

Particle size distribution is one of the most important parameters to consider when using an abrasive additive for the purpose thought here. If the alumina particle size is too coarse for a given coating thickness, a rough surface finish can result, adversely affecting performance and damaging the magnetic head. Tight control of the particle size is very important since state of the art magnetic media formulation requires the use of very fine magnetic particles.[2-3]

Size analysis of these powders demands great care and the use of state of the art analytical techniques such as automatic light extinction sedimentometers, X-ray sedimentometers or automatic laser scattering counter instruments.[4] For any one of these techniques a good dispersion of the powder in a liquid medium prior to analysis is mandatory to obtain good results. The use of a dispersant to control particle surface charge may be needed to achieve a good dispersion. Microscopic examination of slurry to assess the state of dispersion prior to analysis is the preferred technique to assess the appropriateness of the dispersion.

Particle size of the alumina to be used has to be matched to the intended final formulation application. Table I shows some

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typical alumina size specifications for different magnetic media formulation applications.

The increased sinterability associated with very fine aluminas makes them attractive to the production of electronic substrates and packages. Advantages are also found in the forming of green compacts using finer particles since better green densities and better surface rugosity are obtained.[5] The traditional approach to the production of very fine aluminas has been based on mechanical fragmentation[6-9], on chemical means making use of sol-gel precursors[10], on precipitation[11-12], etc. Other more sophisticated approaches include plasma techniques and aerosol techniques.[13-15] It could be said that each one of these approaches present some advantages and some disadvantages. In general, mechanical size reduction produces powders which are irregular in shape and of higher degree of contamination but at a reduced cost. Chemically produced fine powders could present higher purity and controlled morphology but are in almost all cases more expensive.

The use of precipitated spherical aluminas presents advantages. The manufacturing of these aluminas allows for tight control of chemical composition and controlled particle morphology. High purity aluminas are produced while particle size is controlled and particle shape is kept spherical. This type of alumina presents high reactivity during sintering and produces very smooth surface finish making it a prime candidate for the production of high quality electronic substrates and tape casting applications. These aluminas can also be used for magnetic media to produce high quality rigid disks.

An approach to the industrial production of these two types of aluminas developed by KeraMont Corporation is presented in this paper.

MICRONIZED ALUMINAS BY MECHANICAL FRAGMENTATION.

Different grinding techniques were used to accomplish the size reduction of α-alumina. They included dry ball milling, wet ball milling, and stirred ball milling. In addition, air classification techniques were used in combination with mechanical size reduction to obtain ultrafine aluminas.

Dry Ball Milling.

Dry ball milling was accomplished using mills as large as a 52 gallons ceramic jar ball mill. Finer product was obtained as the size of the mill increased. Grinding media consisted of 171 kg of alumina cylinders for the 52 gallon mill, ranging in size from 1 1/8 x 1 1/8 inches to 1/2 x 1/2 inches. Parameters investigated to optimize milling performance were: amount of alumina charged, tumbling speed, and alumina type.

Product size distributions obtained after operating the mill at 100% voids filling (24 kg of alumina) and 70% voids filling (17 kg of alumina) are shown in Figure 1 and Figure 2 respectively. For both runs the speed of the mill was kept at 53% of critical. Operated under these conditions the mill delivered 1.38 kW of power. As shown in Figure 1 and Figure 2, powders with a top size of 3 microns and a median size of 0.43 microns were obtained in either case. The grinding seemed to progress normally at both 100% and 70% voids filling, the 70% resulting in slighter finer products as expected due to the larger specific energy input given to the material under these conditions.

The production of submicron alumina was accomplished with different types of alumina. For example, dry grinding tests were performed on a sample of calcined Alcoa C-33 aluminum tryhydrate and on a sample of Ceralox alumina for comparative purposes. Results are shown in Figure 3 and Figure 4. Notice in these figures, that the Ceralox and Alcoa aluminas were also reduced to median size of 0.4-0.5 microns indicating

that either one could be used. The selection of the appropriate type of raw alumina will be dependent upon the level of impurities allowable in the final product and cost considerations.

Wet Ball Milling.

The finest alumina products were obtained from wet ball milling runs using either alumina or zirconia grinding media. Water or acetone were used as the grinding fluid.

As shown in Figure 5, product with 0.23 microns median size was obtained when using the zirconia media at 25% solids at 58% of critical speed. Also notice that this product only has 0.6% material coarser than 0.5 microns.

Figure 6 shows the size distribution of the product obtained after 24 hrs of grinding with alumina media at 25% solids and 61% of critical speed. Corresponding median size was 0.28 microns with only about 1% of the particles being between 0.6 and 1 microns (top size). Grinding with the alumina media presents as advantages lower cost and reduced level of product contamination.

The percent solids of the mill charge had reduced effect on mill performance. After 24 hours of grinding there was a small difference in the size of the products obtained. For example, product with median size of 0.25, 0.27 and 0.28 microns were obtained when grinding at 25%, 35% and 50% solids respectively. Evidently, mill percent solids will significantly affect mill capacity.

Figure 7 shows a TEM photomicrograph of a typical micronized alumina product. Notice that most of the particles are well below 1 micron, with a median size at around 0.3 microns. Particle shape is irregular. Figure 8, shows a cross section of a sintered sprill with this type of alumina. It was observed that this alumina sinters very easily reflecting that it is very reactive. Also, a grain inhibitor such as magnesium oxide may be needed to control grain size during sintering.

Stirred Ball Milling.

The alternative of using a stirred ball mill to produce submicron alumina was also investigated. This type of mill has been shown to be appropriate for the ultrafine grinding of other of several powders[16-21]. Stirred ball mills have increased capacity and are more energy efficient than conventional ball mills.

A product with 3 microns top size and 0.36 microns median size was obtained after a three hour grind using a Union Process Attritor mill Model S-1 of 1.5 gal. Grinding media consisted of 1/4" in diameter steel balls. Results are presented in Figure 9. Although this mill enhances the size reduction of the coarser particles, the level of iron contamination obtained (approximately 8%) was unacceptable to produce high purity submicron alumina.

To avoid iron contamination, alumina and zirconia media were tried on a polyethylene lined Attritor mill. Results have been plotted in Figure 10 and Figure 11 respectively. A product of 0.47 microns median size was obtained with the alumina balls after 16 hours of grinding, and a median size of 0.37 microns was obtained after 16 hours with the zirconia media. A major reduction in iron contamination was obtained. However, the impurity level caused by media wear was still high.

It can be concluded that the larger the density of the grinding media, the finer the product obtained. Even much shorter grinding times with the steel media resulted in finer products. This is expected since larger specific energy inputs are provided to the mill charge with the heavier media and more energetic ball-ball and ball-impeller interactions are provided. It has been proposed in previous publications that this condition will produce improved

grinding performance usually found in conventional stirred ball milling[17].

Some additional testing with Attritor mills was conducted at Union Process, Akron, Ohio[22], to evaluate alternatives to reduce contamination. An alumina tank, a tungsten carbide impeller, and a polyurethane coated impeller were tried using alumina balls as grinding media. A product with 0.69 microns median size was obtained after 4.5 hrs of grinding with the tungsten carbide impeller while the polyurethane coated impeller produced an alumina powder of 0.61 microns median size after 5 hours of grinding. Less contamination was obtained with the ceramic tank, alumina media, and polyurethane coated impeller.

Another test was performed at Union Process in which the polyurethane coated impeller was used with 3/16" silicon nitride media in an alumina tank. The objective of this experiment was to increase mill performance by using a heavier media. A product with 0.4 microns median size was obtained after 4 hours of grinding which is finer than those obtained with the alumina media. However, a silicon content of 2.6% was measured in the product. This approach has also been followed by other researchers to produce fine pure silicon and silicon nitride.[23]

Grinding Kinetics Modelling

It is always desirable to be able to correlate grinding time with product size. A correlation like this could be used for design and scaleup purposes, especially if the correlation represents different running conditions and explains the behavior of mills of different size. These correlations have been developed in the past between net specific energy input and product fineness. The use of net specific energy facilitates making comparisons among mills of different sizes on the same basis. For the analysis presented here, grinding time was used instead of specific energy since power drawn by the different mills used was not always available. This was done since to a good approximation grinding time is directly proportional to net specific energy provided during a certain period of time. This simplification is acceptable so long the power drawn by the mill is constant along the duration of the grinding.

The approach presented here is based on an empirical energy versus size reduction relationship of the type of the Charles' equation[24-25]. This correlation has been found to be useful in describing the behavior of both tumbling and stirred ball mills[16-19],[8]. According to Charles, the specific energy provided to the particulate assembly being ground can be related to size reduction using the following relationship,

$$\bar{E} = \frac{P \cdot t}{M} = A \left[d_{50,P}^{-\alpha} - d_{50,F}^{-\alpha} \right] \qquad (1)$$

where

$\bar{E}$ = specific energy

P = power drawn by the mill

t = grinding time

M = solids charged into the mill

$d_{50,P}$ = product median size

$d_{50,F}$ = feed median size

A, α = constants

If the extent of the size reduction is large ($d_{50,F} \gg d_{50,P}$), typically found in cases when generating ultrafine powders, like is the case here, the effect of the feed term in Equation (1) becomes negligible. Equation (1) can then be simplified as follows,

$$t = A' \cdot d_{50,P}^{-\alpha} \qquad (2)$$

When grinding progress is well represented by Equation (2), a plot of the product median size versus grinding time should be a straight line of slope $-\alpha$ when plotted on a

log-log chart.

Equation (2) was used to fit data obtained from dry ball milling, wet ball milling, and stirred ball milling. As shown on Figure 12, Figure 13, and Figure 14, in every case the data could be fitted with a straight line indicating that this type of empirical relation can be successfully used to represent grinding progress for any of the grinding devices used in this study. The same of data analysis performed using specific energy instead of time would had allowed for a direct comparison of the relative grinding efficiency among the different mills[17-19].

