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DOE Research and Development Report

The Characterization of Commercial Thorium Oxide Powders

(AWBA Development Program)

T. J. Burke

Bettis Atomic Power Laboratory
West Mifflin, Pennsylvania 15122-0079

May 1982

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(AWBA Development Program)

T. J. Burke

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FOREWORD

The Shippingport Atomic Power Station located in Shippingport, Pennsylvania was the first large-scale, central-station nuclear power plant in the United States and the first plant of such size in the world operated solely to produce electric power. This program was started in 1953 to confirm the practical application of nuclear power for large-scale electric power generation. It has provided much of the technology being used for design and operation of the commercial, central-station nuclear power plants now in use.

Subsequent to development and successful operation of the Pressurized Water Reactor in the Atomic Energy Commission (now Department of Energy, DOE) owned reactor plant at the Shippingport Atomic Power Station, the Atomic Energy Commission in 1965 undertook a research and development program to design and build a Light Water Breeder Reactor core for operation in the Shippingport Station.

The objective of the Light Water Breeder Reactor (LWBR) program has been to develop a technology that would significantly improve the utilization of the nation's nuclear fuel resources employing the well-established water reactor technology. To achieve this objective, work has been directed toward analysis, design, component tests, and fabrication of a water-cooled, thorium oxide-uranium oxide fuel cycle breeder reactor for installation and operation at the Shippingport Station. The LWBR core started operation in the Shippingport Station in the Fall of 1977 and will finish routine power operation on October 1, 1982. After End-of-Life core testing, the core will be removed and the spent fuel shipped to the Naval Reactors Expended Core Facility for detailed examination to verify core performance including an evaluation of breeding characteristics.

In 1976, with fabrication of the Shippingport LWBR core nearing completion, the Energy Research and Development Administration, now DOE, established the Advanced Water Breeder Applications (AWBA) program to develop and disseminate technical information which would assist U. S. industry in evaluating the LWBR concept for commercial-scale applications. The AWBA program, which is concluding in September, 1982, has explored some of the problems that would be faced by industry in adopting technology confirmed in the LWBR program. Information already developed includes concepts for commercial-scale prebreeder cores which would produce uranium-233 for light water breeder cores while producing electric power, improvements for breeder cores based on the technology developed to fabricate and operate the Shippingport LWBR core, and other information and technology to aid in evaluating commercial-scale application of the LWBR concept.

All three development programs (Pressurized Water Reactor, Light Water Breeder Reactor, and Advanced Water Breeder Applications) have been conducted under the technical direction of the Office of the Deputy Assistant Secretary for Naval Reactors of DOE.

Technical information developed under the Shippingport, LWBR, and AWBA programs has been and will continue to be published in technical memoranda, one of which is this present report.

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The chemical and physical characteristics of three thoria powders obtained from commercial vendors were compared with Light Water Breeder Reactor (LWBR) powder specification requirements and a representative LWBR thoria powder lot to determine the suitability of currently available thoria powders for the manufacture of high density breeder reactor fuel. Two of the three vendor powders showed fabrication characteristics essentially equivalent to the LWBR thoria powder lot and could be used to fabricate high density, crack-free LWBR blanket-type thoria pellets using the modified LWBR/AWBA micronize-press-sinter fabrication route. The inability to fabricate acceptable pellets with the third vendor powder was attributed to the high as-received surface area of the powder. The study also showed that process simplification by elimination of the powder micronizing operation was not feasible for the powders evaluated.

THE CHARACTERIZATION OF COMMERCIAL THORIUM OXIDE POWDERS

(AWBA Development Program)

T. J. Burke

I. INTRODUCTION

A. Purpose

The development and manufacture of the thoria and thoria-urania fuel pellets for the Light Water Breeder Reactor (LWBR) highlighted the need for compatibility between the powder properties and the pellet fabrication process (Reference 1). In order to provide a high purity, reproducible thoria powder with the required characteristics for the production of high density fuel pellets for the LWBR program, production of the thoria powder was limited to a single source and all phases of the thorium oxalate conversion process used to produce the powder were carefully controlled.

Adaptation of the LWBR technology for commercial scale application will entail the development of additional sources for supply of reactor grade thoria powder with characteristics equivalent to the LWBR feed powder. The powder evaluation study described in this report was designed to determine the physical

and chemical characteristics of a sampling of thoria powders currently available from commercial vendors, to assess the response of these powders to the LWBR/AWBA pellet fabrication process, and to determine if these powders offer fabrication advantages with a potential for process simplification by elimination of micronizing. The chemical and physical characteristics of the vendor powders are compared with the LWBR powder specification requirements and the characteristics of a typical lot of LWBR thoria, and the density and microstructure of sinterability test pellets are compared with LWBR pellet requirements.

B. Background

The thoria powder used for the LWBR core was manufactured by National Lead of Ohio (NLO) via a thorium oxalate precipitation process (Reference 2). Oxalic acid, $H_2C_2O_4$, solution was sprayed into purified thorium nitrate, $Th(NO_3)_4$, solution under controlled conditions (free nitric acid, precipitation temperature, and precipitation rate) to precipitate thorium oxalate, $Th(C_2O_4)_2 \cdot 6\ H_2O$, according to the following equation:

$$Th(NO_3)_4 + 2\ H_2C_2O_4 \rightarrow Th(C_2O_4)_2 \cdot 6\ H_2O \downarrow + 4\ HNO_3$$

After filtration, washing, and air drying, the precipitated thorium oxalate was decomposed to ThO_2 by air calcination.

Modifications in the parameters for the precipitation and calcination processing steps control the characteristics and sinterability of the powder. A wide range of ThO_2 powders, each with its own characteristics and sinterability properties, can be prepared by adjustment of these parameters (References 4, 5, 9-12).

For example, as the calcining temperature increases above 400°C, the crystallites within the ThO_2 powder particles grow and the particles themselves shrink as discussed by Kinoshitn, et al (Reference 3). Electron microscope studies show that the as-calcined powder consists of square or rectangular, plate-like particles similar to the precursor oxalate (References 3-6). These square plate-like particles are porous and consist of aggregates of crystallites. Upon calcining at higher temperatures or for longer times, the internal porosity gradually sinters out as the ThO_2 grains grow within each particle decreasing the powder surface area (References 4, 5, 6-8).

C. Test Program

Four kilogram batches of thoria powder were purchased from each of three commercial vendors for direct comparison with LWBR* National Lead of Ohio powder Lot Number 158 as a control. This powder lot was used for the fabrication of irradiation test and fuel development pellets for the Advanced Water Breeder Applications (AWBA) programs. The impurity content, powder morphology, surface area, and particle size distribution were measured on samples of each of the vendor powders in the "as-received" condition, and then these tests, except for impurity content, were repeated on powder samples taken after one-pass and two-pass micronization. Sinterability test pellet series were prepared with each of the thoria powders in the as-received and micronized conditions to compare the fabrication behavior of the vendor powders with the standard LWBR powder lot.

II. THORIA POWDER BATCH MICRONIZING AND SAMPLING PROCEDURE

Each of the powder batches from the three commercial vendors and LWBR NLO powder Lot 158 was sampled for powder characterization tests and pellet sinterability test fabrication according to the flow diagram shown in Figure 1. In this sampling procedure, each test batch (four kilogram) was first weighed and then mixed in a 4-quart V-type blender for 10 minutes. After blending, grab samples, considered representative of the powder in the as-received condition, were withdrawn for chemical and surface area analysis and for characterization of powder particle size and shape. In addition, a one kilogram portion of the as-received material was taken for fabrication of the pellet sinterability test series. The remainder of the batch was then single-pass micronized using a powder feed rate of 150 grams per minute, dry blended for three minutes in the V-blender, and then sampled to provide characterization samples and powder for pellet fabrication representative of the one-pass micronized condition. The balance of the powder batch was then micronized for a second time at a feed rate of 50 grams per minute, re-mixed in the V-blender for three minutes, and then sampled, as before, to provide characterization

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*National Lead Company, Technical Division, Box 39158, Cincinnati, Ohio, 45239, under contract to the Bettis Atomic Power Laboratory manufactured the ThO_2 powder for the LWBR Program.

samples. Approximately two kilograms of two-pass micronized powder remained in the batch for fabrication of sinterability test pellet series representative of the two-pass micronized condition.

III. THORIA POWDER CHARACTERIZATION

A. Chemical Analysis (Reference 13)

Thoria powder metallic impurities were determined by standard emission spectrographic methods with estimated precisions of ±30%. Carbon content was determined by standard combustion methods. The method utilized measures total carbon including absorbed carbon dioxide. Loss of weight on ignition was determined by heating in air at 1000°C.

B. Specific Surface Area

Thoria powder specific surface area values were determined by the BET (Brunauer, Emmett-Teller) nitrogen adsorption method using a Micromeritics Surface Area Analyzer, Model 2200. Instrument bias was corrected by instrument calibration with a National Bureau of Standards titania powder of known surface area.

C. Particle Size Distribution

Powder particle size distribution was measured with a Micromeritics Sedigraph Model 5000GB Particle Size Analyzer. The instrument determines, by means of a finely collimated beam of X-rays, the concentration of particles remaining in suspension at various sedimentation depths as a function of time. Ultrasonic agitation and a Calgon surfactant were used to disperse the powder prior to sedimentation. Three sedimentation determinations were performed for each sample. The values shown in Table 5 are averages of the three determinations.

D. Scanning Electron Microscopy (SEM)

Microscopy of the thoria powders was performed by the Physics Methods Laboratory, Analytical Chemistry Division, Oak Ridge National Laboratory.

IV. FABRICATION OF PELLET SINTERABILITY TEST SERIES

The Sinterability Test Pellet Fabrication Flow Diagram is shown in Figure 2.

The thoria powder, either in the as-received, one-pass micronized, or two-pass micronized condition, was agglomerated with a solution of 1.25 w/o Carbowax 6000 and 0.2 w/o Sterotex in Oxylene M-6 in a planetary mixer. Carbowax 6000 is a polyethylene glycol with a molecular weight between 6000 and 7500. Sterotex is an ester of glycerol and stearic acid. Oxylene M-6 is a Freon fluorinated methylene chloride. The damp agglomerates were hand granulated through USS #16, 20, and 30 sieves, and dried to constant weight in an air oven at 45 degrees C. The batch sieve analyses of the granules at this point in processing are summarized in Table 1.

Five solid, dished-end pellets were compacted at each of the following green densities: 56, 58, 60, 62 and 64 percent of theoretical in a 0.607 inch diameter die at a length to diameter ratio of one. Powder charges for the pressing operation were individually weighed and hand fed to the carbide-lined die in the Dorst TPA-45 compacting press. The tooling used in the press set-up was identical to that used for the production of blanket-type pellets for the LWBR (Reference 2). Press adjustments were made during the course of compaction runs to assure double-action pressing throughout.

The green pellets were individually identified and loaded into Inconel boats for binder removal pretreatment in flowing CO_2 at 925°C for three hours. After pretreatment, the pellets were transferred to molybdenum boats and sintered at 1790°C for 12 hours in wet hydrogen. The detailed pretreating and sintering parameters used throughout this study are summarized in Appendix A.

After sintering, the individually identified pellets were measured and weighed to determine geometric density according to the procedures described in Appendix B. Following density evaluation, a single pellet sample representative of each pellet green density group was withdrawn for microstructure evaluation and batch samples were taken for chemical analysis.

V. RESULTS AND DISCUSSION

A. Powder and Pellet Chemical Composition

Table 2 shows the chemical composition of the four vendor powders, and the maximum allowable impurity limits for LWBR thoria powder. The chemical analyses

show that the Vendor A powder meets all LWBR impurity requirements except Ca and Cl + Br. Cl + Br exceeds the allowable limits by 380 ppm. Ca would be unacceptable for LWBR binary fuel fabrication, and only 20 ppm in excess of the specified LWBR limit for use in thoria pellet fabrication. The addition of small quantities (210 ppm) of calcia by coprecipitation during powder manufacture enhanced the densification of LWBR thoria pellets (Reference 2). The Vendor C and Vendor N thoria powders showed only the Cl + Br contents to be in excess of the specification limit for these elements by 64 and 8 ppm, respectively. The Vendor S powder analysis showed that C, Ca, Cl + Br, Mg, and Si exceeded the allowable specification maxima by 800, 190, 79, 32, and 70 ppm respectively. In this case, it is presumed that the high carbon results from the high adsorbtive capacity of the high surface area Vendor S powder for CO_2. The surface of the thoria particles and their internal pores become coated with almost a complete monolayer of CO_2 upon exposure to air at room temperature (Reference 6). The carbon contents listed in Table 2, thus, contain an adsorbed CO_2 contribution which increases with increasing powder surface area.

The high values of C, Br, and Cl are usually reduced to acceptable levels during high temperature sintering. The Ca content of thoria powder is readily adjusted during powder manufacture, however, the effect of high values of Mg & Si require evaluation on a case basis. The minor deviations in these impurities above the LWBR chemical specification limits should not affect powder fabricability.

The chemical analysis data summarized in Table 3 relates the chemical composition of sintered pellets prepared with Vendor A thoria in the as-received and one-pass and two-pass micronized conditions with the chemical composition of the as-received powder. The pellet analyses show no increase in the elements Fe, Cr, or Ni which might be expected to be picked up from the stainless steel hardware of the micronizer (Reference 6). The data also show reduction in C, Ca, Cl + Br, and F content in the sintered pellets, while the element N showed a substantial increase. The increase in nitrogen content is attributed to contamination of the furnace atmosphere with nitrogen purge gas. The formation of nitrides on the surface of zirconia pellets sintered under identical conditions was traced to nitrogen contamination of the hydrogen atmosphere. The data show essentially no change in the balance of impurity elements.

B. Surface Area

Surface Areas obtained for vendor powders in the as-received and the one-pass and two-pass micronized conditions are summarized in Table 4.

The surface areas of the as-received Vendor A and Vendor N powders were 3.83 and 4.91 m^2/gm, respectively and were well within the limits of 3.0 to 6.0 m^2/gm required for as-calcined LWBR thoria (Reference 1). Vendor C powder, however, measured slightly below the minimum requirement with a surface area of 2.84 m^2/gm. The as-received Vendor S powder showed a surface area of 15.25 m^2/gm, the highest value observed.

The comparative response of Vendor A, C, S and N powders to one-pass and two-pass micronizing is shown by the surface area plots in Figure 3. In spite of the different starting surface areas, Vendor A and Vendor C powders showed an equivalent response to both one-pass and two-pass micronizing. One-pass micronizing increased the surface area of both of these powders by a factor of 1.9, and two-pass micronizing further increased the surface area of both powders by a factor of 2.6 over the as-received values. The surface area of Vendor N powder increased by a factor of 1.4 after one-pass and by a factor of 1.6 over the as-received value after the second micronizing pass. The Vendor S powder, which showed a very high as-received surface area of 15.8 m^2/gm, showed an increase factor by a 1.1 after one-pass and 1.3 over the as-received value after the second pass.

C. Particle Size Distribution

Table 5 summarizes the Sedigraph sedimentation particle size distribution data for the vendor powders in the as-received, one-pass and two-pass micronized conditions.