The Charles' equation allows for predictions of product median sizes after a given grinding time. A more detailed mathematical representation of the grinding process is obtained when using the population balance model for first-order grinding kinetics. This model allows for predictions of complete product size distribution after a given grinding time knowing the feed size distribution and the kinetic parameters used in its mathematical derivation. The model allows for the fitting of a set of selection function and breakage functions based on an experimental data set or the prediction of product size distribution when these kinetic parameter are known. Model development has been explained in detail by several researchers[26-27]. A detailed description of the model formulation is beyond the scope of this paper. This model has been successfully implemented in a computer program named ESTIMILL[28] which can be used to describe both batch and continuous grinding operations.

Using the ESTIMILL program, grinding data obtained for one of the dry ball milling experiments was fitted to determine a set of breakage and selection functions and to determine model applicability to this grinding operation. Experimental and predicted data are shown on Figure 15. It can be concluded that the first order grinding kinetics model made good predictions of experimental results for the shorter grinding times only. As the retention time increased, corresponding to larger specific energy inputs, predicted product size distributions deviated from the experimental ones to a larger extent.

Losing the applicability of linearity after large retention times is typical for ultrafine grinding applications like those studied here. This phenomenon has been reported previously[29] and is believed to be caused by changing grinding conditions inside the mill. After extended grinding times the material being ground becomes very fine and of increased strength as most microcracks have been reduced during the initial size reduction stages. At the same time, the grinding media is unable to keep the same efficiency of grinding and linearity through out the grinding process is lost. The accurate representation of this type of system requires the use of more complicated models[17].

Air Classification.

Three different test were performed on micronized alumina obtained from the 52 gal. ball mill to evaluate the alternative of using air classification of the product after grinding to remove the particles coarser than 1.5 microns. Test were run at Alpine, Majac, and Nisshin. All tests were run on an alumina sample with a top size of 3 microns and a median size of 0.44 microns. On each one of these tests, the feasibility of removing the coarser particles was demonstrated.

The Alpine test was performed in a Turboplex Ultra Fine Classifier 50 ATP[30]. The fine fraction obtained had a median size of 0.44 microns and showed improvement only in the coarser range of the distribution.

Majac used an A-12 Acucut Classifier[31]. Under best operating conditions, a powder with a median size of 0.46 microns was obtained in the fine fraction and a powder with a median size of 0.34 microns was obtained from the air filter. About 50% of the feed powder was collected into these two

fractions.

The Nisshin tests were performed in a Turbo Classifier TC-15N[32-34]. Under best operating conditions, the air cyclone underflow had a median size of 0.40 microns and a powder with a median size of 0.29 microns was obtained from the cyclone overflow filter. About 40% of the feed powder was collected into these two fractions.

It could be concluded, that advanced air classification technology could be used to improve the quality of the magnetic media alumina product by removing the coarser particles. However, no more than 50% of the product reported to the fine fraction as product and some contamination was added in the classification process. The incremental cost derived from the use of such process plus the extra equipment cost make this alternative less attractive for this application.

Product Specifications

Results reported in the previous sections demonstrate the feasibility of producing several different grades of submicron aluminas to be used in magnetic media formulations, as an abrasive powder, or in the manufacturing of high quality alumina electronic substrate. After consideration of product fineness and purity level requirements, Keramont designed a process to produce different grades of micronized aluminas at reduced cost. Commercial submicron alumina products developed by Keramont Corporation have been named KeraMag aluminas. Their specifications are given in Table II.

SPHERICAL ALUMINAS

Spherical aluminas are produced at Keramont facilities at Novara, Italy. The emphasis of this technology is in the production of controlled morphology powders of a given size. The technology is based on the precipitation of spherical particles out of a solution under very well controlled conditions. The process is being tested at a semi-commercial scale at Novara. The product has been named HIDEV AN1 alumina. Most important properties for HIDEV AN1 alumina have been summarized in Table III. Figure 16 shows an SEM photomicrograph of HIDEV AN1 alumina. Notice the spherical morphology of the particles and the uniformity of the particle size.

In addition to being spherical, this type of alumina is produced with a very narrow size distribution. As shown in Table III these are high purity aluminas. After sintering, they produce a regular microstructure with controlled grain size. Figure 17 shows a SEM photomicrograph of the grain structure obtained for sprill fire at standard conditions (1550°C, 2 hrs). No additives were used to control grain structure. Fully dense bodies have also been obtained at a temperature of 1500°C.

HIDEV AN1 can be sintered at to more than 98% of the theoretical density without the addition od a grain growth inhibitor producing very uniform microstructure with average grain size of about 3 micron. This type of microstructure leads to high mechanical properties. For example, flexural strength of 480 MPa (four point bending test) has been achieved on a uniaxially pressed compact. When forming through isopressing was used, the flexural strength increased to 550 MPa. These excellent mechanical properties are better than those obtained for other high purity aluminas available on the market.

HIDEV AN1 alumina also exhibits highly smooth surface and no aggregates making it very appropriate for the production of slips to be used in the casting of tape with highly smooth surface. Production of thin substrate for electronics and other applications for bioprotesis and structural composites also benefit from these properties.

This alumina could also be used for magnetic media formulations intended for the production of high quality rigid disks where

tight tolerance are imposed on media surface.

The Keramont process to produce HIDEV AN1 also allows for inclusion of different elements as dopants that will be uniformly distributed in the alumina matrix. Using this approach Keramont has developed alumina/zirconia submicron powder of spherical shape and with narrow size distribution. Available zirconia content ranges between 4% and 15%. The main advantage of this powder is the high degree of chemical uniformity making it a prime candidate for the production of alumina/zirconia composites.

Differences between particle morphology, surface rugosity, and purity, makes KeraMag and HIDEV AN1 two products of very different nature to meet different application objectives. However, the production of high purity precipitated alumina requires more processing steps than those required for mechanical fragmentation causing a significant difference in their cost structure. The applicability of either one of these products will have to be matched to requirements by the end user.

CONCLUSIONS.

Results obtained in this study demonstrate the feasibility of producing ultrafine alumina powders through the use of mechanical fragmentation. Product with median size as fine as 0.23 microns and top size of one micron were produced using conventional milling techniques. These aluminas are intended to be used for magnetic media, as abrasives, and other ceramic applications. The technology developed was able to produce ultrafine alumina powders of high purity at a reduced cost.

Modelling of grinding operations investigated showed that an empirical energy-size reduction relationship is appropriate to correlate product median size with grinding time. A more sophisticated modelling approach based on the macroscopic population balance model for first order grinding kinetics was successfully applied to the first 8-10 hours of grinding. For longer retention time linearity was lost and model predictions deviate from experimental data.

Spherical aluminas developed by Keramont exhibited higher purity level and could be produced at different sizes with a very narrow size distribution and very smooth surface. Sintered bodies made with these aluminas showed superior micro-structure with uniform grain size.

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TABLE I

TYPICAL ALUMINA SIZE REQUIREMENTS
FOR MAGNETIC MEDIA APPLICATIONS

Top Size (microns)	Median Size (microns)	Coating Thickness (microinches)	Applications
3.0	0.7 - 0.5	200 or more	- Video tape - Audio tape - Computer tape
2.0	0.5 - 0.4	80 - 200	- Flexible disks - Video tape - Audio tape - Computer tape
1.5	0.4 - 0.3	40 - 80	- Rigid disks - High density flexible disks
1.0	0.3 - 0.2	40 or less	- Rigid disks - High density flexible disks

TABLE II

KERAMAG ALUMINA SPECIFICATIONS

Keramont Corporation Micronized Aluminas

Size Distribution and Specific Surface Area

	(a) Top Size	(a) d 70	(a) d 50	(a) d 30	(b) Area
	(m i c r o n s)				(sq.m /g)
KeraMag-A	3.0	1.0	0.55	0.35	9.5
KeraMag-B	3.0	0.8	0.44	0.30	13.4
KeraMag-F	2.0	0.45	0.35	0.22	15.0
KeraMag-SF	1.0	0.35	0.25	0.18	20.9

Green and Fired Density

	(c) Green Density (g/cc)	(d) Fired Density (g/cc)	Linear Shrinkage (%)
KeraMag-B	2.06	3.86	18.29
KeraMag-SF	1.90	3.99	19.94

Other Physical Properties

Phase : alpha-alumina
Specific Gravity : 3.82 g/cc
Purity : 99.74% alumina
Loss on Ignition : 0.5% (at 1000°C)

TYPICAL CHEMICAL ANALYSIS (Wt %) (e)

Na2O	SiO2	Fe2O3	MgO	CaO
0.0038	0.0265	0.0031	0.0013	0.0027

(a) Light Absorption Sedimentometer
(b) Single point BET
(c) Tested on 10 gram pellets, one inch diameter at 5000 psi.
(d) Fired at 1500°C for one hour.
(e) Calculated from ICP analyses

TABLE III

HIDEV AN1 ALUMINA SPECIFICATIONS

Keramont Corporation Spherical Alumina

PARTICLE SIZE [a]

Top Size = 1.0 - 2.0 μm

Median Size = 0.3 - 0.6 μm

PROPERTIES		CHEMICAL ANALYSIS [b]	
Purity	>99.96%	Na	30 ppm
Crystal Phase	α-alumina	Si	80 ppm
Loss on Ignition (1000°C)	0.3%	Fe	35 ppm
Medium Cristallite Size	1300-1400 A	Mg	5 ppm
		Ca	60 ppm
Density	>3.8 g/cc	K	10 ppm
Morphology	spherical	Ti	20 ppm
Surface Area	4 - 6 m^2/g	Zr	10 ppm

GREEN AND FIRED DENSITY

Green Density = 1.9 g/cc [c]

Fired Density = 3.9 g/cc [d]