In order to show a direct comparison of the as-received particle size distributions of the vendor powders, the applicable data from Table 5 are plotted in Figure 4. The curves show the similarity in size distributions for Vendor A, C, and N powders. The distributions for Vendor A and C powders are essentially identical for the size range twenty to two microns, whereas the Vendor N powder is essentially identical with the A and C powders down to 3 microns size. The powder from Vendor S is clearly a much finer material than the powders from the other vendor sources with 76% of the as-received material finer than 1 micron.

Figures 5, 6, 7, and 8 are plots of the data in Table V to show the effect of one-pass and two-pass micronizing on the size distribution of the individual vendor thoria powders. The curves for the Vendor A, C, and N powders show that the greatest shift in size distribution occurred as a result of the first micronizing pass. This treatment shifted all three size distributions to 100% finer than 4 microns equivalent spherical diameter. The second micronizing pass produced a substantial increase in the finer than one micron size fractions. This micronizing behavior is equivalent to that observed by Clayton in his study of LWBR thoria powders (References 3-6). One or two-pass micronize milling of Vendor S powder resulted in a slight shift in the particle size distribution of the powder as shown by the distribution curves of Figure 8.

D. Scanning Electron Microscopy (SEM)

Figures 9, 10, 11, and 12 show the SEM photographs of the vendor thoria powders in the as-received condition. The square and rectangular plate-like particles shown in Figures 9, 10, and 12 (Vendors A, C and N, respectively) appear to be typical thorium oxalate pseudomorphs (References 3-6). Examination of Figure 11 (Vendor S) shows a combination of what appears to be fractured oxalate pseudomorph platelets rounded by milling, platelets coated with finely divided particles, and agglomerates of finely divided particles produced during the milling operation.

E. Sinterability Test Pellet Series Evaluation

Attempts to fabricate defect-free pellets with both as-received and micronized Vendor S thoria powders by the process described in Section IV were unsuccessful. Green pellets compacted at 62% and 64% TD showed cracks and pressure lamination defects upon ejection from the die. Pellets compacted at the lower green densities of 56%, 58% and 60% TD with as-received and one-pass and two-pass micronized feed powder, although apparently defect-free in the green condition, cracked and spalled during subsequent sintering. Collection of meaningful dimensional, density or microstructure data from these pellets was impossible, however, these results confirm prior experience (References 1 & 2).

Tables 6, 7 and 8 summarize the dimensional and density evaluation of the sinterability test pellet series prepared with the Vendors A, C and N powders. Figure 13 shows sinterability curves for pellets made with each of the three vendor powders in the as-received condition. These curves provide a direct

comparison of the relationship between green density and sintered density for each of the vendor powders. Figures 14 and 15 provide the same sinterability comparison for pellet series made with each of the three vendor powders in the one-pass and two-pass micronized conditions.

Figure 13 shows that the as-received thoria powders produced pellets with sintered densities in three widely separate ranges that can be correlated with starting powder surface areas. For example, Vendor N powder, with a surface area of 4.9 m^2/gm, produced pellets in the highest density range (92.0% - 94.5% TD), Vendor A pellets made with powder having a surface area of 3.8 m^2/gm fell into an intermediate range (86.6% - 91.0% TD), and Vendor C pellets, made with powder of surface area 2.8 m^2/gm, fell into the lowest range (71.6% - 81.3% TD). A comparison of the slopes of the density curves shows the qualitative difference in the rate of change of sintered density with change in green density for the three pellet series and reflects differences in fabrication behavior of the powders. The curves also indicate the upper limits for compaction of defect-free pellets for each of the as-received powders, namely 64% TD for Vendor A powder and 62% TD for both Vendor C and N powders.

The pellet sinterability plots for one-pass micronized powders are shown in Figure 14. These curves show that the mechanical activation which resulted from the one-pass micronizing treatment provided a substantial increase in the sintered density of each of the vendor pellet series. The reduction in the slopes of the curves, compared with the curves for the pellets made with as-received powder, indicates a definite decrease in the effect of green density on sintered density. The micronizing treatment also improved the compactability of the Vendor N and C powders and permitted the compaction of defect free pellets up to a green density level of 64% TD. The sintered density range of the pellets made with Vendor N powder was increased to 97.0% to 97.2% TD, while Vendor A and Vendor C pellet densities were increased to the ranges 95.3% to 96.5% and 92.3% to 96.8%, respectively. Vendor N, A, and C Powders showed surface area values of 7.05, 7.34, and 5.44 m^2/gm, respectively, after the one-pass micronizing (see Table 4).

The plots shown in Figure 15 indicate the change in pellet sinterability which occurred as a result of the second micronizing pass. The Vendor N pellets showed only a small change in sintered density, while the pellets made with

Vendor A and Vendor C powders showed a substantial increase over the densities attained after the single pass micronizing treatment. The second micronizing pass had little effect on the slope of the density curve for the Vendor C pellets, whereas the Vendor N and Vendor A curves assumed definite negative slopes. The change in slope suggests an increase in porosity as discussed in the following sections. Vendor N, A, and C powders showed surface area values of 7.96, 10.08, and 7.45 m^2/gm, respectively, after the second micronizing pass (see Table 4).

The negative slopes shown for Vendor N and Vendor A powders suggest that the second micronizing treatment overactivated these powders and produced a detrimental effect on sintered density. Re-examination of Figure 14 at this point shows that adequate sinterability was obtained with the first micronizing pass for the Vendor N powder to produce pellet densities of 97% TD. Examination of Figures 14 and 15 show a different behavior for the Vendor A powder. The Vendor A curves suggest that a less severe second micronizing treatment would have produced pellets with sintered densities > 96% TD. Less severe micronizing may be achieved by increasing the powder feed rate. Figures 14 and 15 also show the substantial improvement in sinterability of Vendor C powder obtained from the second micronizing pass which produced pellets with densities > 96% TD over the green density range employed.

The pellet density data from Tables 6, 7 and 8 are replotted in Figures 16, 18, and 20 in order to emphasize the effects of the one-pass and two-pass micronizing treatments used in this study on the sinterability of pellets made with each of the vendor powders. In conjunction with the sinterability curves, representative microstructures of pellet axial sections presented in Figures 17, 19, and 21, are arranged to show the interrelationship between powder condition, green and sintered pellet densities and pellet microstructure for each vendor powder. The photomicrographs shown were taken at the geometric center location of the axial sections and reflect the internal grain size values shown in Table 9.

1. Vendor A Pellet Sinterability Curves and Microstructure

The density curves of Figure 16 show the change in sinterability of pellets fabricated with as-received and with one-pass and two-pass micronized Vendor A powder. The pellet grain size values shown in Table 9 indicate a well developed

grain structure only at the edges of pellets made with as-received powder. The acceptable LWBR grain size range was ASTM 4-13. However, well-developed polyhedral grains were readily observable in pellets made with one-pass and two-pass micronized powders over the green density range investigated. The microstructures of pellets made with one-pass micronized powder show a predominance of intragranular porosity except at the 64% TD green density level where a substantial quantity of intergranular pores become evident as seen in Figure 17. In contrast, the microporosity in pellets made with two-pass micronized powder is primarily intergranular and increases with increasing green density. This increase in the amount of intergranular porosity may account for the decrease in sintered density which occurs in pellets made with the two-pass micronized powder (Reference 1).

The data indicate that high density, crack-free pellets with well-developed microstructures can be fabricated with one-pass micronized Vendor A thoria powder. The as-received Vendor A powder was incapable of producing pellets with sintered densities greater than 91% TD or microstructures with well-developed edge and center grain structures using the fabrication techniques described in Section IV. Based on the behavior of the pellets made with two-pass micronized material, this comminution treatment enhanced microstructure development, but produced a pellet sinterability curve with a negative slope indicating a reduction of density as a result of increasing intergranular porosity.

2. Vendor C Pellet Sinterability Curve and Microstructure

The density curves shown in Figure 18 demonstrate that the one-pass micronizing comminution treatment also improved the fabricability and sinterability of the Vendor C as-received powder by increasing the pellet as-sintered densities into the range of 92.3% to 96.8% TD. In these plots, the sinterability curves for pellets made of as-received and one-pass mcironized powders are essentially straight lines with steep, positive slopes. The sinterability curve for pellets made of two-pass micronized powder, however, is a level line showing essentially constant sintered densities in the range 97.1% to 97.6% TD over the entire green density range.

The grain size measurements for microstructures representative of the Vendor C pellets shown in Table 9 indicate very fine grain structure in pellet sections made with as-received powder. Pellets made with one-pass micronized

powder showed improved grain development for pellets compacted at 62% and 64% TD. Pellets made with two-pass micronized powder show well developed grain structures with intergranular porosity over the entire green density range (See Figure 19).

Examination of as-polished axial sections of pellets made with Vendor C powder showed that crack defects occurred only in sintered pellets made with two-pass micronized powder compacted at green density levels of 64% and 66% TD.

The data indicate that high density, crack-free pellets with well-developed microstructures can be fabricated with one-pass micronized vendor powder compacted at 64% and 66% TD or with two-pass micronized powder compacted at green densities in the range of 56% to 60% TD. The as-received powder was incapable of producing pellets with sintered densities greater than 81.3% TD or grain size greater than ASTM 13. The upper limit for the compaction of defect-free green pellets with as-received powder was 62% TD, however, the one-pass micronizing treatment apparently improved the compactability of the powder and permitted fabrication of defect-free pellets over the range 56% to 64% TD. As indicated previously, however, the compaction range of two-pass micronized material was limited by cracking to 56% to 60% TD but pellets sintered in this range reached densities in excess of 97% TD.

3. Vendor N Pellet Sinterability Curves and Microstructures

The density curves shown in Figure 20 for Vendor N powder show, once again, that one-pass micronizing treatment improved the compactability and sinterability of the as-received powder and produced pellets with as-sintered densities in the range of 97.0% to 97.2% TD. In this case, the sinterability curve is essentially level, with a tendency to slope slightly upward at the higher green densities. The sinterability curve for pellets made with two-pass micronized powder, however, assumes a negative slope similar, but less severe than that observed for Vendor A pellets made with two-pass micronized powder (See Figure 16 for comparison with Figure 20).

The grain size values shown for the Vendor N powders in Table 9 show very fine grain structures in pellets made with as-received powder and compacted at green densities of 56, 58, and 60% TD, while a fine grained microstructure (ASTM 10-11) can be observed in pellets compacted with this as-received powder at green densities of 60% and 62% TD. Pellets compacted at 64% TD failed upon

ejection from the die. Well developed microstructures composed of polyhedral grains with grain boundary porosity, are seen to be typical of as-sintered Vendor N pellets made of both one-pass and two-pass micronized feed powder. See Table 9 and Figure 21. For both materials, however, the green density range for the fabrication of defect-free pellets was limited by excessive radial cracking which appeared in as-sintered pellets compacted at density levels of 62% and 64% TD.

The data for Vendor N material indicate that high density, crack-free pellets with well-developed microstructures can be fabricated with one-pass and two-pass micronized Vendor N powder compacted in the green density range of 56% to 60% TD. The as-received powder was incapable of producing pellets with sintered densities in excess of 94.5% TD, and the upper limit for the fabrication of defect-free pellets with this material was a green density of 58% TD. Although the increase in powder activity resulting from both one-pass and two-pass micronizing permitted the formation of well-developed microstructure in pellets compacted over the entire green density range, the occurrence of crack defects limited the useful range for the fabrication of defect-free pellets to green densities in the range of 56% to 60% TD for both powder comminution levels.

VI. SUMMARY OF RESULTS

The three vendor-source powders evaluated in this study showed the plate-like particle morphology typical of oxalate-derived thoria similar to LWBR NLO powder. The difference in impurity content, surface area, particle characteristics and fabricability of the vendor powders, however, suggest substantial differences in the thorium oxalate conversion critical parameters compared with the NLO process.

Vendor S powder, which showed a high value of surface area (15.25 m^2/gm), was unacceptable for the fabrication of high density crack-free thoria pellets by the modified LWBR press-sinter process. This confirms prior LWBR development and manufacturing experience wherein feed powders with surface areas much in excess of about 8 m^2/gm present problems in the fabrication of crack-free pellets.

None of the as-received thoria powders evaluated, including the NLO powder, was capable of yielding high density (96% TD) LWBR-type blanket-size thoria pellets.

High density (96% TD) crack-free pellets with acceptable microstructures (ASTM 3-13) were produced with single-pass micronized powders by compaction under the following conditions:

Vendor	Powder Surface Area (m^2/gm)	Green Density (% TD)
A	5.4	60, 62
C	7.3	62, 64
N	7.1	56, 58, 60

High density (96% TD) crack-free pellets with acceptable microstructures (ASTM 3-13) were produced with two-pass micronized powders by compaction under the following conditions:

Vendor	Powder Surface Area (m^2/gm)	Green Density (% TD)
A	10.1	56
C	7.5	56, 58, 60
N	8.0	56, 58

This data confirms the well-established relationship between powder surface area and compactability, i.e., the higher the surface area, the lower the green density at which compaction cracking occurs.

VII. CONCLUSIONS

1. Comparative scanning electron microscopy evaluation indicated that the as-received vendor powders were produced by thorium oxalate precipitation processes similar to that employed for the production of LWBR thoria powders. Differences in these powders may be attributed to variations in precipitation and calcination processing steps used in powder production, and in one case, to the use of post-calcining comminution.

2. In general, the chemical composition of the vendor powders compared favorably with the LWBR powder. In two of the vendor powders, the impurity elements which exceed LWBR limits would be substantially reduced in pellet sintering. The high values of Mg and Si found in one powder may be a cause for concern but requires further evaluation.

3. Two of the three vendor thoria powders evaluated successfully produced high density, crack-free LWBR blanket-type thoria pellets with acceptable microstructures when processed by the modified LWBR/AWBA micronize-press-sinter process. The poor response of the third vendor powder evaluated was attributed to the high as-received surface area of the powder.

4. Sinterability tests performed with vendor and the LWBR reference powder on pellets prepared from as-received thoria powders showed that high density pellets with acceptable microstructures could not be produced without mechanical activation of the powders to obtain sinterability. Process simplification by elimination of the powder micronizing operation is, therefore, not considered feasible for these powders.

VIII. REFERENCES

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APPENDIX A

Pretreating and Sintering Parameters

I. Pretreatment

Atmosphere: Flowing CO_2. Gas flow rates: 450 ft^3/hr at furnace exit, 150 ft^3/hr at furnace entrance.

Furnace Boats: Inconel

Heating Schedule: Heat to 425°C at 50°C/hr, hold at temperature for 4 hours, resume heating at 50°C/hr to 925°C and hold at temperature for 3 hours.

II. Sintering

Atmosphere: Wet hydrogen (dew point = 75°F)

Furnace Boats: Molybdenum

Heating Schedule: Heat to 1790°C at 110°-115°C/hr and hold at temperature for 12 hours.

APPENDIX B

Dished-End Solid Pellet Dimensional and Weight Inspection Procedures

1. Outside Diameter (OD) Measurement

Each pellet was measured at three locations approximately 1/16 inch from each end and at the center of the pellet ±1/16 inch. Each pellet was rotated approximately 60° after each measurement. Each reading was recorded to 4 decimal places. The hand micrometers had 1/8 inch anvils with 0.0001 inch graduations, and were checked with gage blocks or plug gages at the nominal pellet dimension ±0.005 inch being measured. Micrometers were required to be true within ±0.0002 inch. Standards were checked before and after inspection of each pellet batch.

2. Length Measurement

Each pellet measured with 1/8 inch anvil micrometers to establish minimum and maximum length. Each measurement recorded to 4 decimal places. Calibrations of the micrometers for this measurement was performed by the same procedure outlined above for OD measurements.

3. Pellet Weight Measurement

Pellet weights were obtained on a Precision Balance with a readout of 0.001 gm minimum. Class "S" weight standards were used to adjust the balance near the nominal weight after every 10th pellet inspected. The balance was adjusted to read true at the value of the standard. Balance recheck acceptance was ±0.0005 inch.

Table 1

Sieve Analysis of Granular Press Feed

Vendor	Powder Condition	Weight Percent Retained on USS Sieve Number				
		25	40	70	140	-140
A	As-Received	0	0	0.4	1.9	97.7
A	One-Pass Micronized	0	30.6	39.5	15.3	14.6
A	Two-Pass Micronized	0	29.8	40.5	16.8	12.9
C	As-Received	0	0.8	10.0	11.1	78.0
C	One-Pass Micronized	0	20.0	36.0	20.1	23.9
C	Two-Pass Micronized	0	25.2	38.7	18.5	17.6
S	As-Received	0	7.4	33.9	26.9	31.8
S	One-Pass Micronized	0	19.3	36.3	21.6°	22.8
S	Two-Pass Micronized	0	17.5	35.3	23.2	24.0
N	As-Received	0	1.0	8.5	17.0	73.5
N	One-Pass Micronized	0	36.7	37.2	14.5	11.5
N	Two-Pass Micronized	0.1	54.3	26.1	10.7	8.8

TABLE 2

CHEMICAL COMPOSITION OF AS-RECEIVED THORIA POWDERS

Vendor:	A	C	S	N	LWBR Chemical Requirements Maximum Individual Limit
Chemistry No.:	80-562	80-507	80-512	81-520	
Batch No.:	80-3471-1	80-3471-2	80-3471-3	80-3471-3	
Vendor No.:	E3784	T-1088	PAZ-258	Lot 158	
Element					
		Parts Per Million			
Al	<50	<50	<50	<50	100
B	<0.25	<0.25	0.61	<2.5	1
C	334	284	1400	264	500
Ca	170	<25	240	<25	(1)
Cl + Br	395	79	94	23	15
Co	<2.5	<2.5	<2.5	<2.5	10
Cr	23	<15	<15	28	80
Cu	4	5	<2.5	<2.5	10
F	12	<5	10	3	20
Fe	<25	46	<25	<25	75
Mg	15	<10	82	<10	50
Mn	<2.5	<2.5	2.5	<2.5	5
Mo	5	<5	6	<5	25
N	14	16	8	20	40
Ni	<5	<5	<5	51	50
Si	<50	<50	170	<50	100
Ti	<2.5	12	10	<5	15
Hg	<5	<5	<5	<5	2
V	<2.5	<2.5	2.5	<2.5	10
Sn	8	10	<5	9	Not Specified
LOI (Percent)	0.348	0.197	0.832	0.060	Not Specified
Th (Percent)	87.42	87.95	86.73	87.82	87.0 min.

(1) Thoria with Calcium 150 ppm max, Thoria without calcium 50 ppm max.

TABLE 3

CHEMICAL COMPOSITION OF POWDER AND PELLETS
MADE WITH POWDER OBTAINED FROM VENDOR A

Chemical No.:	80-562	81-508 As-Sintered Pellets		
Element	Powder As-Received	As-Received	One-Pass Micronized	Two-Pass Micronized
Al	<50 PPM	52 PPM	<50 PPM	<50 PPM
B	<0.25	<0.25	<0.25	<0.25
C	334	20	20	18
Ca	170	68	64	56
Cl + Br	295	12	--	--
Co	2.5	2.5	2.5	2.5
Cr	23	<15	<15	<15
Cu	4	<2.5	<2.5	3
F	12	5	<5	<5
Fe	<25	27	<25	<25
Mg	15	<10	<10	<10
Mn	<2.5	<2.5	<2.5	<2.5
Mo	5	<5	<5	<5
N	14	54	52	26
Ni	<5	<5	<5	<5
Si	<50	<50	<50	<50
Ti	<2.5	<2.5	<2.5	<2.5
Hg	5	<5	<5	<5
V	<2.5	<2.5	<2.5	<2.5
Sn	8	7	7	<5

TABLE 4

BET Surface Area of Thoria Powders

In The As-Received, One-Pass, and Two-Pass Micornized Conditions

Vendor	Powder Condition	Surface Area M^2/GM
A	As-Received	3.83
A	One-Pass Micronized	7.34
A	Two-Pass Micronized	10.08
C	As Received	2.84
C	One -Pass Micronized	5.44
C	Two-Pass Micronized	7.45
S	As-Received	15.25
S	One-Pass Micronized	16.71
S	Two-Pass Micronized	19.25
N	As-Received	4.91
N	One-Pass Micronized	7.05
N	Two-Pass Micronized	7.96

TABLE 5

Sedigraph Particle Size Distrubutions of Thoria Powders in the As-Received, One-Pass Micronized, and Two-Micronized Conditions

Equivalent Spherical Diameter (Microns)	Percent by Weight Less Than											
	Vendor N Powder			Vendor A Powder			Vendor C Powder			Vendor S Powder		
	As-Received	One-Pass Micronized	Two-Pass Micronized	As-Received	One-Pass Micronized	Two-Pass Micronized	As-Received	One-Pass Micronized	Two-Pass Micronized	As-Received	One-Pass Micronized	Two-Pass Micronized
20				100			100			100		
15	100			99			99			99		
10	93			93			94			97		
8	81			80			86			95		
6	57			62			70			93	100	
5	46			50			58			92	99	
4	37	100	100	39	100	100	44	100	100	90	98	100
3	31	99	99	27	99	99	30	98	99	89	97	99
2	24	98	98	14	95	98	12	94	97	85	94	98
1	13	93	95	6	76	91	1	72	88	76	86	92
0.8	8	87	93	3	64	84	0	61	82	70	81	89
0.6	4	76	87	1	51	70		48	71	61	73	83
0.5	2	65	81	0	43	63		42	63	55	67	85
0.4	0	54	71		36	53		35	55	46	58	66
0.3		41	56		30	44		29	47	36	47	53
0.2		25	32		21	33		20	33	22	33	38
0.1			3		10	13		6	9	3	5	11

TABLE 6

SUMMARY OF DIMENSIONS AND DENSITY OF AS-SINTERED THORIA PELLETS AS A FUNCTION OF POWDER CONDITION AND GREEN DENSITY - VENDOR A POWDER

Batch No.	Feed Powder Condition	Test Pellet Group	Nominal Green Density %TD	As - Sintered Pellet			
				Length (in.)	Diameter (in.)	Weight (gms)	Geometric Density %TD
80-3471-1	As-Received	1	56	.523	.531	16.4157	86.6
		2	58	.528	.534	16.6148	87.9
		3	60	.533	.537	17.6056	89.1
		4	62	.538	.540	18.1874	90.1
		5	64	.543	.544	18.7783	91.0
80-3471-1-M1	One-Pass Micronized	1	56	.508	.510	16.1544	95.3
		2	58	.514	.514	16.7456	95.7
		3	60	.519	.520	17.3188	96.2
		4	62	.524	.525	17.8018	96.3
		5	64	.530	.530	18.4596	96.5
80-3471-1-M2	Two-Pass Micronized	1	56	.508	.507	16.0902	96.0
		2	58	.515	.514	16.6888	95.8
		3	60	.522	.519	17.2335	95.5
		4	62	.529	.525	17.8093	95.0
		5	64	.536	.531	18.4246	94.7

TABLE 7

SUMMARY OF DIMENSIONS AND DENSITY OF AS-SINTERED THORIA PELLETS AS A FUNCTION OF POWDER CONDITION AND GREEN DENSITY - VENDOR C POWDER

Batch No.	Feed Powder Condition	Test Pellet Group	Nominal Green Density %TD	As - Sintered Pellet			
				Length (in.)	Diameter (in.)	Weight (gms)	Geometric Density %TD
80-3471-1	As-Received	1	56	.556	.562	16.1686	71.6
		2	58	.556	.561	16.9686	75.4
		3	60	.555	.560	17.5749	78.6
		4	62	.557	.559	18.1929	81.3
		5	64	Pellets Laminated			
80-3471-2-M1	One-Pass Micronized	1	56	.519	.515	16.2851	92.3
		2	58	.521	.518	16.8725	93.9
		3	60	.528	.523	17.5896	94.8
		4	62	.531	.527	18.4392	96.0
		5	64	.536	.531	18.7925	96.8
80-3471-2-M2	Two-Pass Micronized	1	56	.511	.505	16.2804	97.2
		2	58	.517	.510	16.8598	97.6
		3	60	.523	.517	17.4595	97.2
		4	62	.529	.523	18.0720	97.2
		5	64	.535	.529	18.6835	97.1

TABLE 8

SUMMARY OF DIMENSIONS AND DENSITY OF AS-SINTERED THORIA PELLETS AS A FUNCTION OF POWDER CONDITION AND GREEN DENSITY - VENDOR N POWDER

Batch No.	Feed Powder Condition	Test Pellet Group	Nominal Green Density %TD	As - Sintered Pellet			
				Length (in.)	Diameter (in.)	Weight (gms)	Geometric Density %TD
80-3471-4	As-Received	1	56	.521	.516	16.3983	92.0
		2	58	.524	.520	16.9798	93.3
		3	60	.529	.524	17.5400	94.0
		4	62	.533	.530	18.1724	94.5
		5	64	Pellets Laminated			
80-3471-4-M1	One-Pass Micronized	1	56	.511	.505	16.2417	97.0
		2	58	.516	.512	16.8568	97.0
		3	60	.522	.518	17.4516	97.0
		4	62	.528	.524	18.0835	97.1
		5	64	.534	.530	18.7441	97.2
80-3471-4-M2	Two-Pass Micronized	1	56	.510	.505	16.2577	97.3
		2	58	.516	.512	16.8973	97.1
		3	60	.522	.520	17.6064	97.1
		4	62	.529	.525	18.1918	96.9
		5	64	.534	.531	18.7063	96.7

TABLE 9

SUMMARY OF ASTM GRAIN SIZE OF AS-SINTERED THORIA PELLETS
AS A FUNCTION OF POWDER CONDITION AND GREEN DENSITY

Powder Condition	Nominal Green Density % TD	Average ASTM Grain Size								
		Vendor A 80-3471-1			Vendor C 80-3471-2			Vendor N 80-3471-4		
		Edge	Midpoint	Center	Edge	Midpoint	Center	Edge	Midpoint	Center
As-Received	56	9-10	<13	<13	<13	<13	<13	<13	<13	<13
	58	9-10	<13	<13	<13	<13	<13	<13	<13	<13
	60	9-10	<13	<13	<13	<13	<13	<13	<13	<13
	62	9-10	<13	<13	<13	<13	<13	<13	10-11	<13
	64	8-9	<13	<13	<13	<13	<13	--	--	--
One-Pass Micronized	56	6.0	6.0	6	<13	<13	<13	9-10	4-5	5
	58	6.0	6.0	6	<13	<13	<13	9	4-9	4
	60	6.0	6-7	7	<13	12	<13	8-9	3-4	3
	62	6.0	6-7	7	10-11	8-11	10-11	7-9	4-5	4
	64	6.0	6-7	7	9	5-6	5	7	4	4
Two-Pass Micronized	56	4.0-6.0	6-7	7	6.0	4	4	4	5	5
	58	4.5-6.0	7.0	7	6.0	5	5	5	5	5
	60	5.0-5.5	7.0	7	6.0	5	5	4-5	5	5
	62	6.0	7.0	7	6.0	5	5	4-5	4	4
	64	6.0	7.8	8	6.0	5	5	4-5	4	4