(a) Light Absorption Centrifugal Sedimentometer
(b) ICP
(c) Die pressing at 900 kg/sq.cm
(d) Fired at 1550°C for two hours

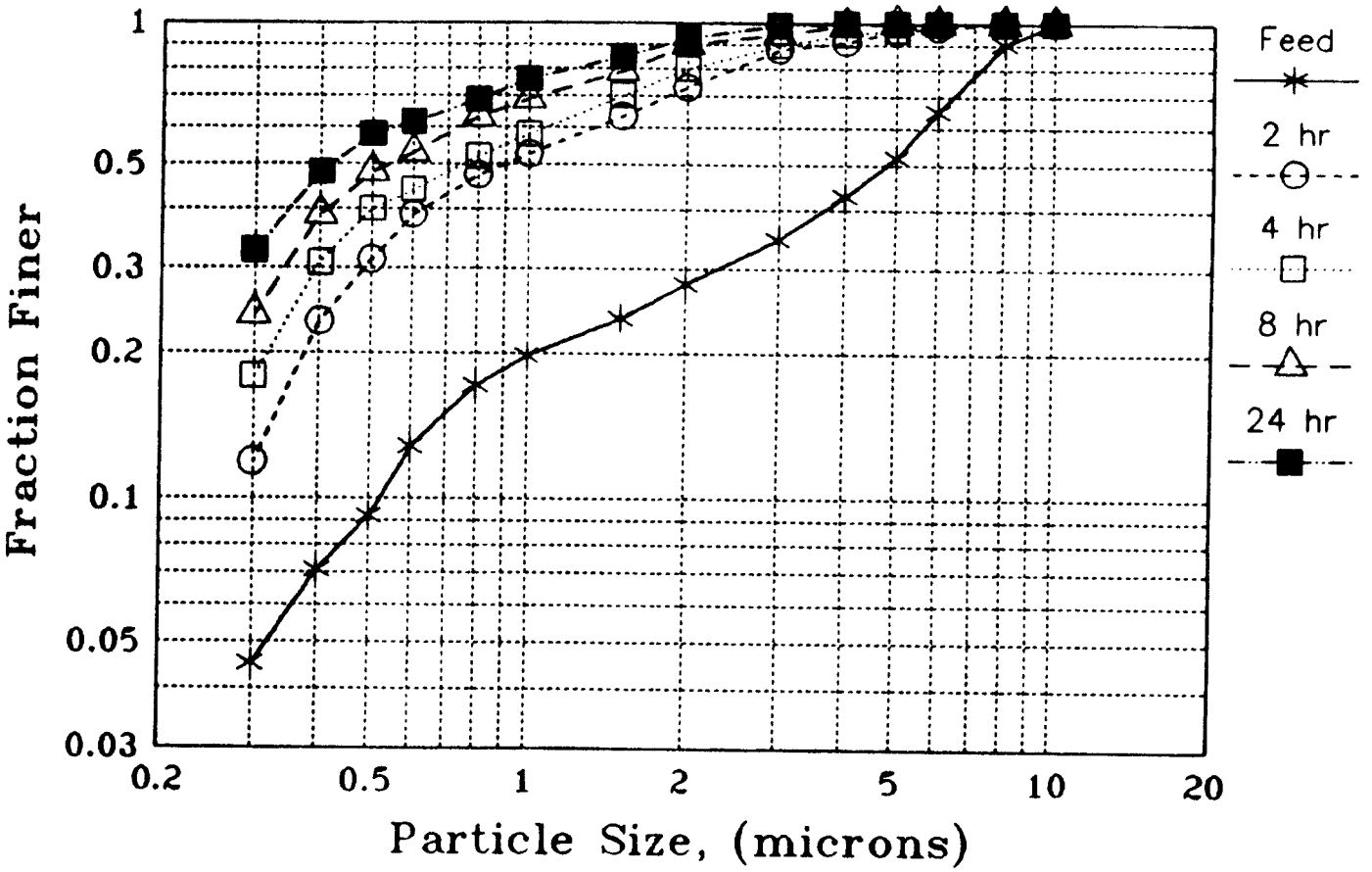

Figure 1. Product size distributions after grinding alumina in a 52 gal. Norton ball mill on a dry mode at 100% voids filling. (MMA-886-10)

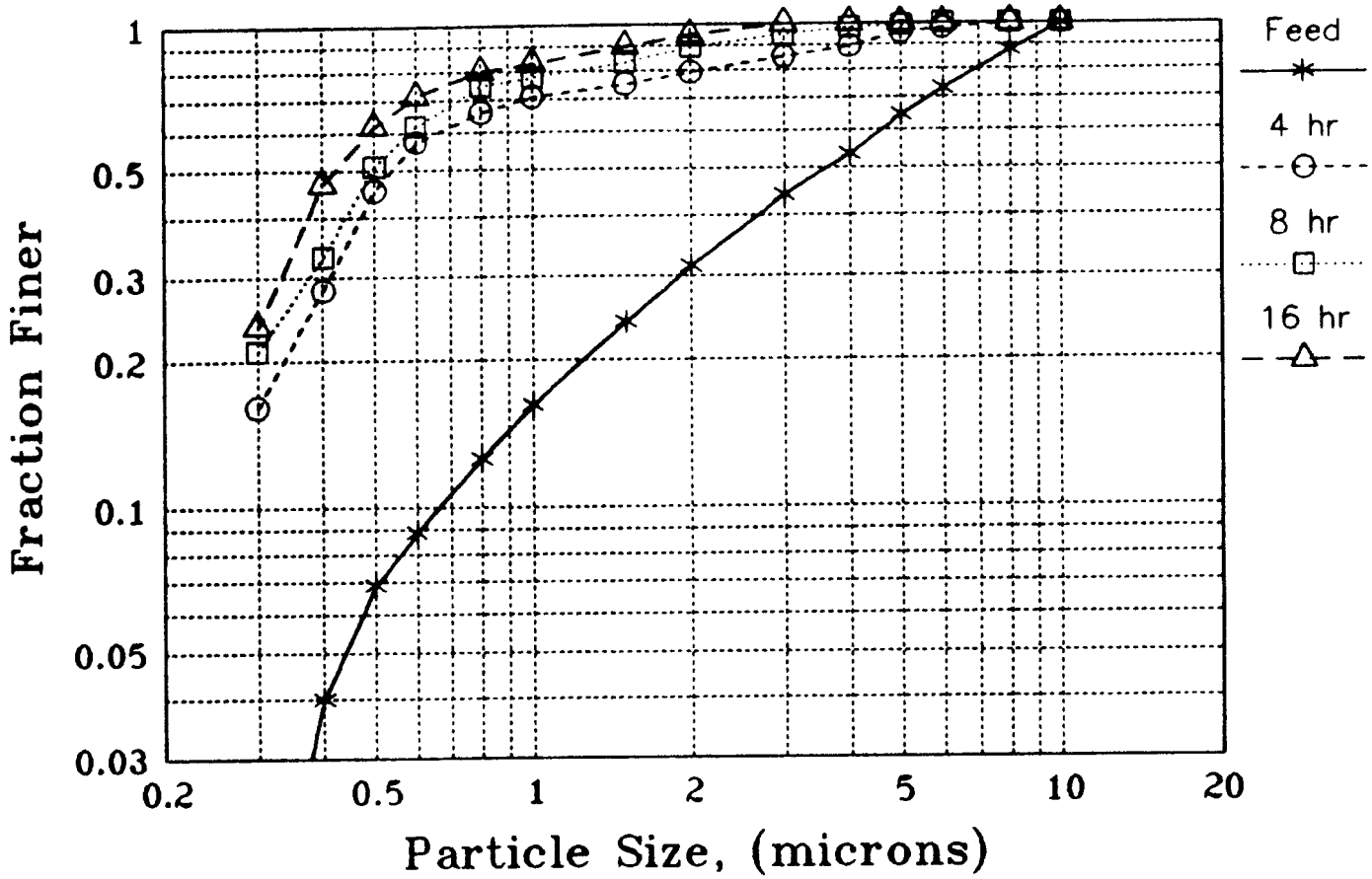

Figure 2. Product size distributions after grinding alumina in a 52 gal. Norton ball mill on a dry mode at 70% voids filling. (MMA-1086-4)