FIGURE 1

THORIA POWDER MICRONIZING AND SAMPLING FLOW DIAGRAM

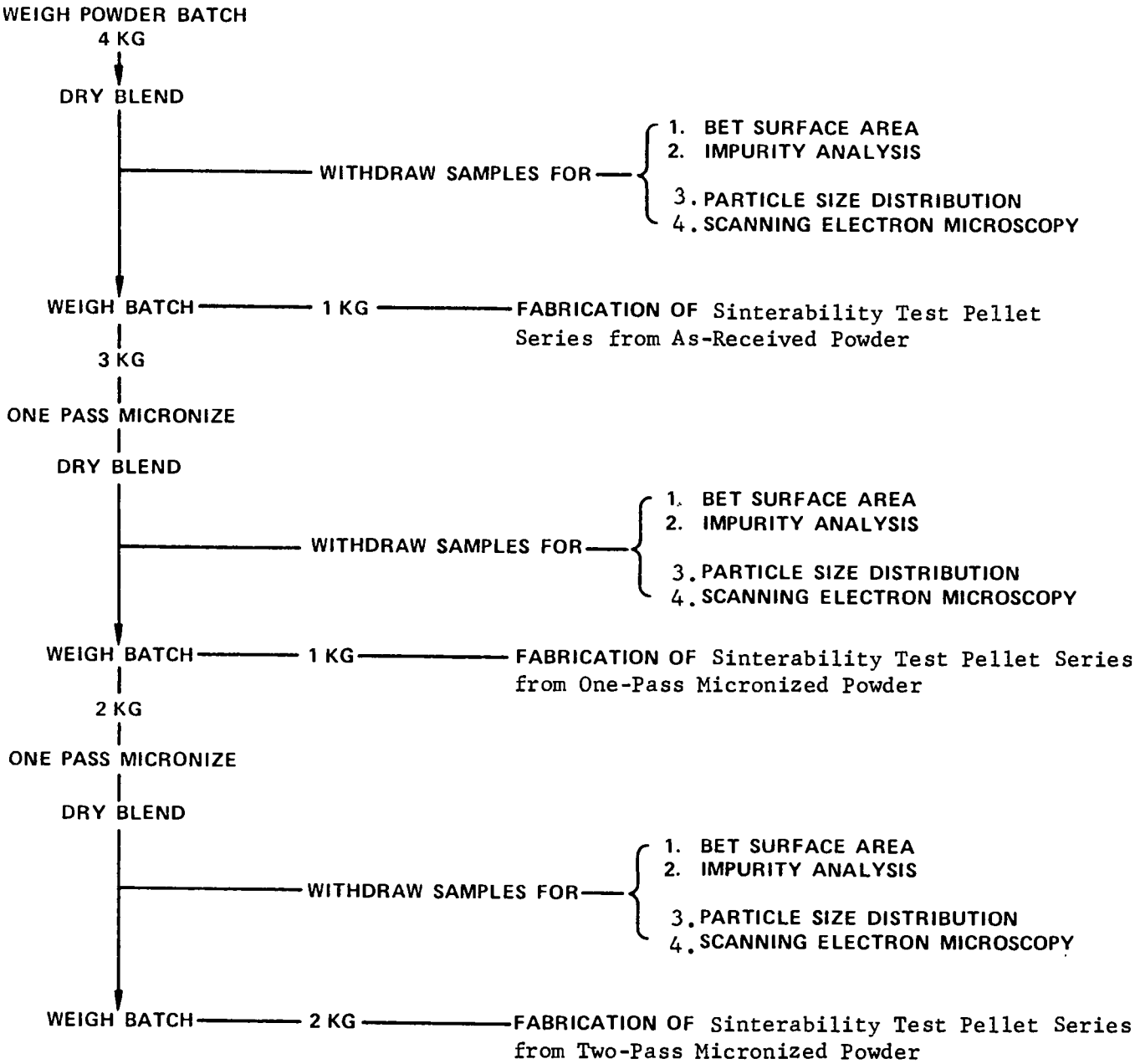


FIGURE 2
PROCESS FLOW DIAGRAM FOR THE PREPARATION OF THORIA SINTERABILITY TEST PELLETS

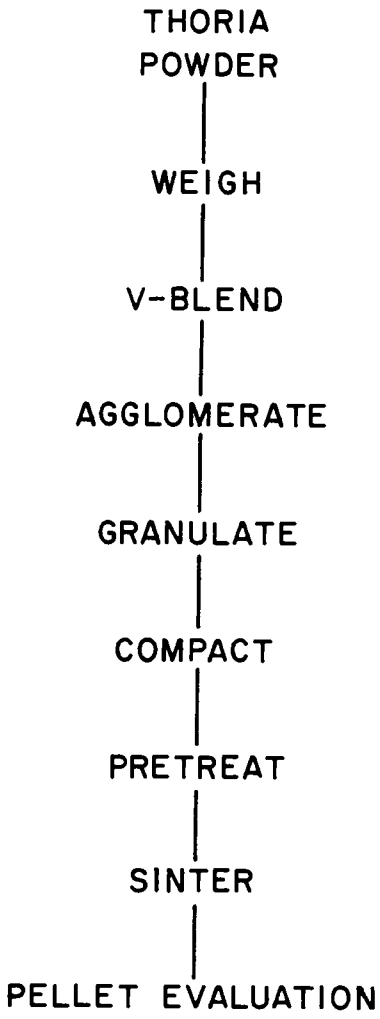


FIGURE 3
EFFECT OF MICRONIZING ON THE SURFACE AREA OF THORIA POWDERS

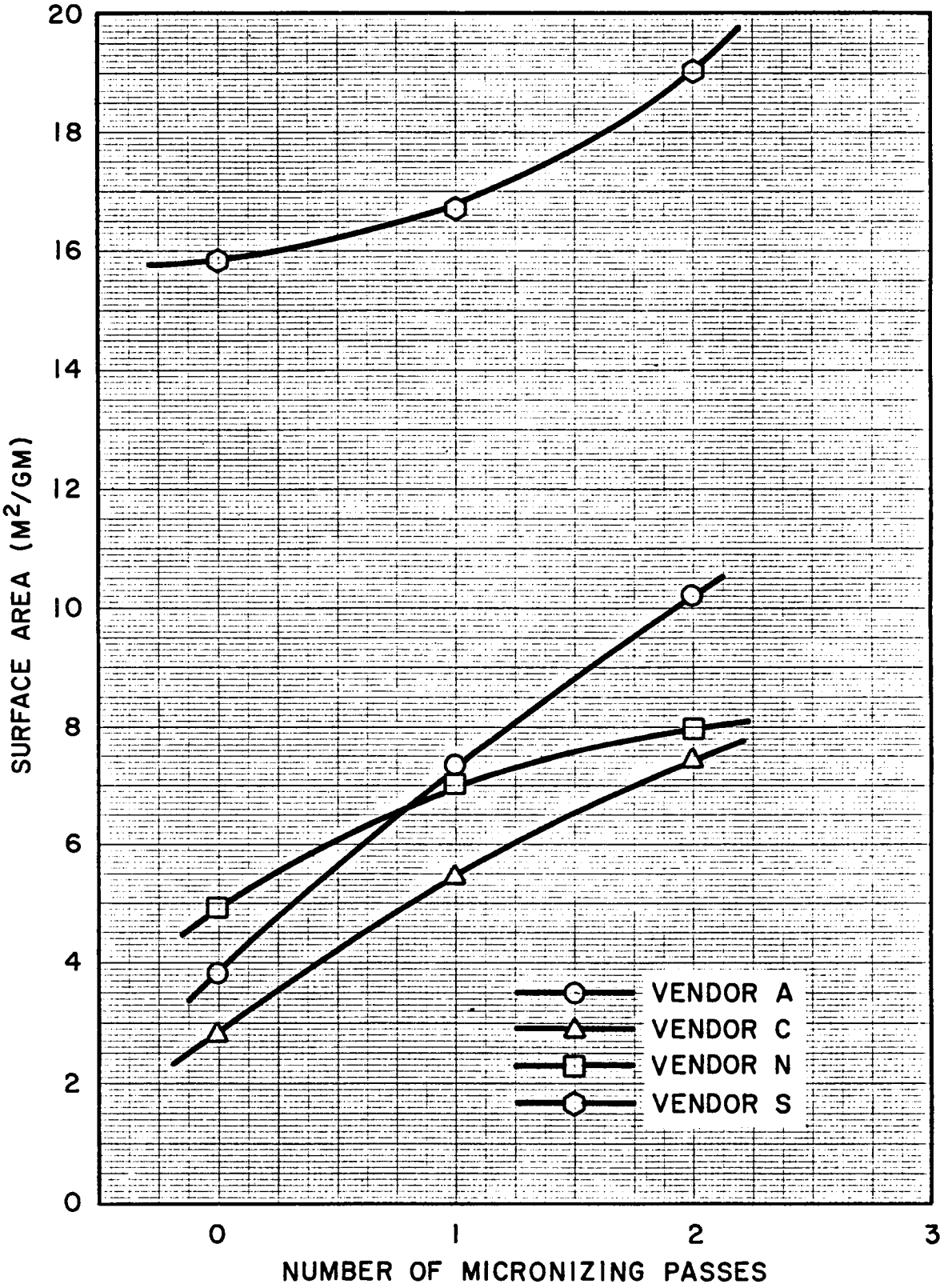


FIGURE 4

PARTICLE SIZE DISTRIBUTION PLOTS FOR VENDOR POWDERS IN THE AS-RECEIVED CONDITION

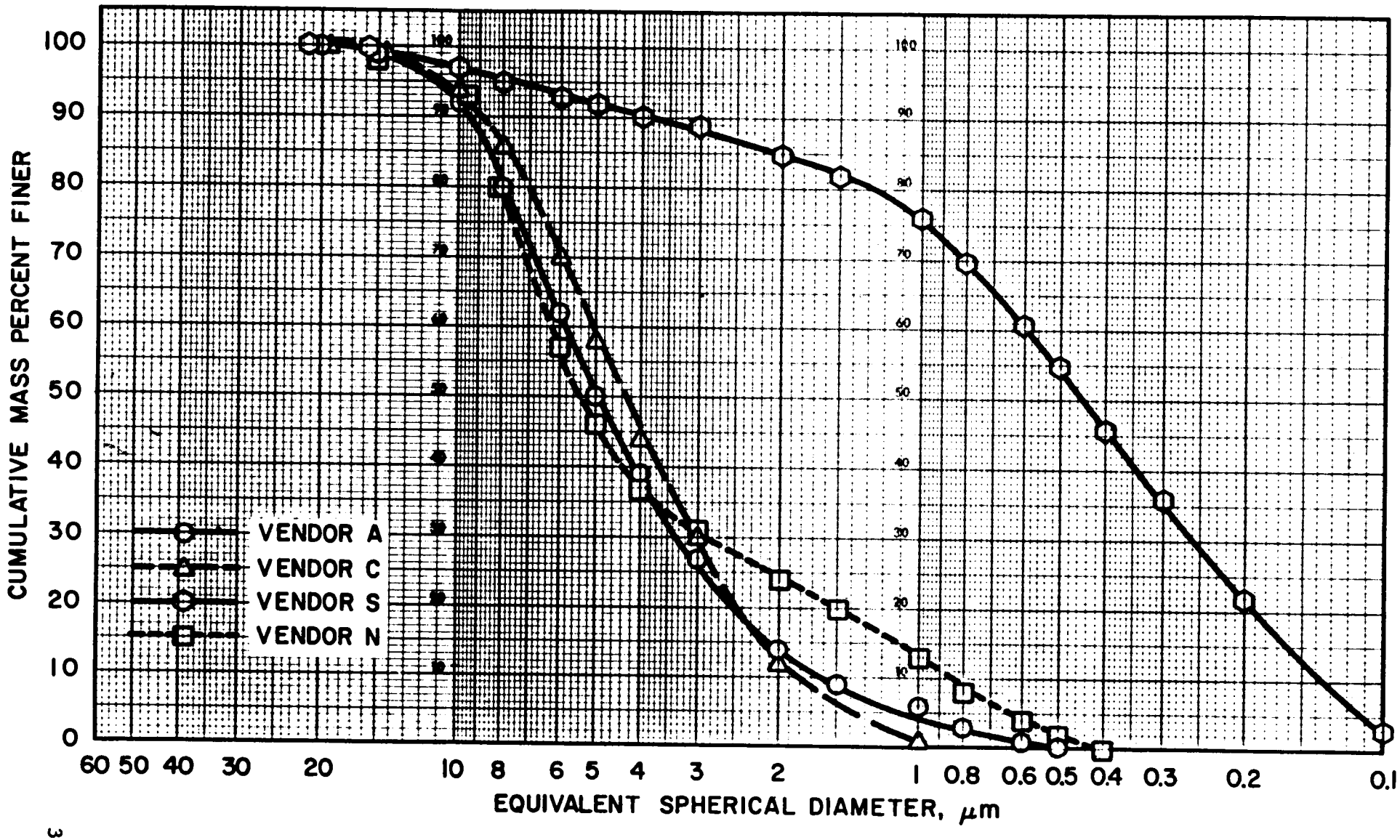


FIGURE 5

PARTICLE SIZE DISTRIBUTION PLOTS FOR VENDOR A POWDER IN THE AS-RECEIVED CONDITION AND AFTER ONE-PASS AND TWO-PASS MICRONIZING

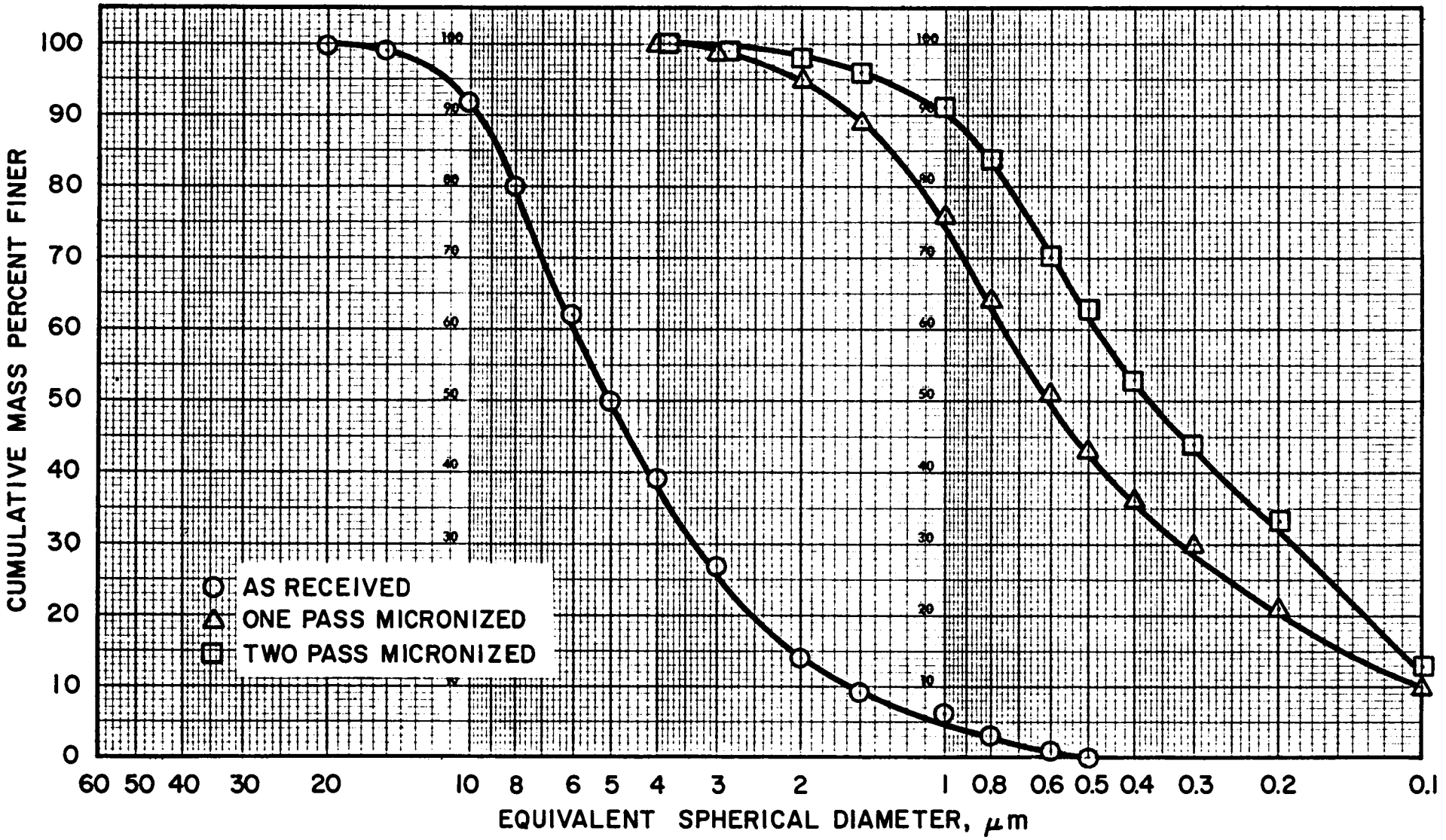


FIGURE 6

PARTICLE SIZE DISTRIBUTION PLOTS FOR VENDOR C POWDER IN THE AS-RECEIVED CONDITION AND AFTER ONE-PASS AND TWO-PASS MICRONIZING

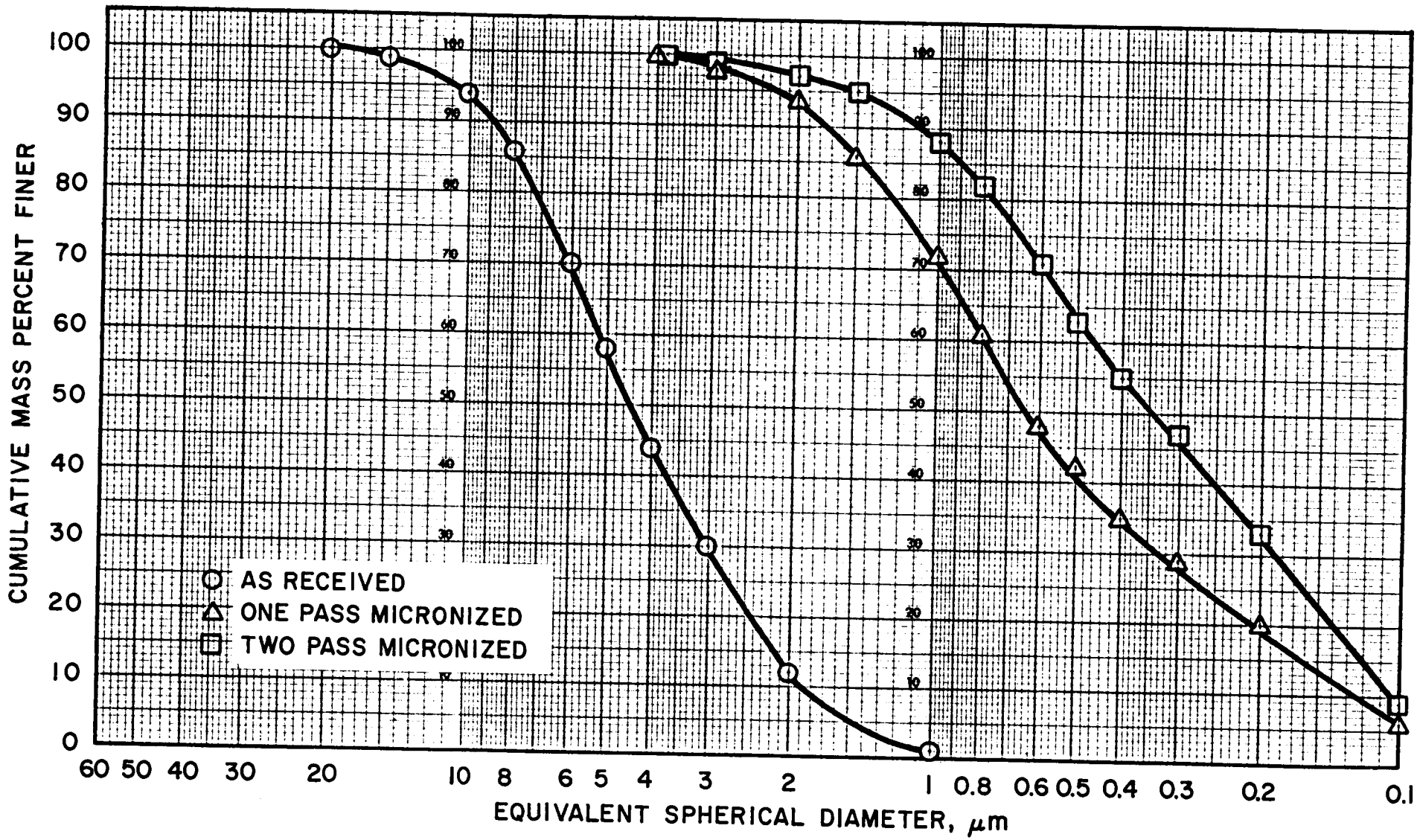


FIGURE 7

PARTICLE SIZE DISTRIBUTION PLOTS FOR VENDOR S POWDER IN THE AS-RECEIVED CONDITION AND AFTER ONE-PASS AND TWO-PASS MICRONIZING

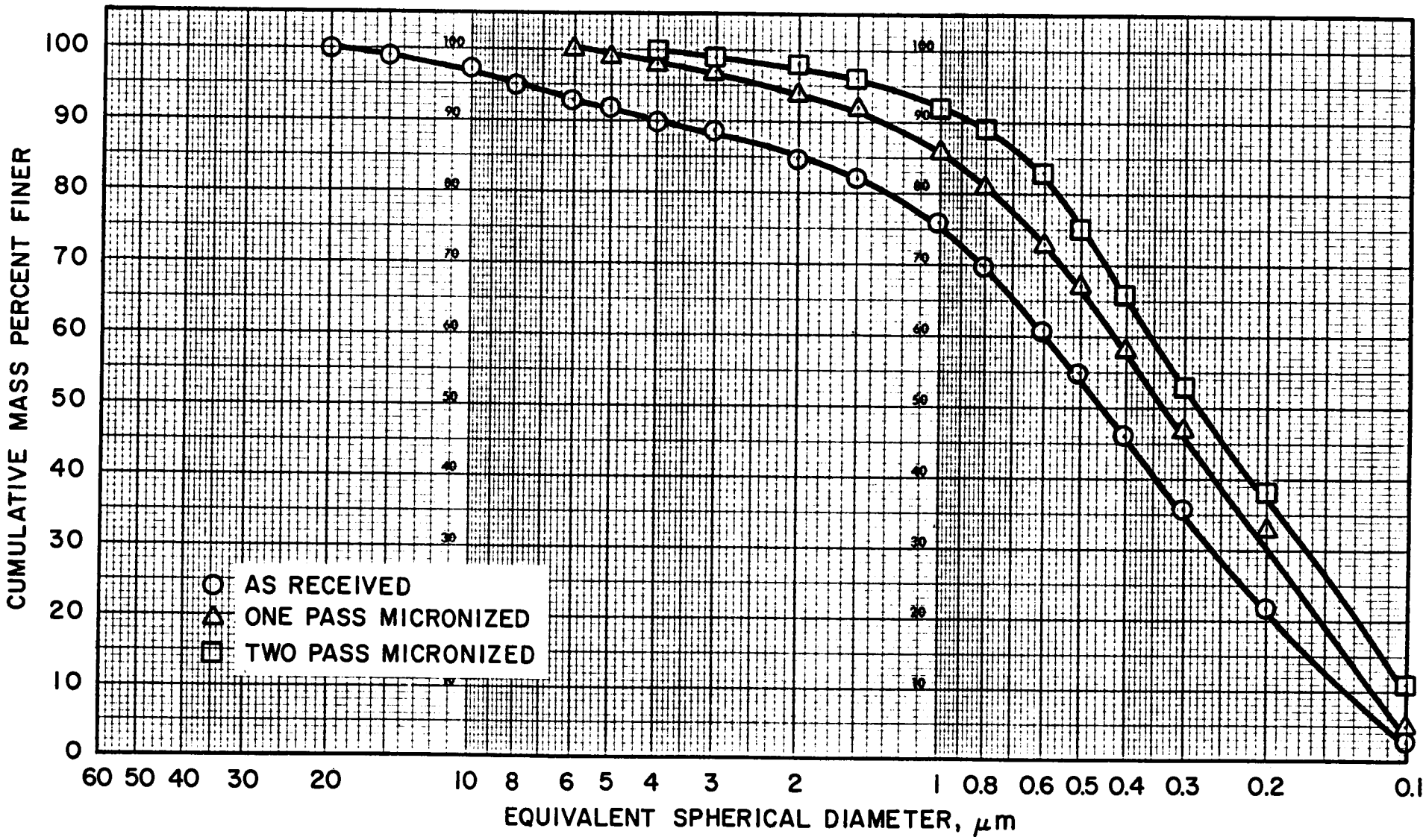
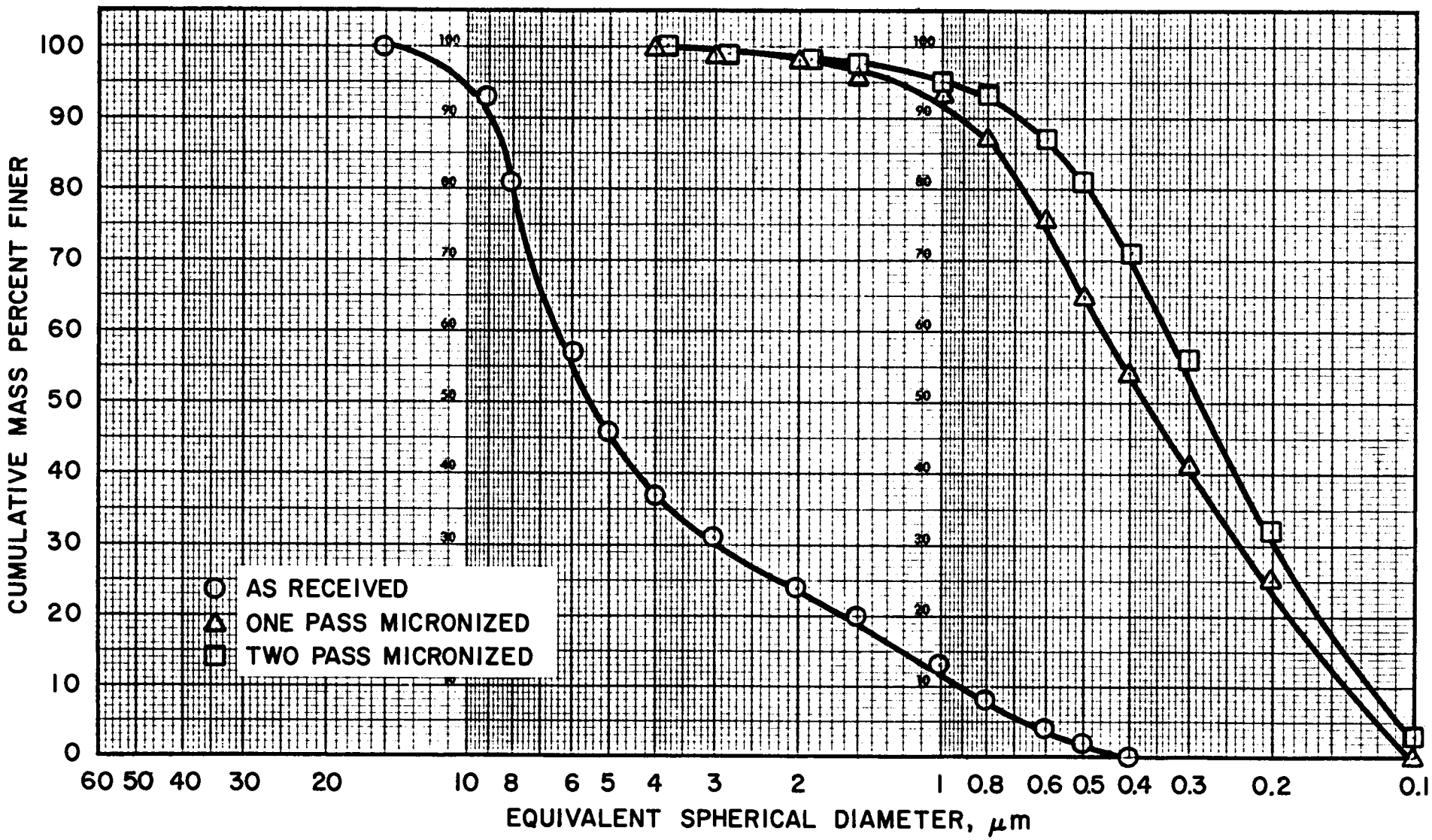
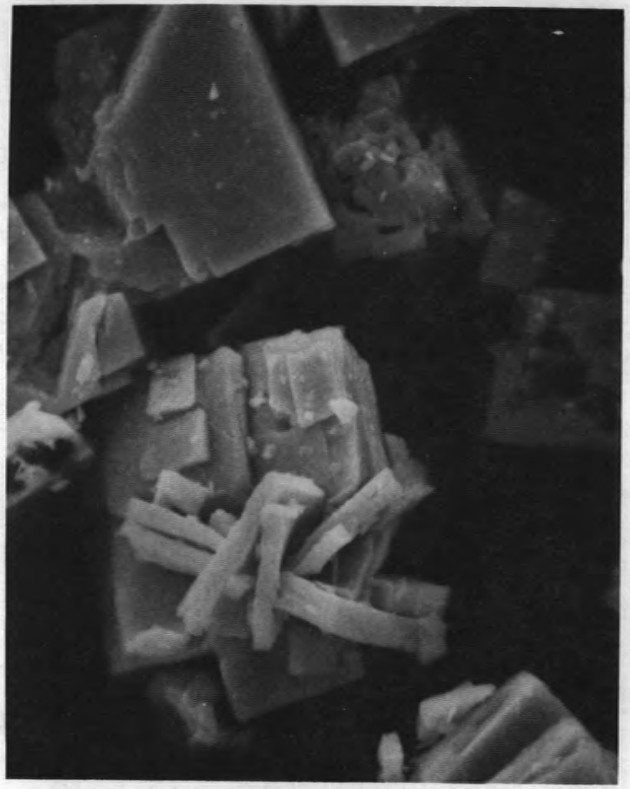


FIGURE 8

PARTICLE SIZE DISTRIBUTION PLOTS FOR VENDOR N POWDER IN THE AS-RECEIVED CONDITION AND AFTER ONE-PASS AND TWO-PASS MICRONIZING





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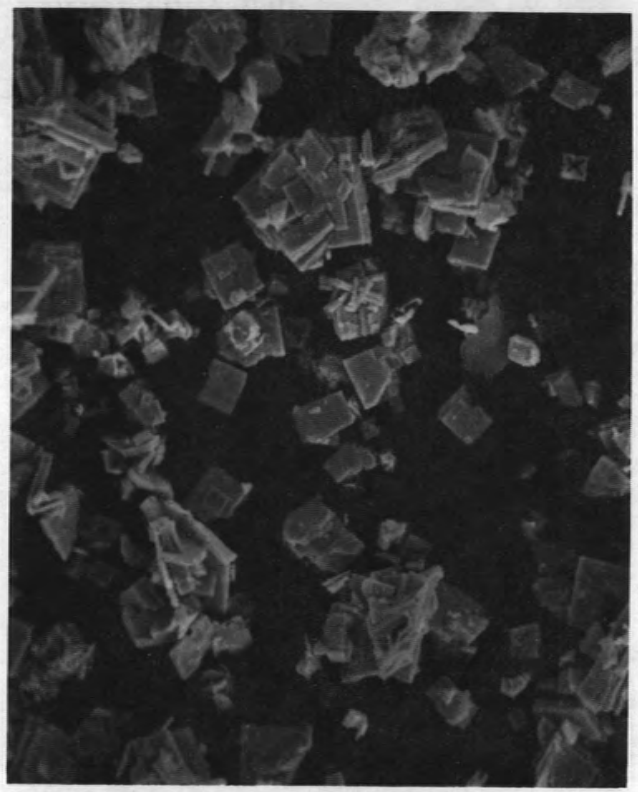
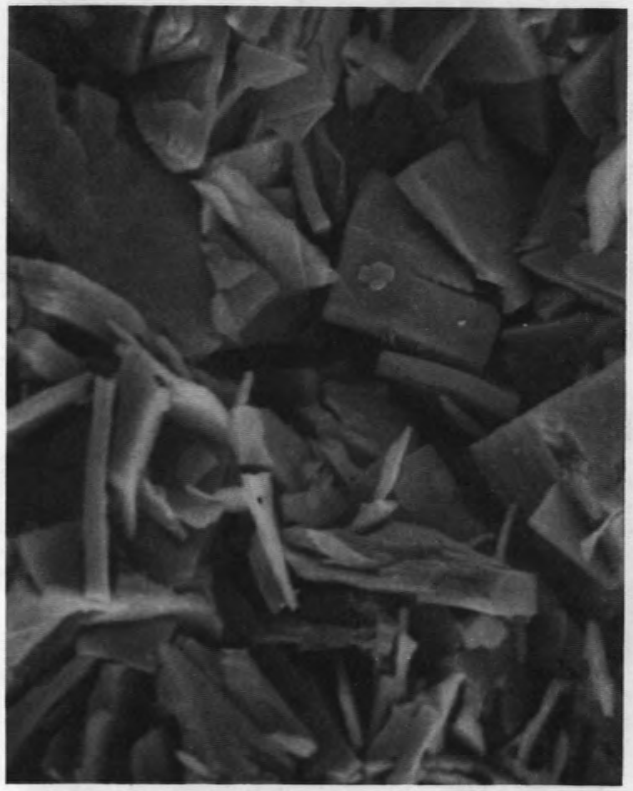