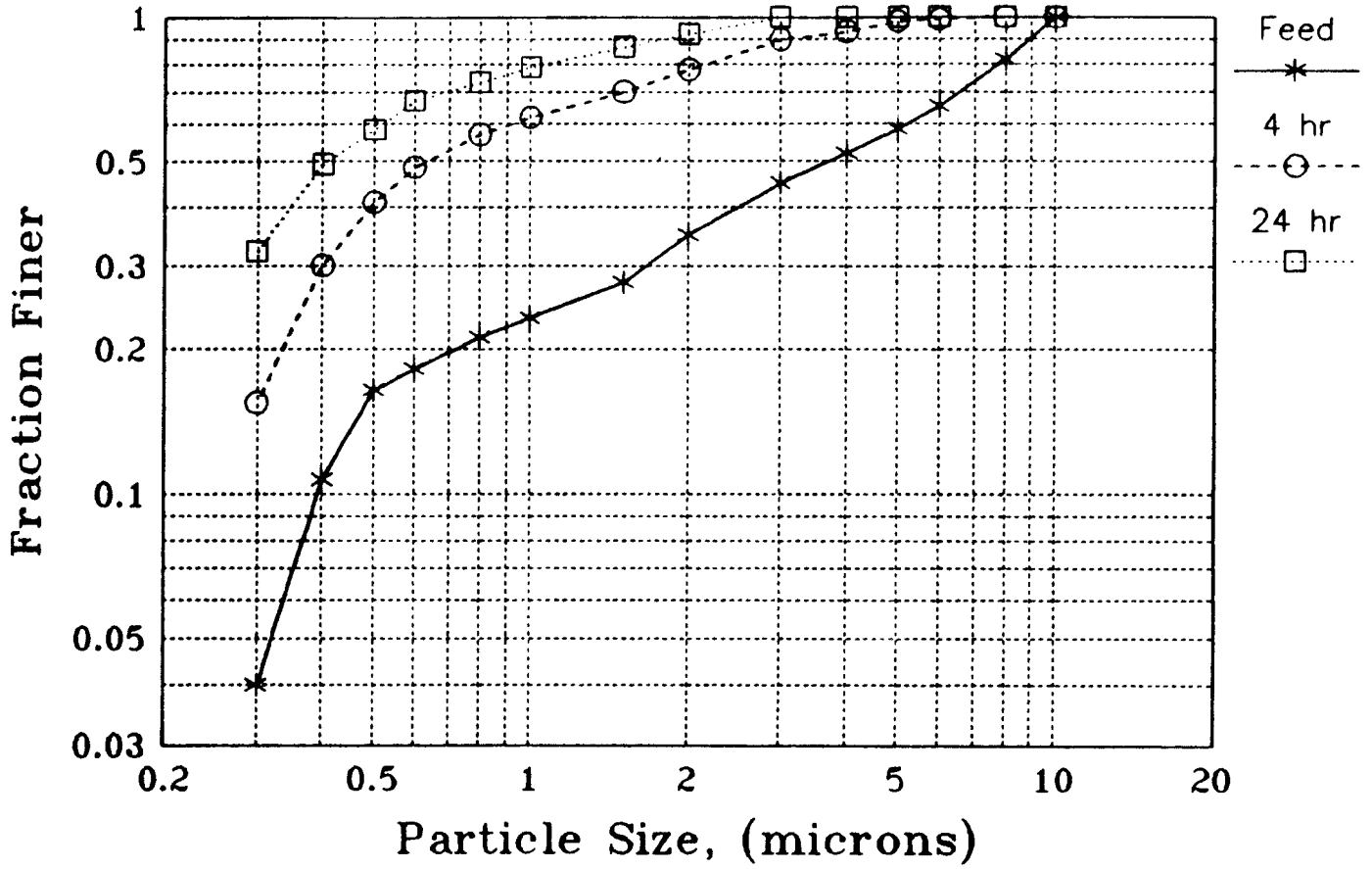

Figure 3. Product size distributions after grinding a sample of calcined Alcoa-C33 alumina in a 1.5 gallon ceramic ball mill. (MMA-1286-1)

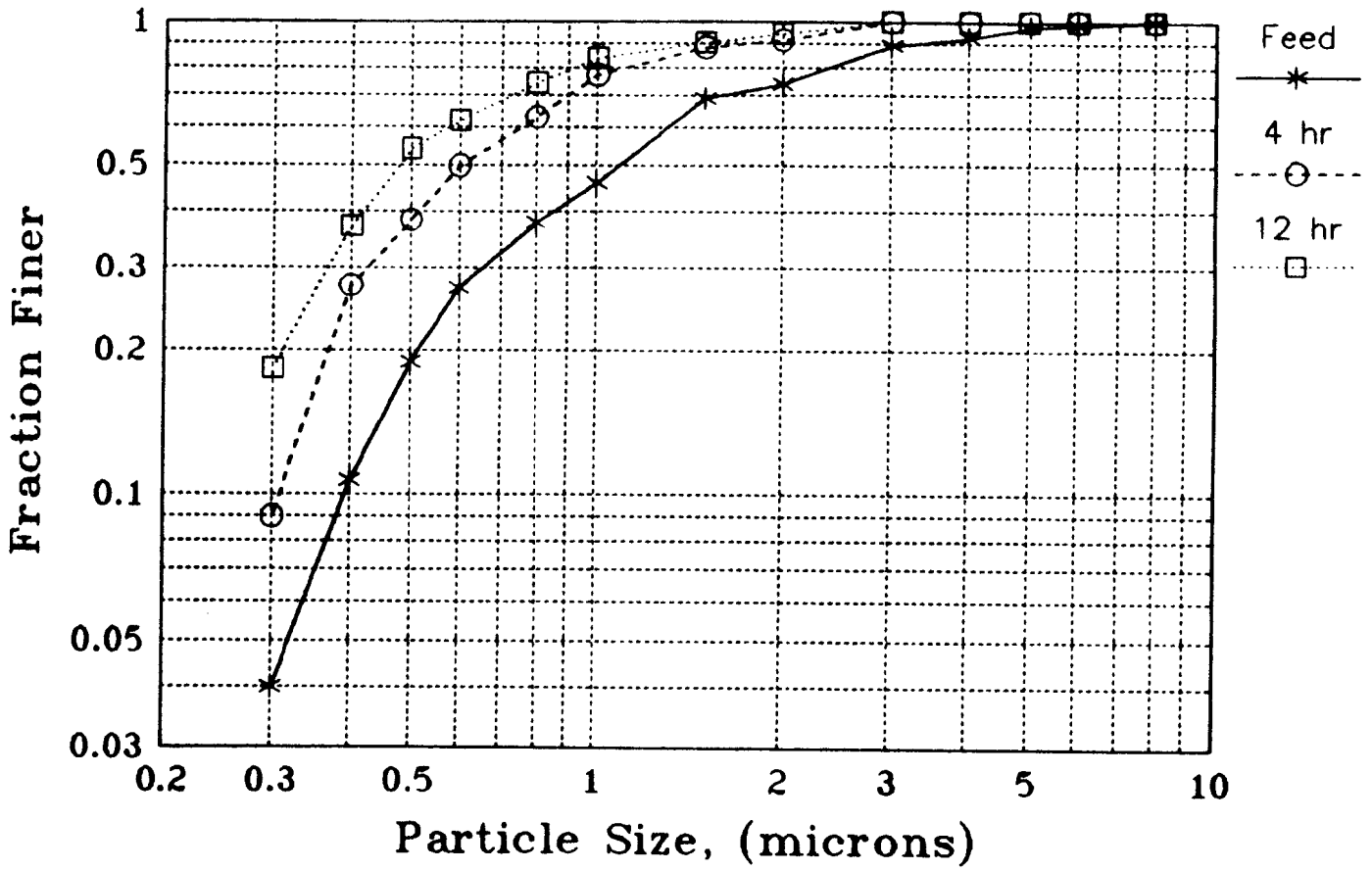

Figure 4. Product size distributions after grinding a sample of calcined Ceralox alumina in a 1.5 gallon ceramic ball mill. (MMA-1186-3)

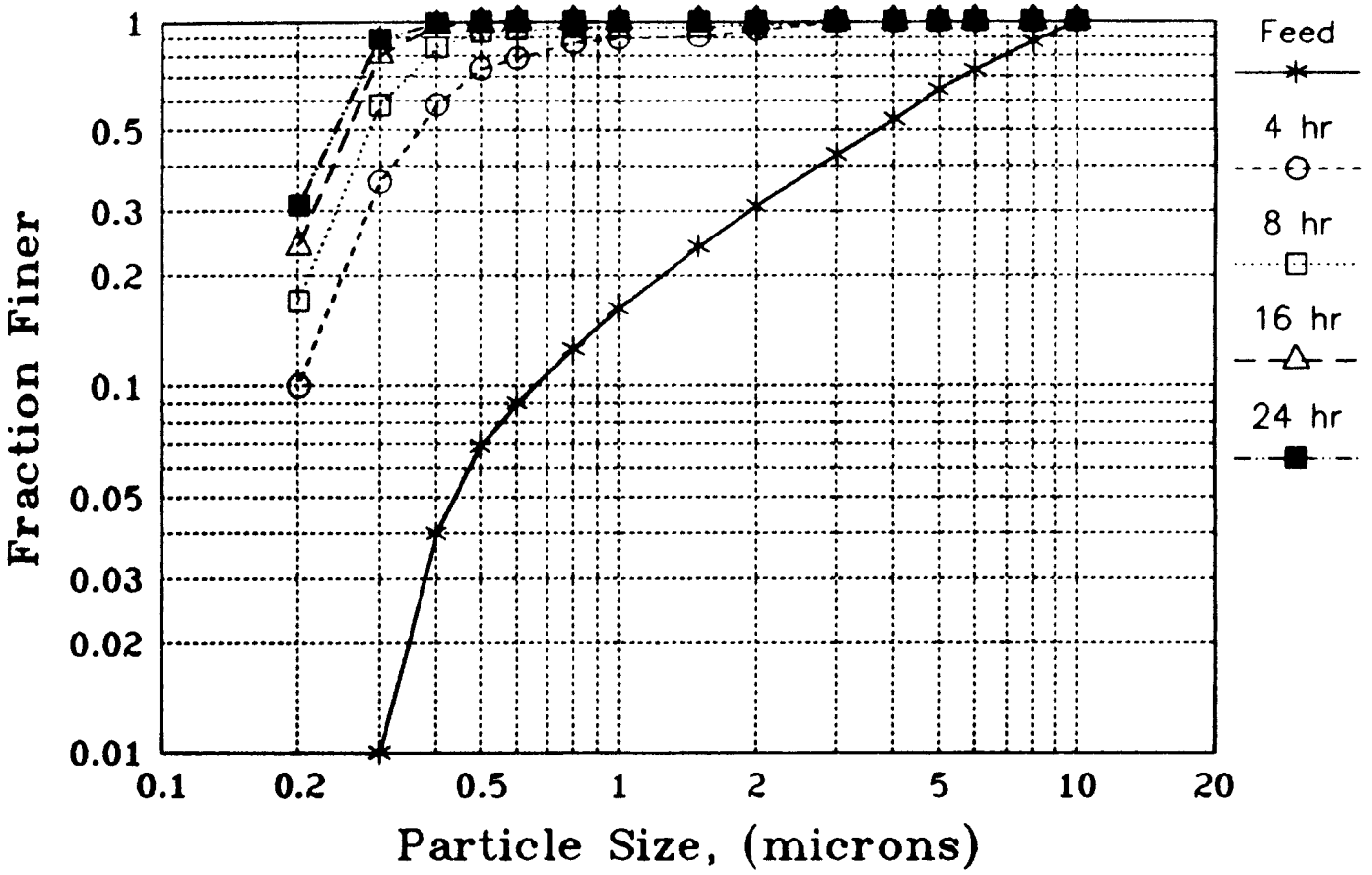

Figure 5. Product size distributions after grinding alumina in a 1.5 gal. ceramic ball mill on a wet mode with zirconia media. (MMA-1086-3)

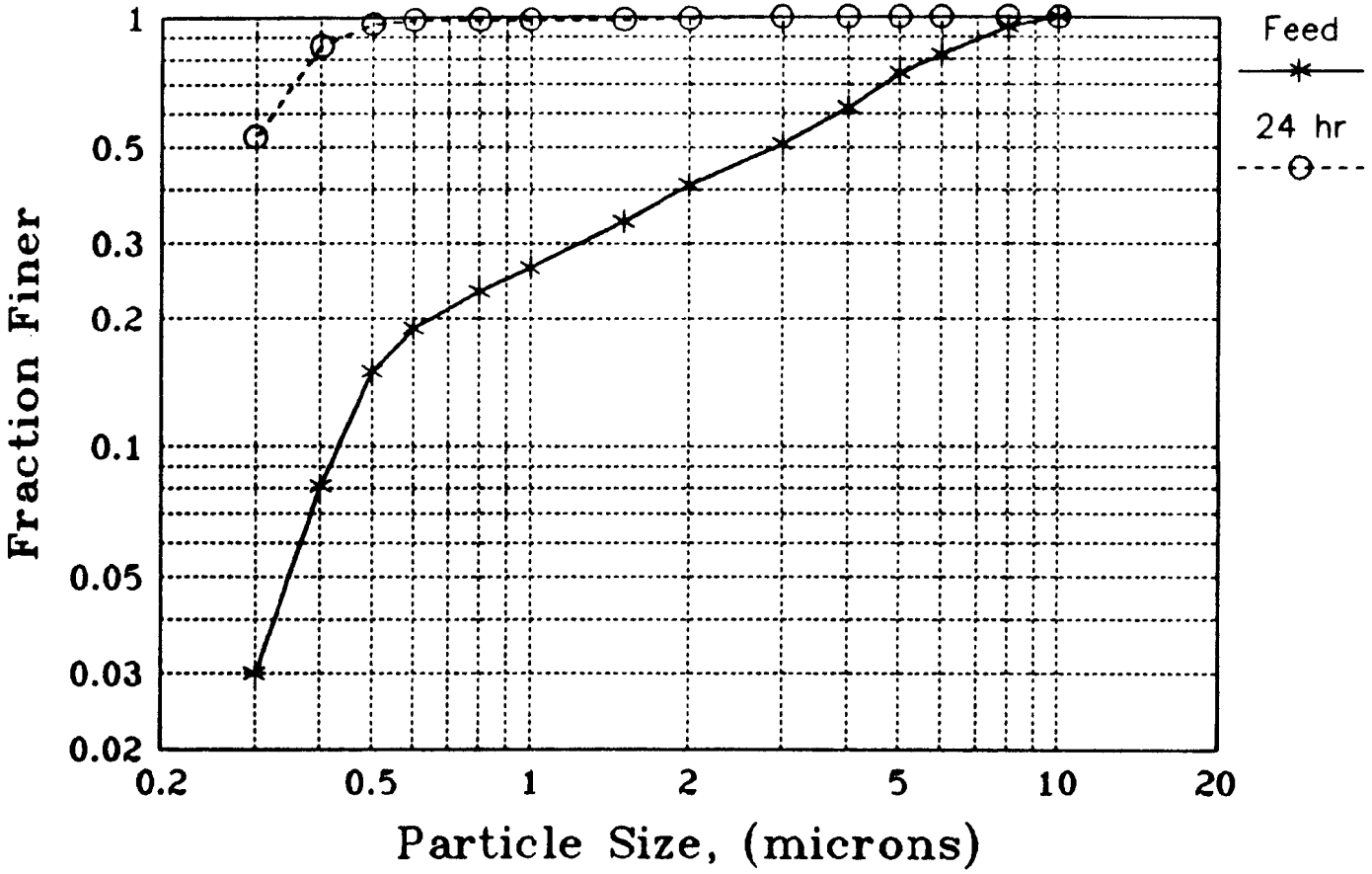

Figure 6. Product size distributions after grinding alumina in a 6.6 gal. ceramic ball mill on a wet mode with alumina media. (MMA-687-7)

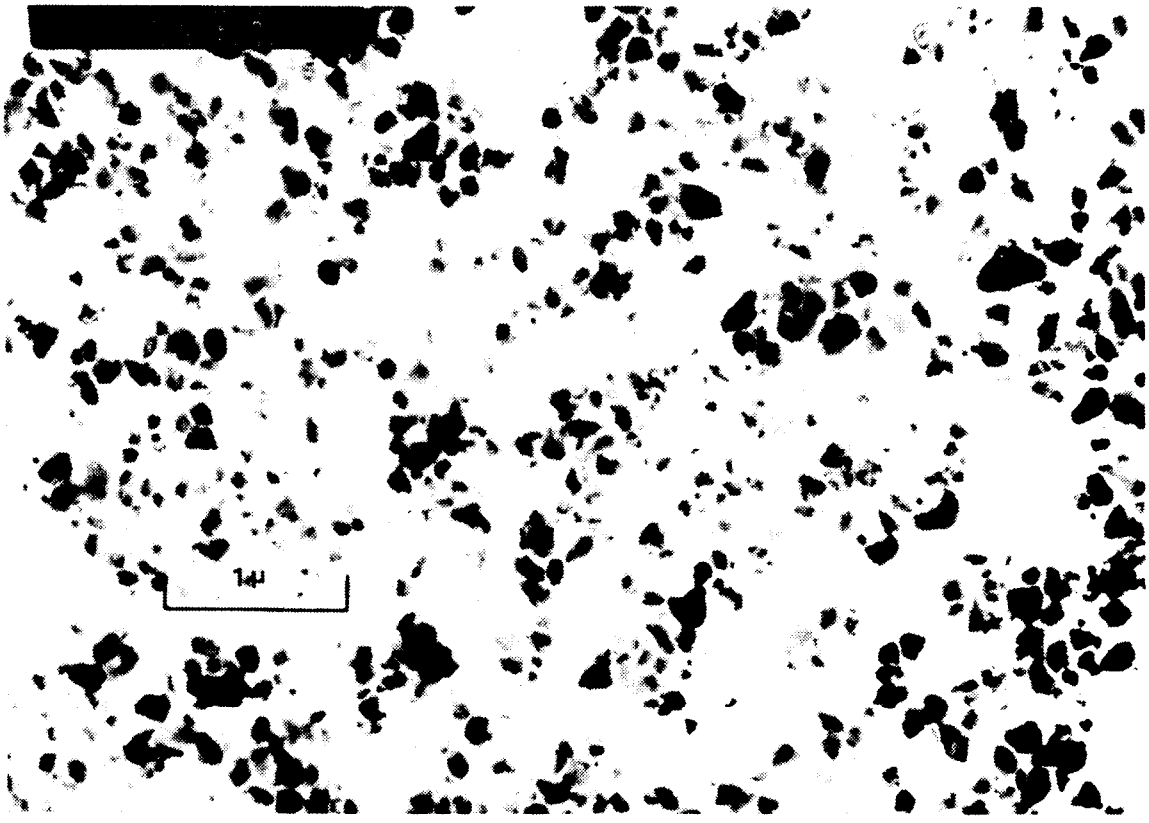

Figure 7. TEM photograph of submicron alumina produced after wet milling.

Figure 8. SEM photomicrograph of a fracture surface of a pellet made with submicron alumina sintered at 1550 C for two hours.