Sample No. S15264 Mag: 1000X

Batch Number 80-3471-1

SCANNING ELECTRON MICROGRAPHS OF VENDOR A POWDER IN THE AS-RECEIVED CONDITION

Figure No. 9



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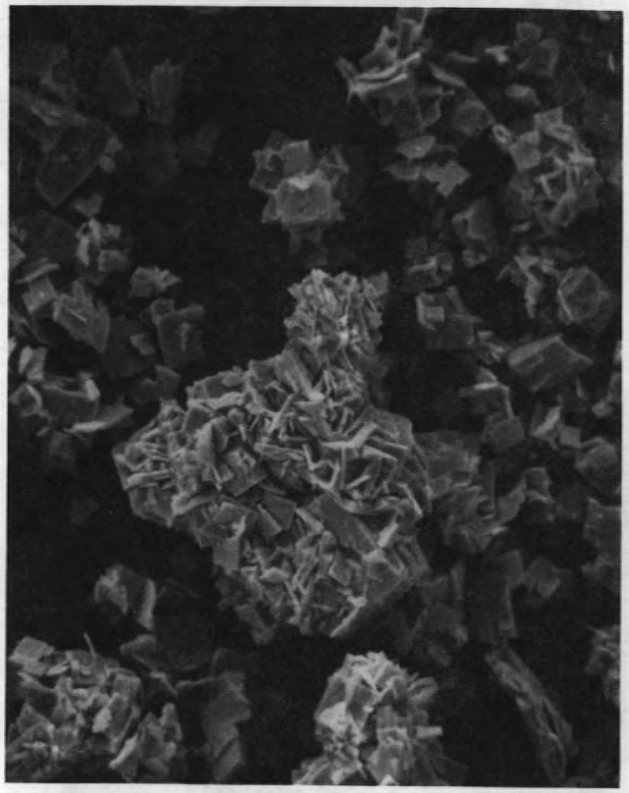
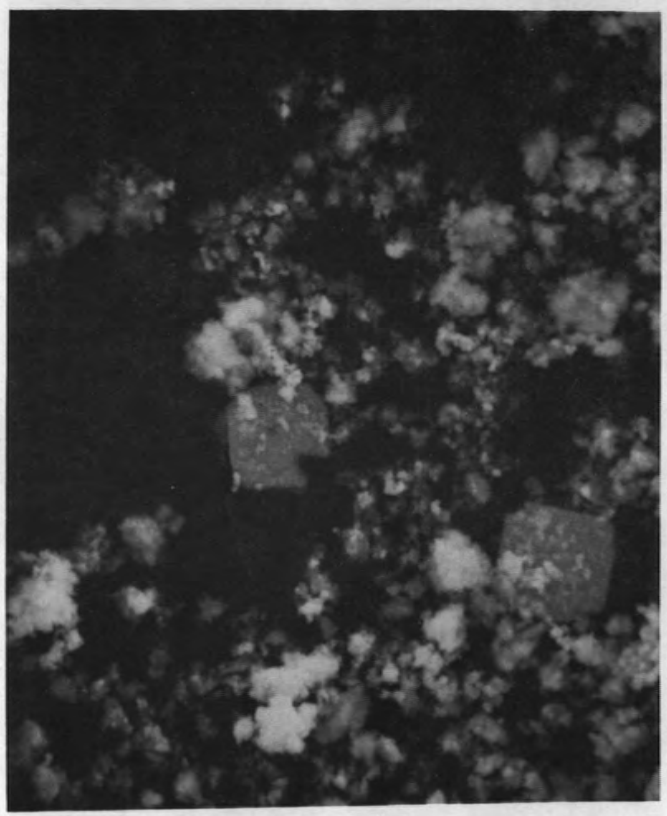


Sample No. S15264 Mag: 1000X

Batch Number 80-3471-2

SCANNING ELECTRON MICROGRAPHS OF VENDOR C POWDER IN THE AS-RECEIVED CONDITION

Figure No. 10



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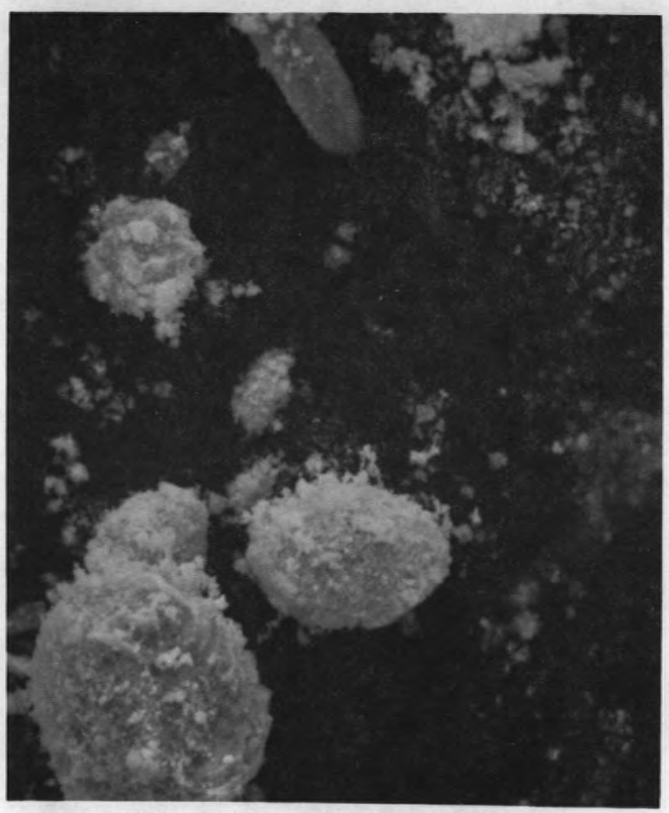
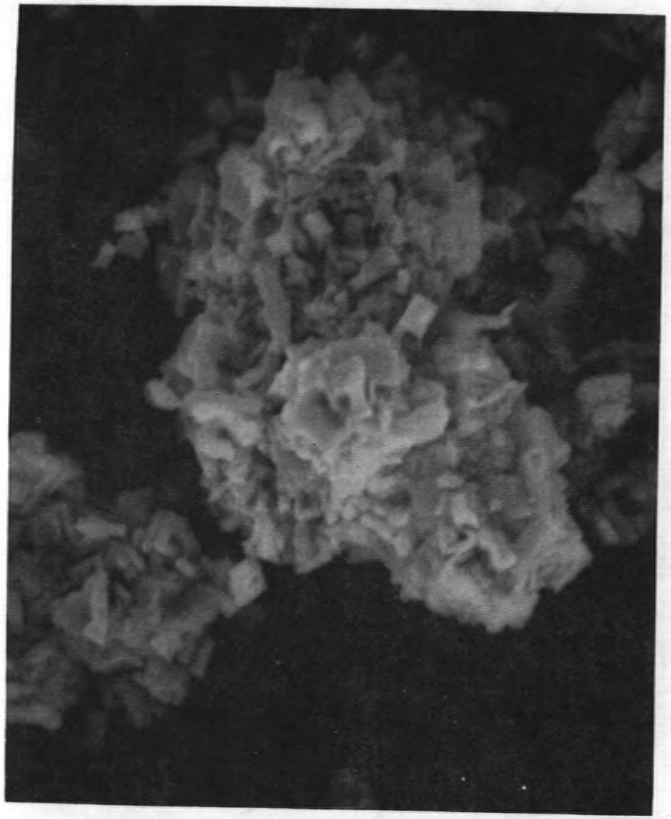


Sample No. S15341 Mag: 2000X

Batch Number 80-3471-3

SCANNING ELECTRON MICROGRAPHS OF VENDOR S POWDER IN THE AS-RECEIVED CONDITION

Figure No. 11



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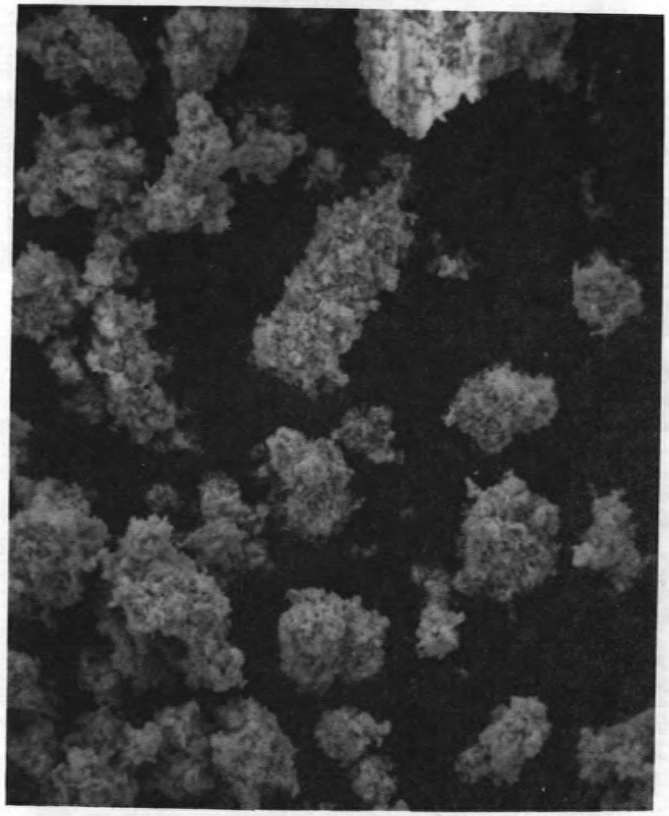