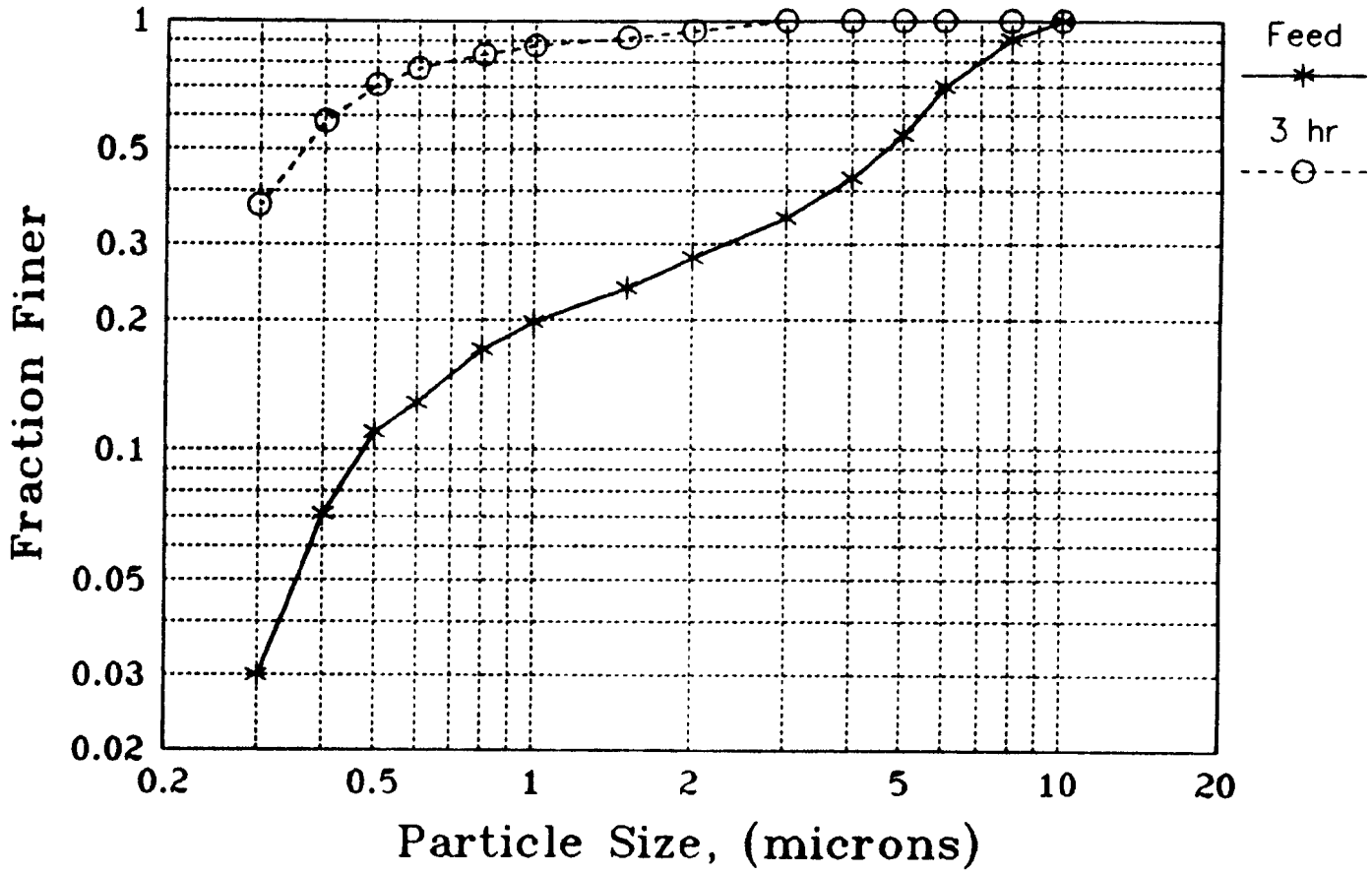

Figure 9. Product size distributions after grinding alumina in a 1.5 gal. Attritor mill with steel media. (MMA-786-31)

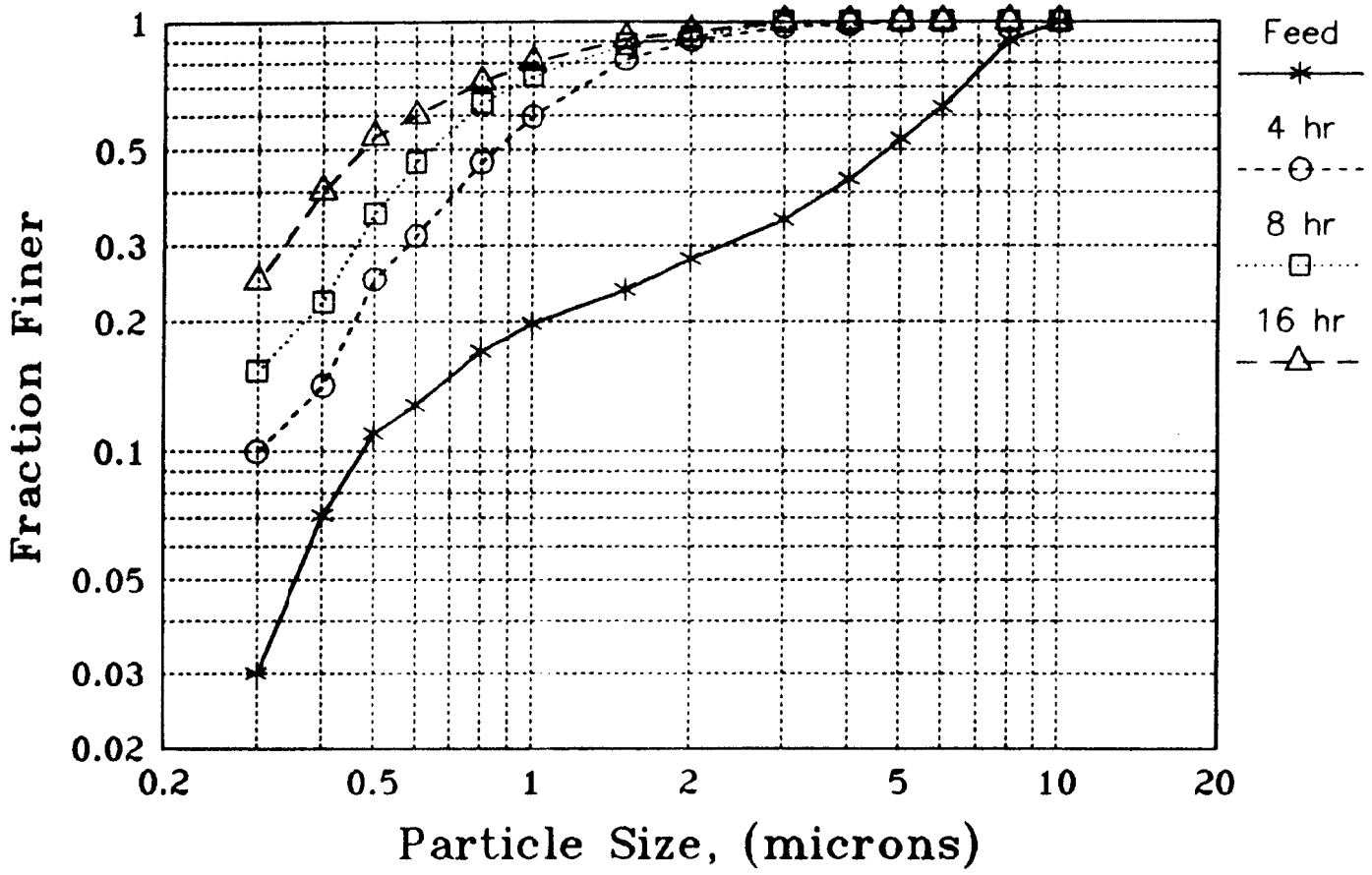

Figure 10. Product size distributions after grinding alumina in a 1.5 gal. Attritor mill with alumina media. (MMA-786-1)

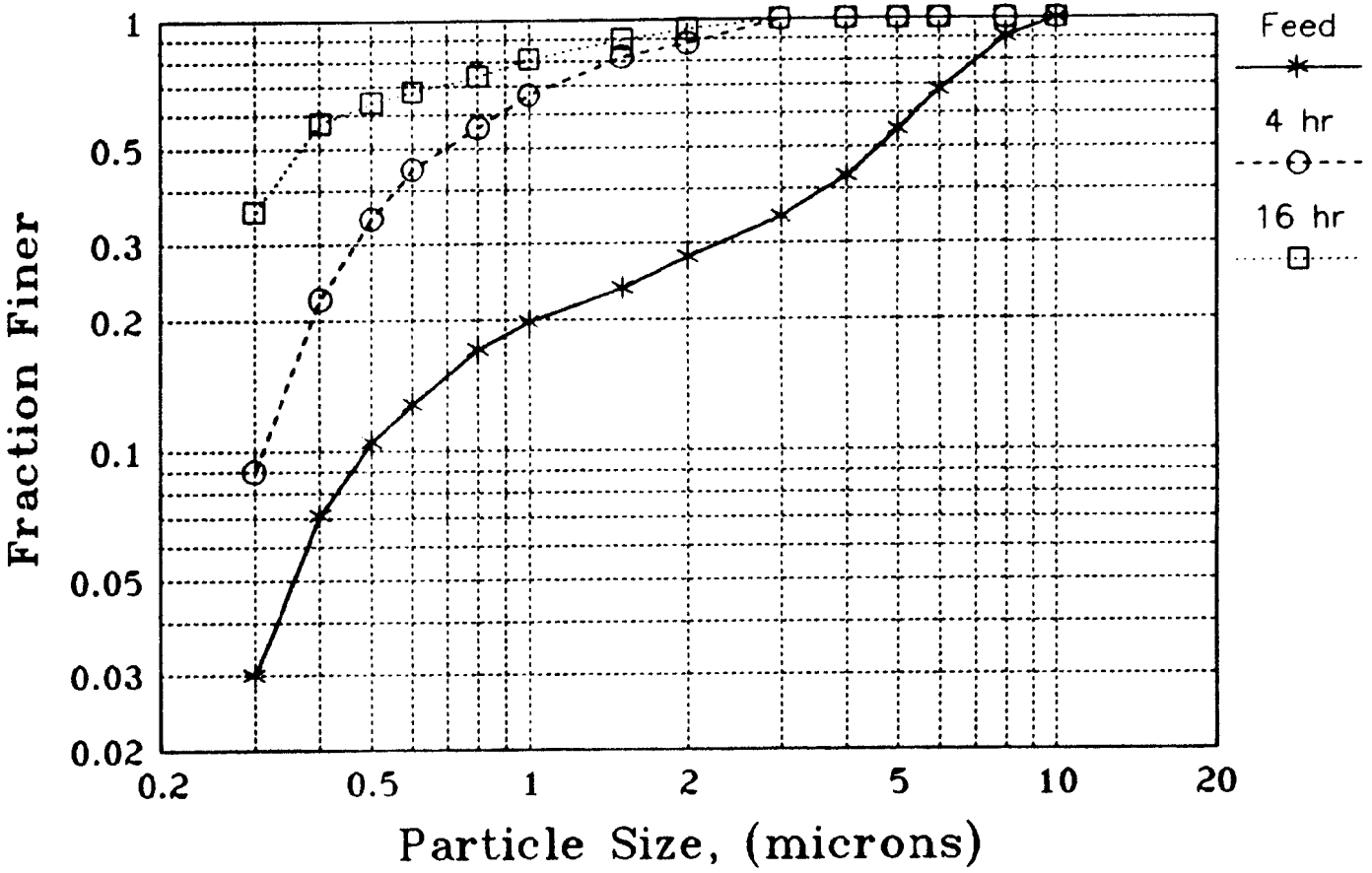

Figure 11. Product size distributions after grinding alumina in a 1.5 gal. Attritor mill with zirconia media. (MMA-786-2)