Sample No. S15321 Mag: 1000X

Batch Number 80-3471-4

SCANNING ELECTRON MICROGRAPHS OF VENDOR N POWDER IN THE AS-RECEIVED CONDITION

Figure No. 12

FIGURE 13

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH AS-RECEIVED THORIA POWDER FROM VENDORS A,C AND N

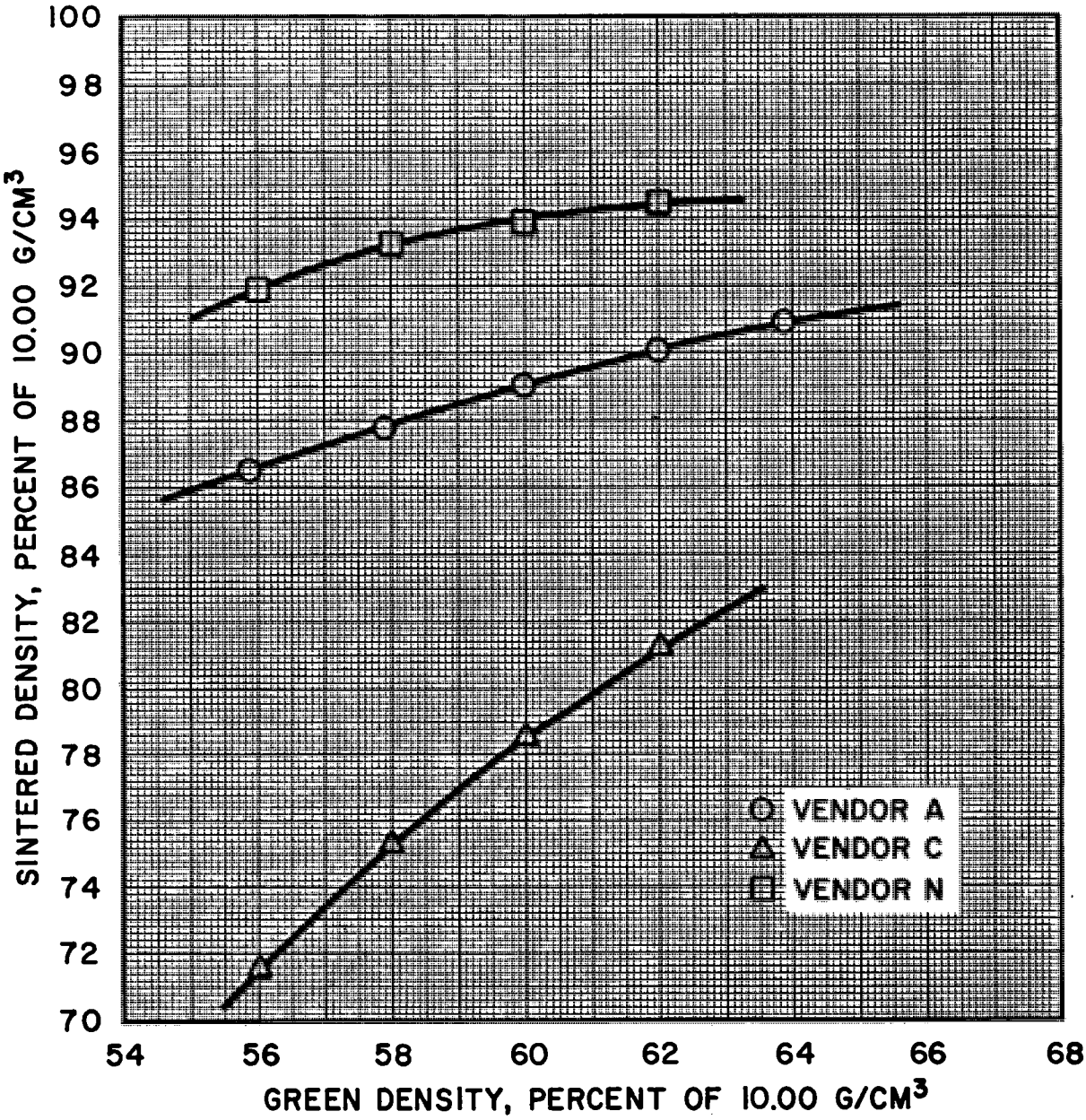


FIGURE 14

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH ONE-PASS MICRONIZED THORIA POWDER FROM VENDORS A,C AND N

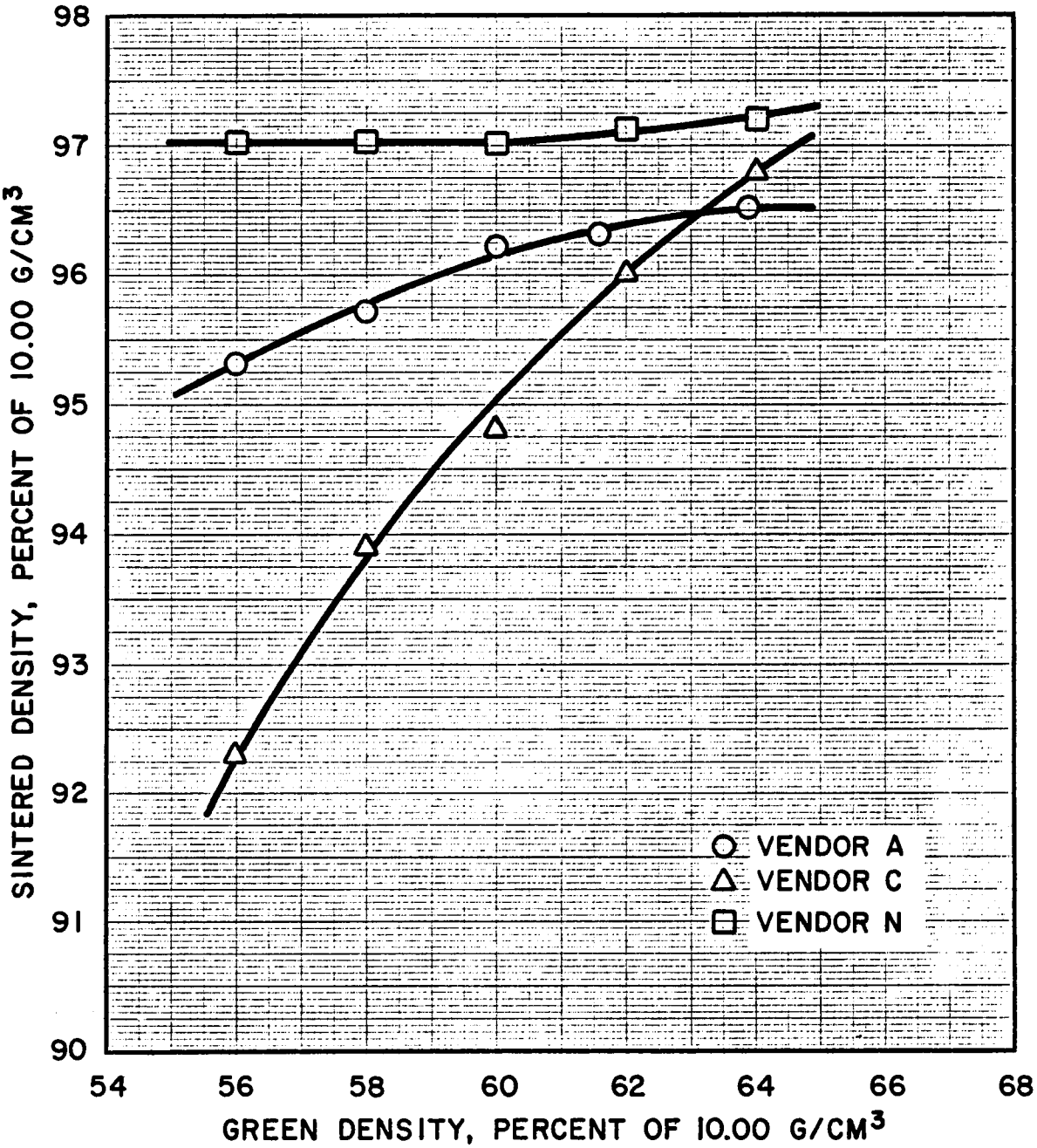


FIGURE 15

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH TWO-PASS MICRONIZED THORIA POWDER FROM VENDORS A,C AND N

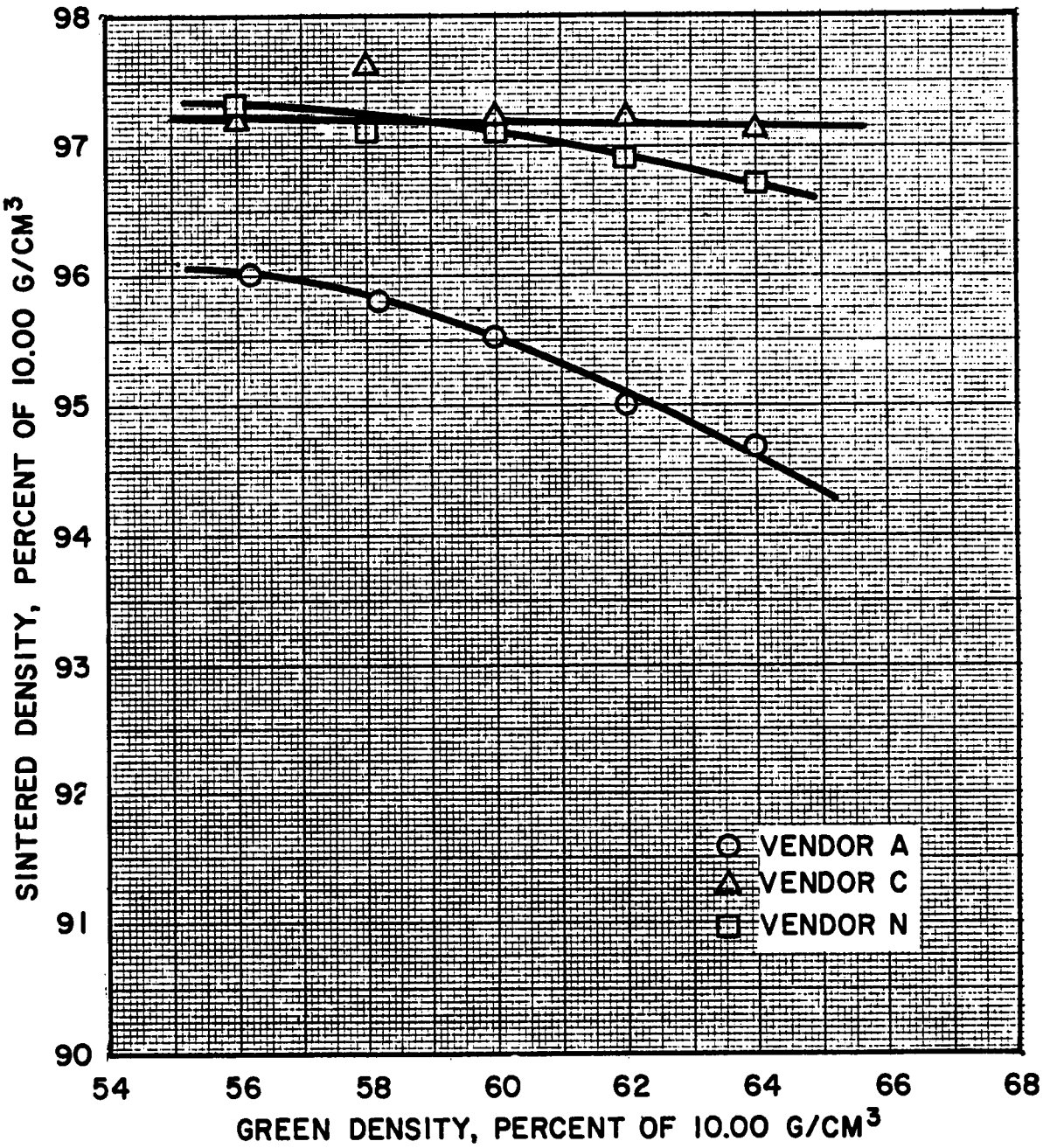


FIGURE 16

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH AS-RECEIVED, ONE-PASS MICRONIZED, AND TWO-PASS MICRONIZED VENDOR A THORIA POWDER

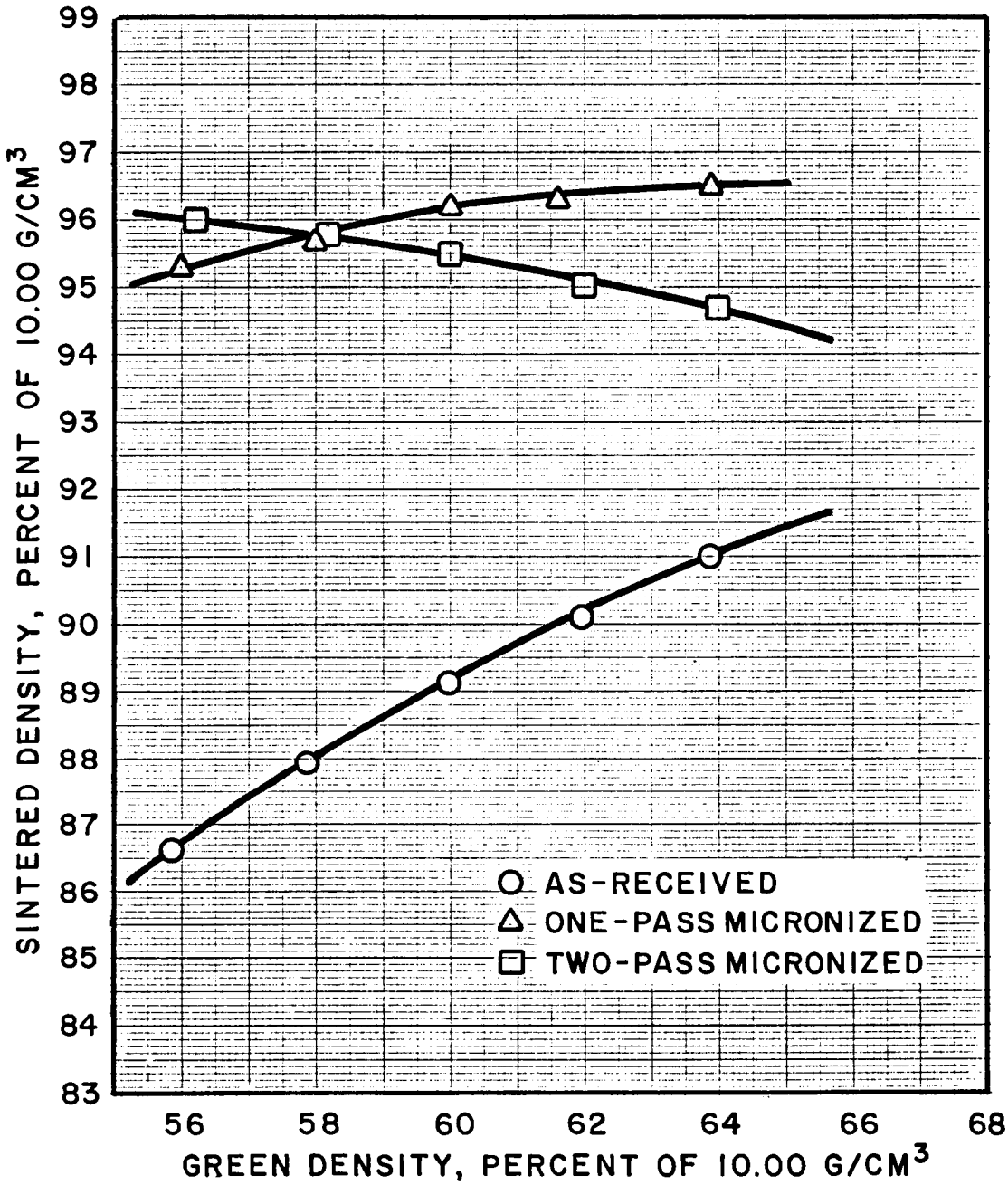


FIGURE 17
THE RELATIONSHIP BETWEEN GREEN DENSITY AND MICROSTRUCTURE FOR PELLETS
PREPARED WITH AS-RECEIVED, ONE-PASS AND TWO-PASS MICRONIZED VENDOR A THORIA POWDER

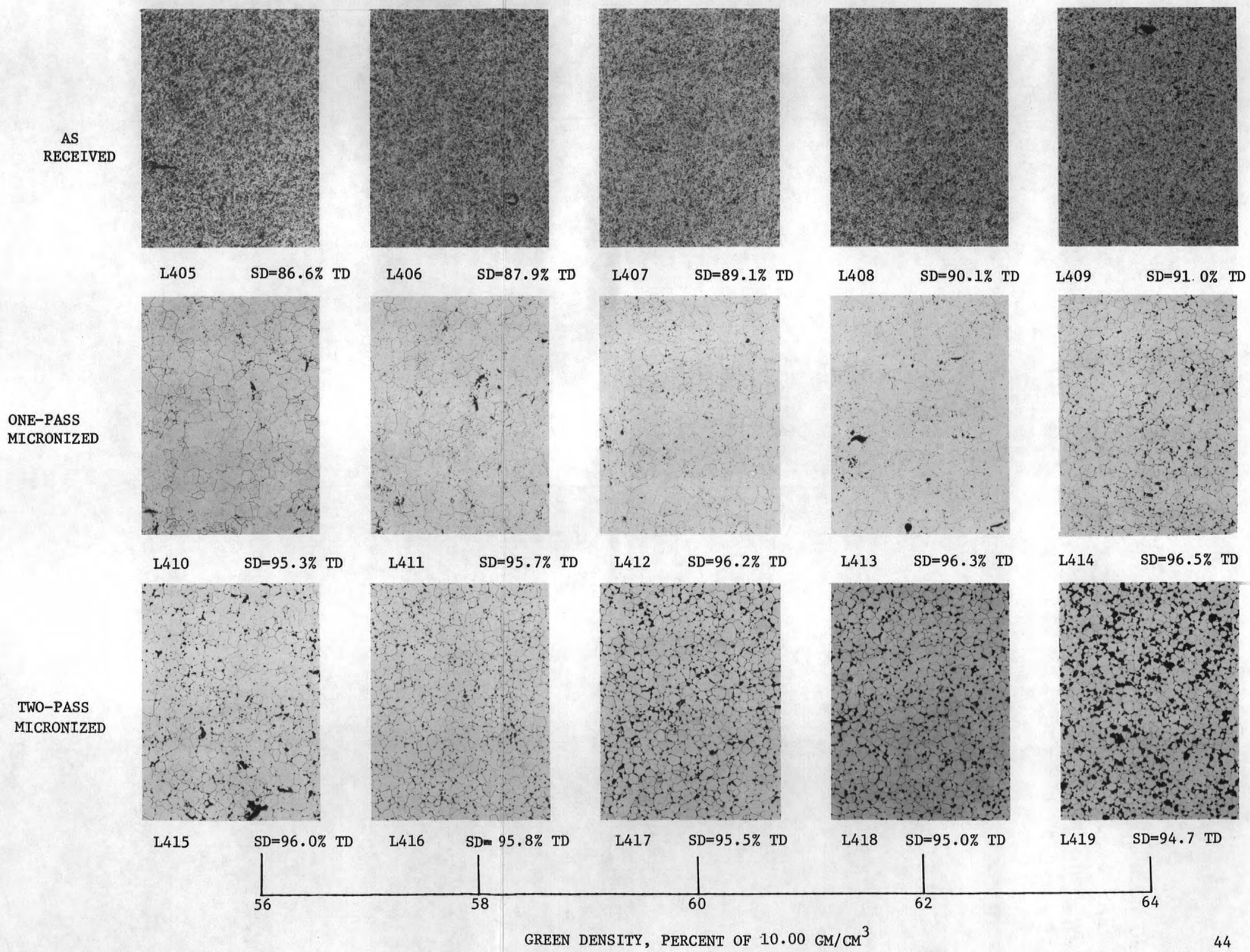


FIGURE 18

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH AS-RECEIVED, ONE-PASS MICRONIZED, AND TWO-PASS MICRONIZED VENDOR C THORIA POWDER