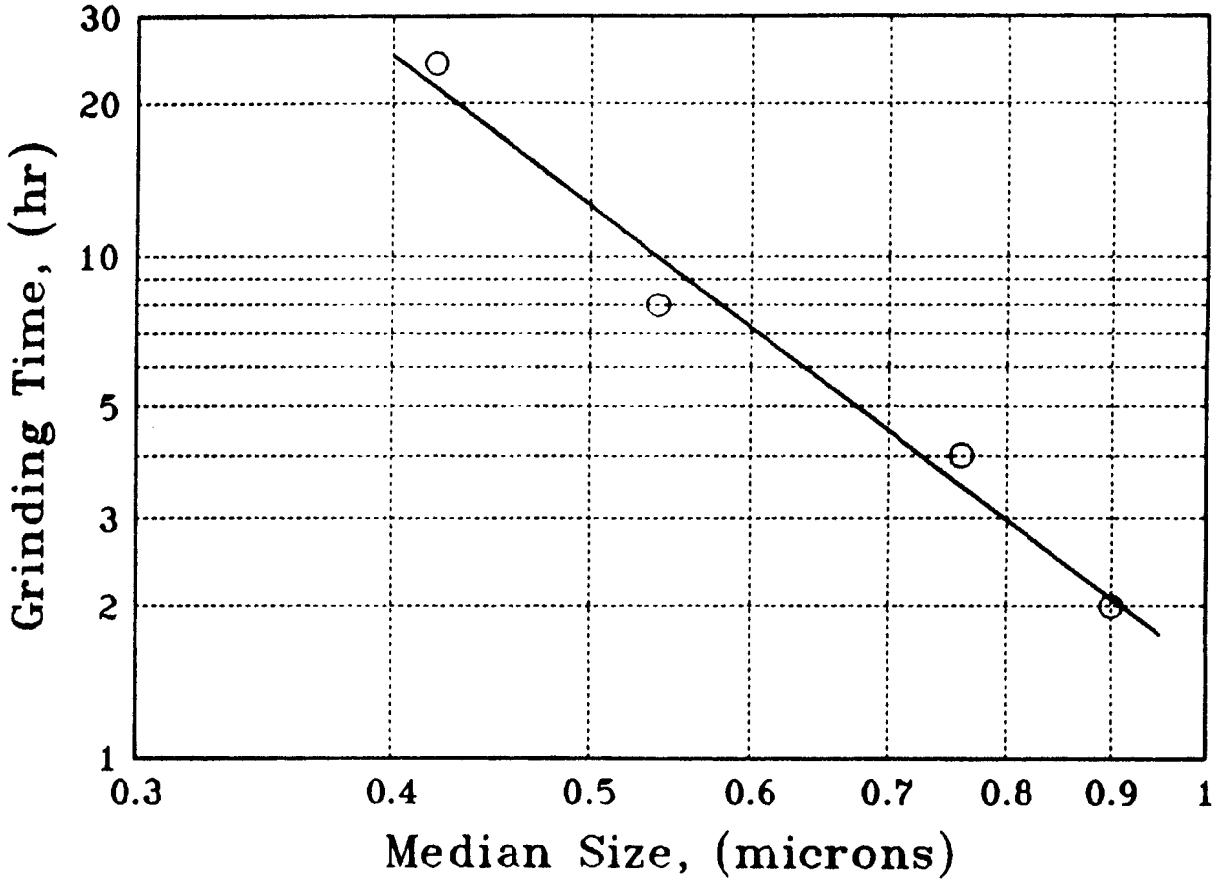

Figure 12. Grinding time-size reduction relationship for the dry ball milling of alumina in a 52 gal. Norton mill at 100% voids filling. (MMA-886-10)

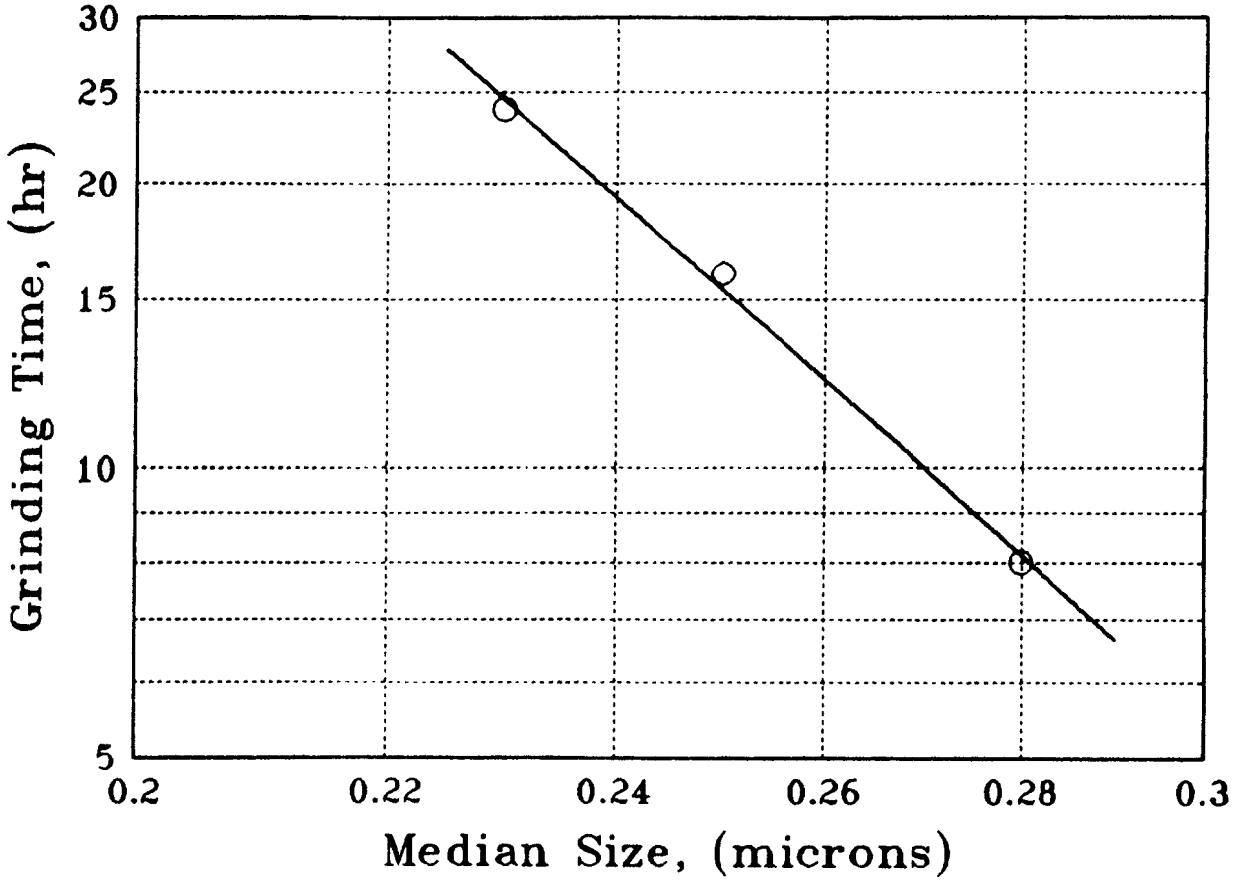

Figure 13. Grinding time-size reduction relationship for the wet ball milling of alumina in a 1.5 gal. ceramic mill with zirconia media. (MMA-1086-3)

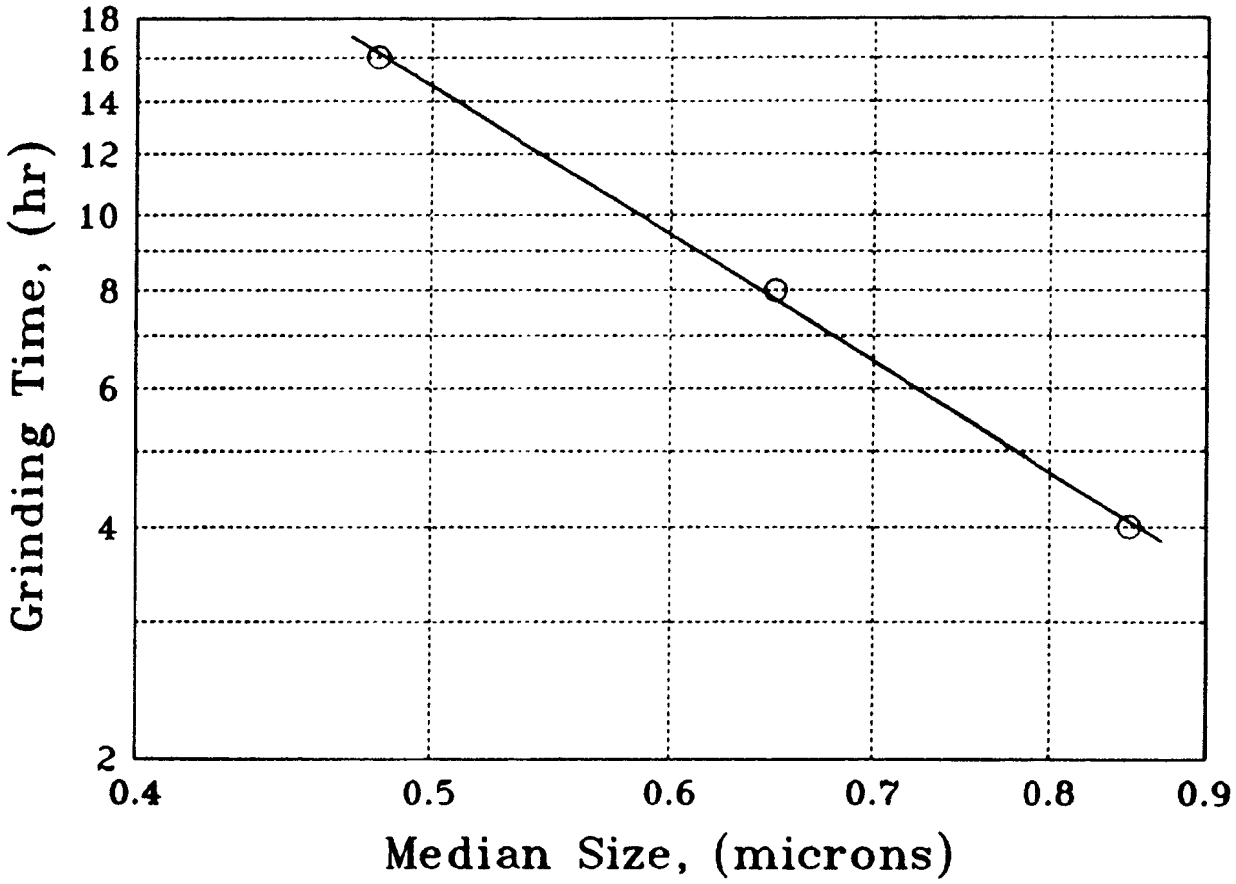

Figure 14. Grinding time-size reduction relationship for the stirred ball milling of alumina in a 1.5 gal. Attritor mill charged with alumina media. (MMA-786-1)

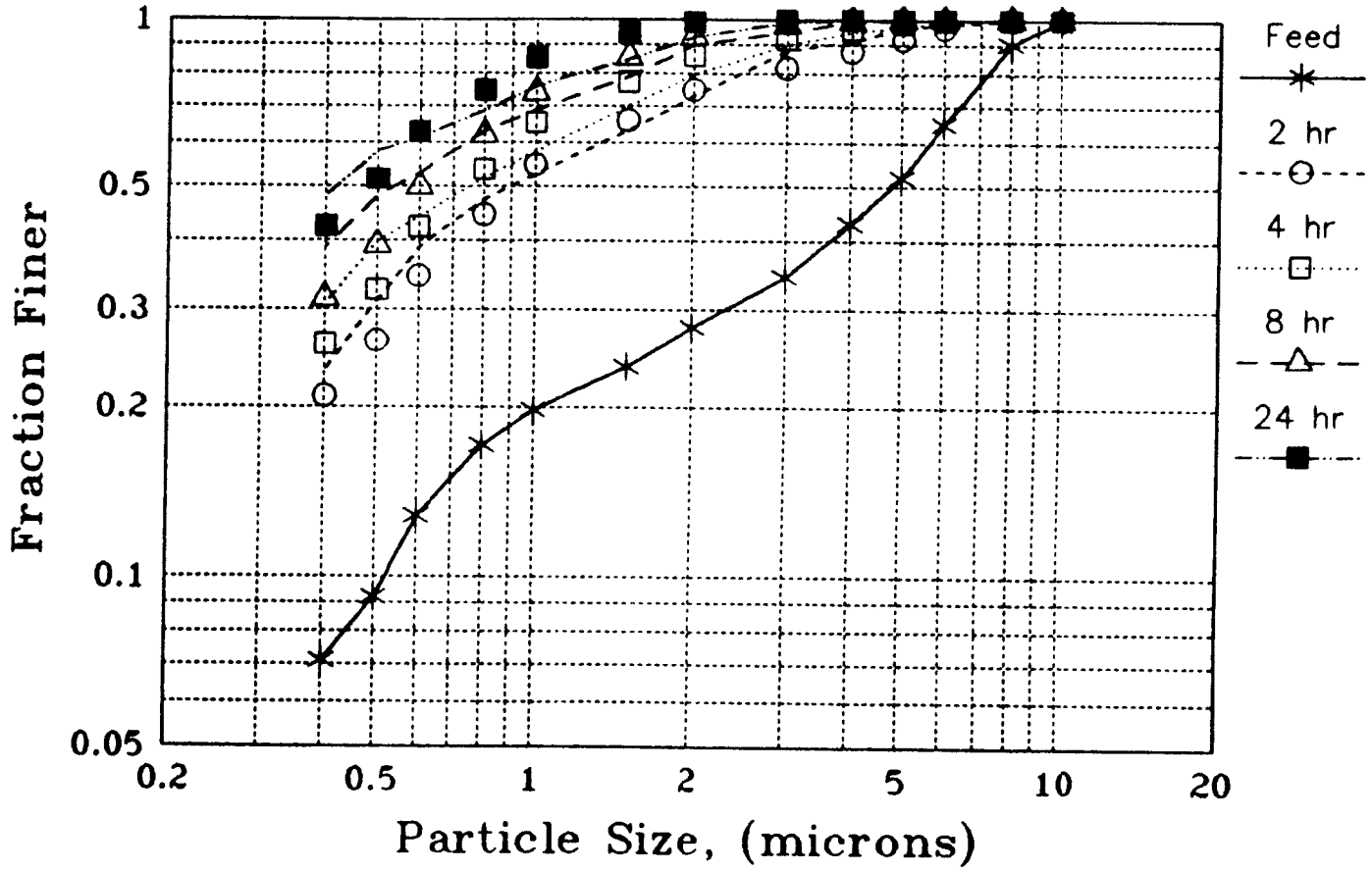

Figure 15. First order grinding kinetics model fit for the data obtained after grinding alumina in a 52 gal. Norton ball mill on a dry mode at 100% voids filling. (MMA-886-10)

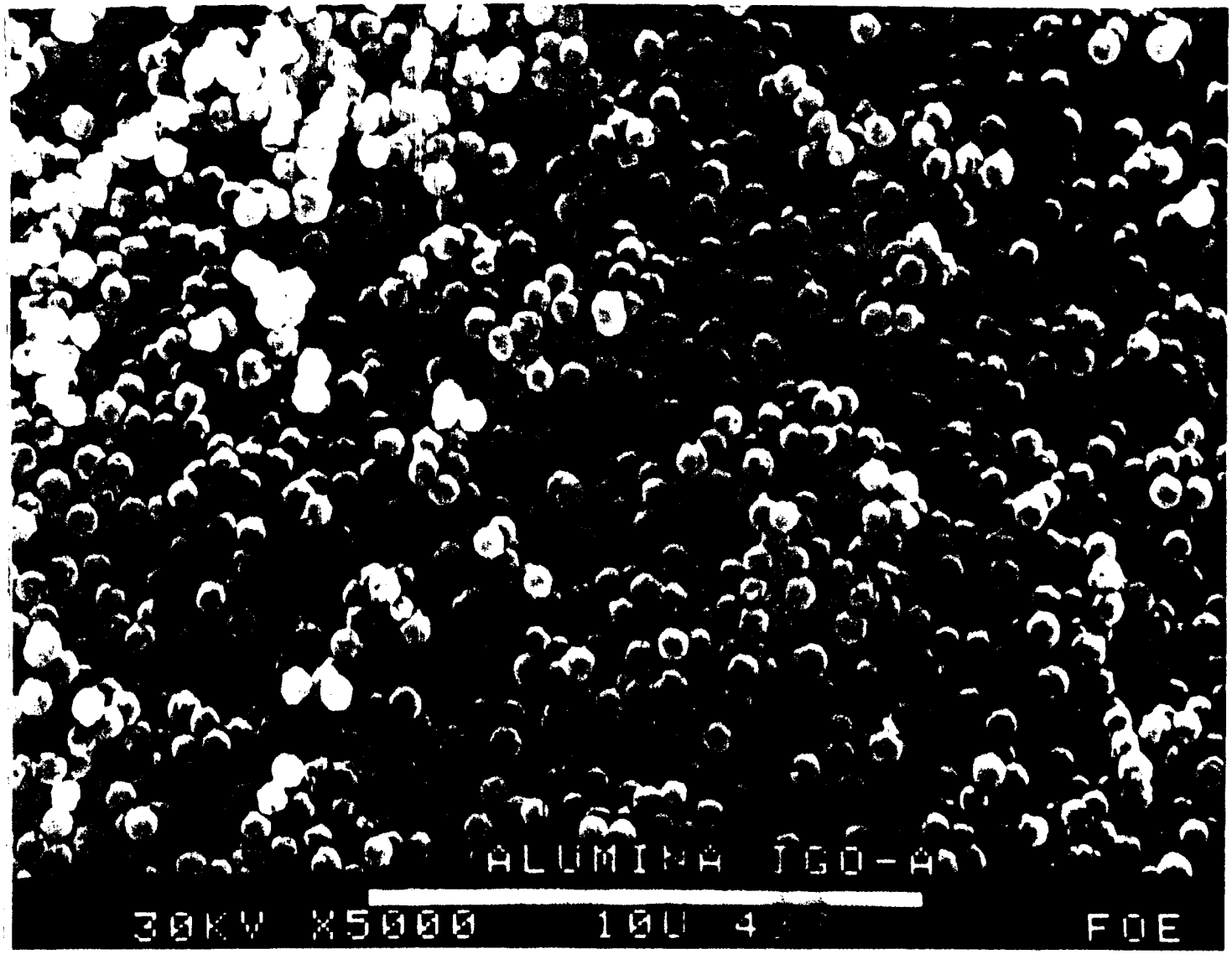

Figure 16. SEM photomicrograph of HIDEV AN1 alumina.

Figure 17. SEM photomicrograph of a fracture surface of a pellet made with HIDEV AN1 alumina sintered at 1550 C for two hours.