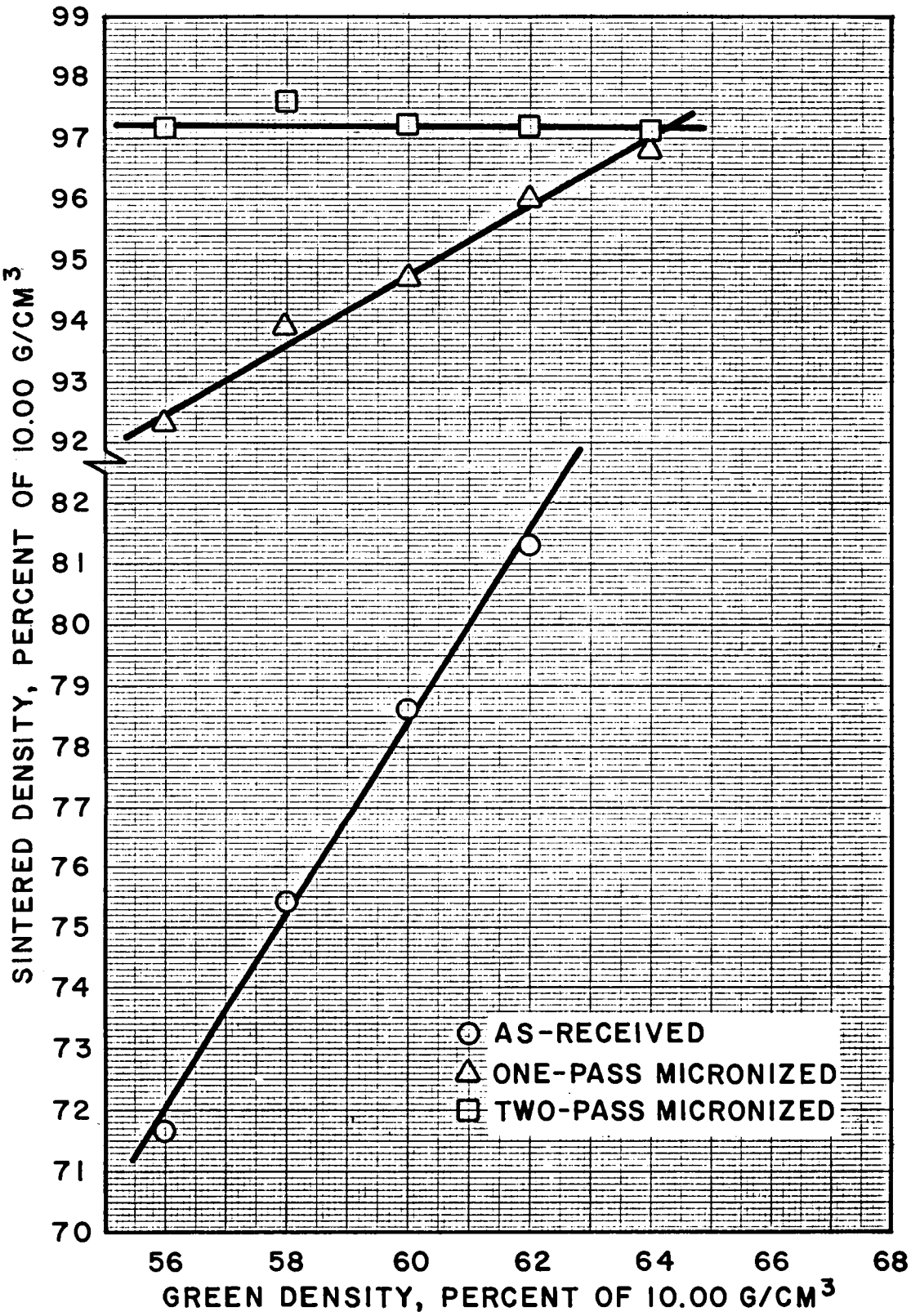


FIGURE 19
THE RELATIONSHIP BETWEEN GREEN DENSITY AND MICROSTRUCTURE FOR PELLETS
PREPARED WITH AS-RECEIVED, ONE-PASS AND TWO-PASS MICRONIZED VENDOR C THORIA POWDER

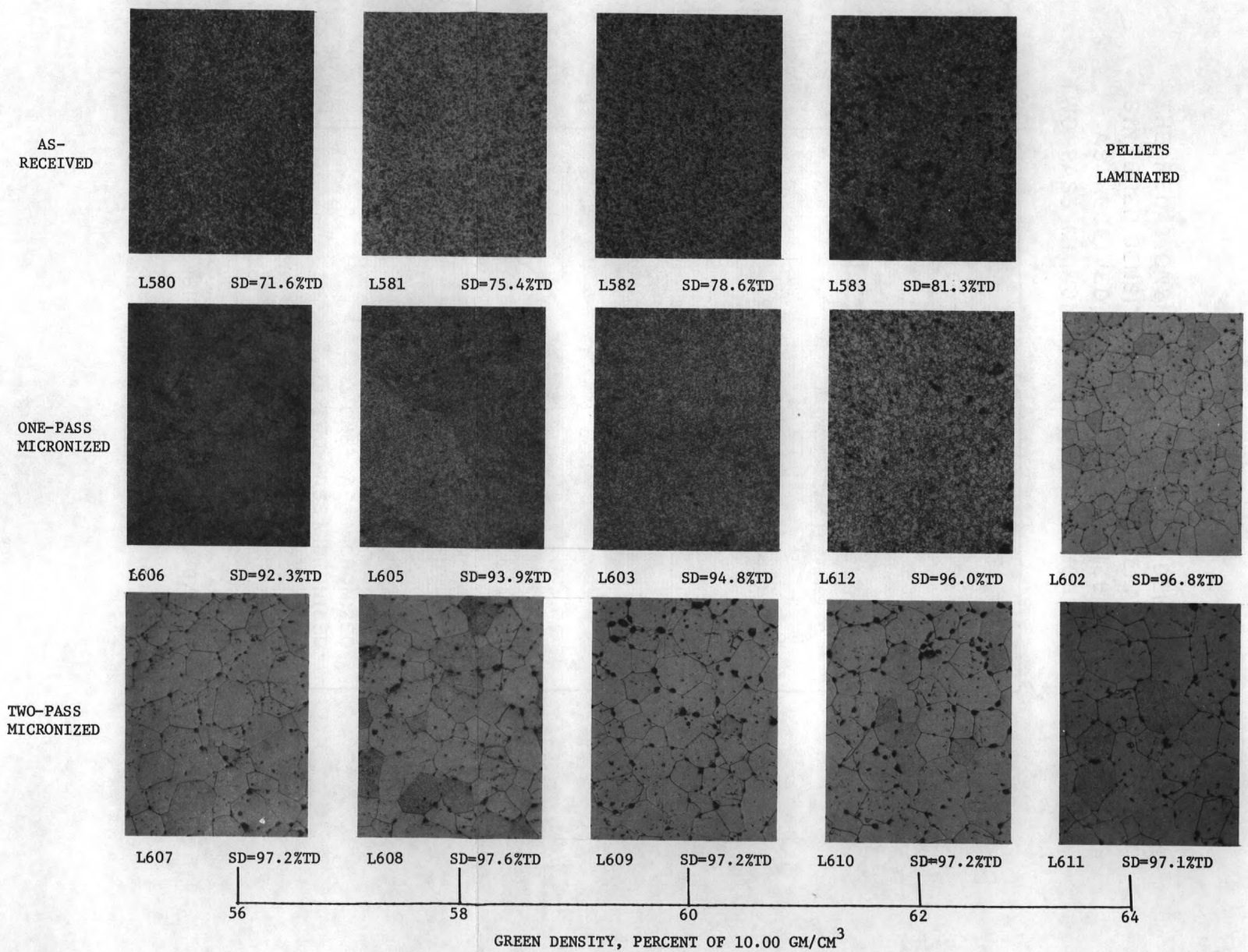
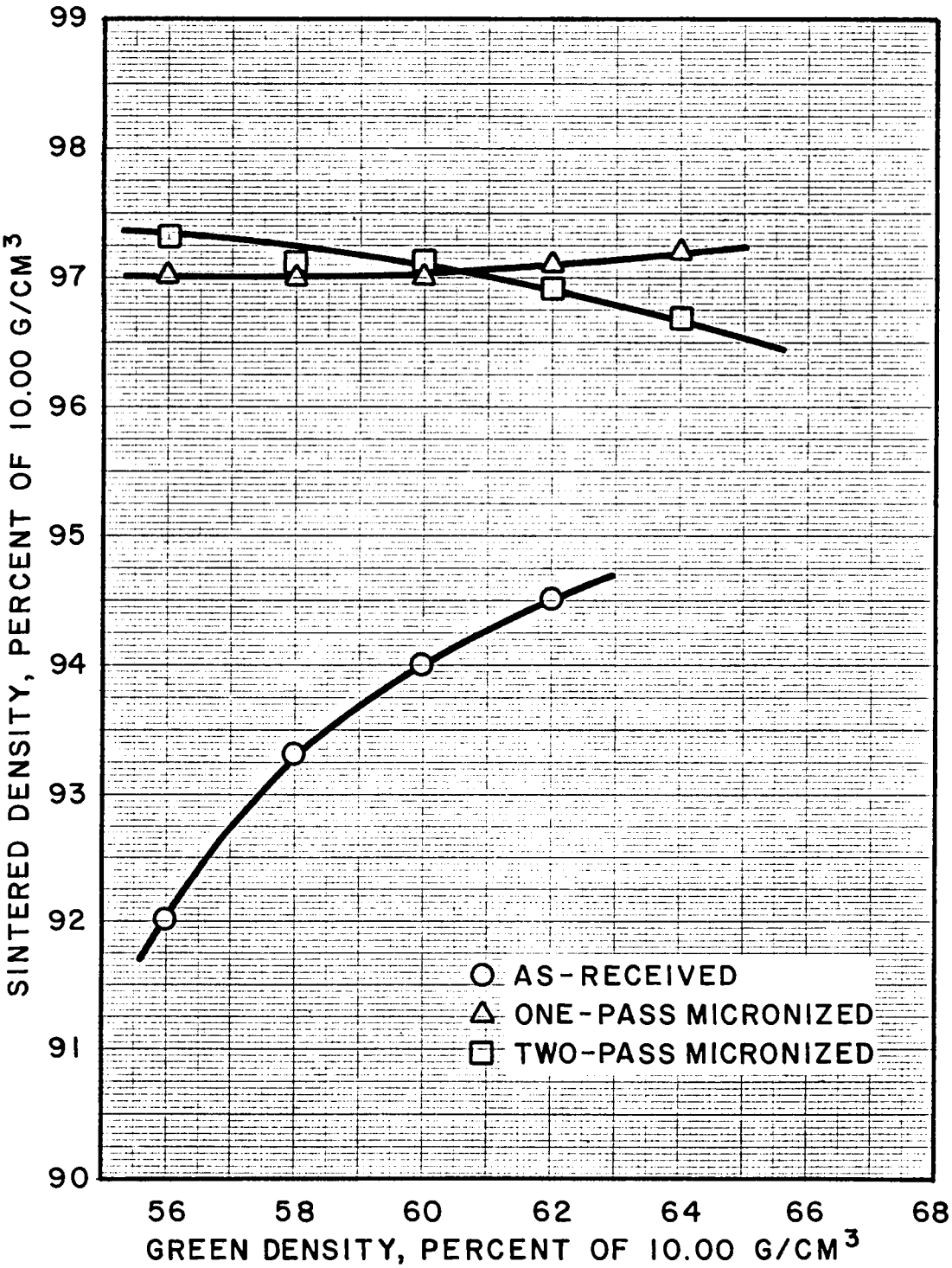


FIGURE 20

THE RELATIONSHIP BETWEEN GREEN DENSITY AND SINTERED DENSITY FOR PELLETS PREPARED WITH AS-RECEIVED, ONE-PASS MICRONIZED, AND TWO-PASS MICRONIZED VENDOR N THORIA POWDER



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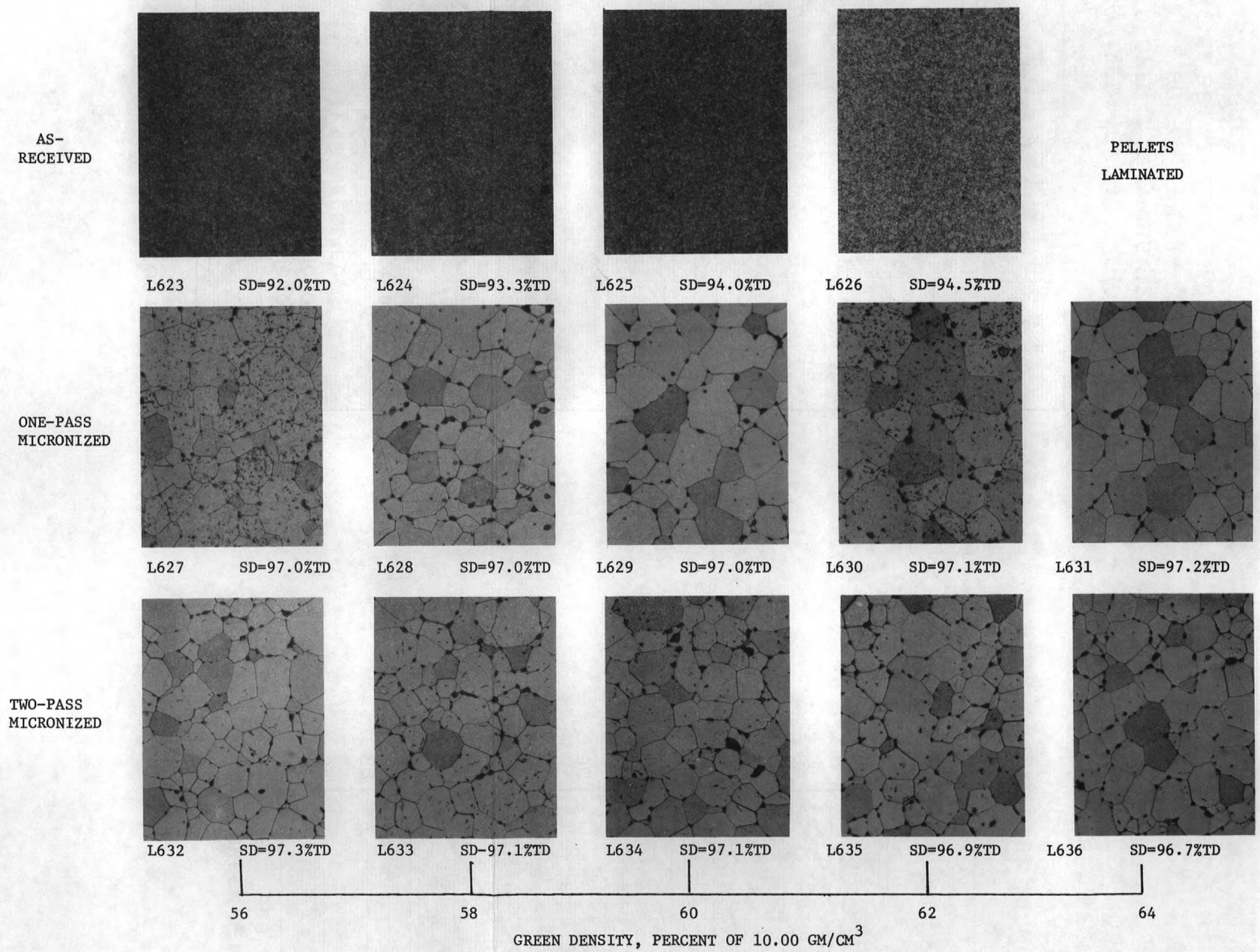
FIGURE 21
THE RELATIONSHIP BETWEEN GREEN DENSITY AND MICROSTRUCTURE FOR PELLETS
PREPARED WITH AS-RECEIVED, ONE-PASS AND TWO-PASS MICRONIZED VENDOR N THORIA POWDER