

# Attritor Milling of WC + 6% Co: Effects on Powder Characteristics and Compaction Behaviour

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This study examines the effects of attritor milling on the characteristics and bulk behaviour of fine tungsten carbide powders (initial median particle size  $\approx 1.5 \mu\text{m}$ ) blended with 6 wt. % cobalt (mps  $\approx 1.5 \mu\text{m}$ ). Experiments are performed in order to (1) develop a process model relating the specific energy input to the milled median particle size; (2) examine the effect that changes in milling variables have on the specific energy - median particle size relationship; (3) observe the effects of variation in the initial particle size distribution on the as-milled particle size distribution, the compaction characteristics of the powder, and the shrinkage which will occur during sintering.

The process model is based on Charles' equation;

$$E = A (d^{-\alpha} - d_0^{-\alpha})$$

in which  $\bar{E}$  = the specific energy consumed in milling,  $d$  and  $d_0$  = the initial and milled median particle sizes respectively, and  $A$  and  $\alpha$  are constants. Computer curve fitting techniques are employed to determine the values of the coefficient and exponent in the above equation. The resulting model predicts the experimental data within about  $\pm 10\%$  over a significant range of  $d$  and  $\bar{E}$  values. The apparent density and compactibility of the attritor milled powders are observed to be very sensitive to the milled particle size distribution.

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The attritor developed by A Szegvari in 1956 (1) has found a place in the cemented carbide industry for the milling and blending of the carbide and binder powders. Faster than the conventional ball mill, the attritor offers increased productivity by shortening slurry retention times, thus increasing mill throughput.

Although the attritor has proved to be a faster comminution method, the transition from ball milling to attritor milling has not been without its problems. Because of the greatly increased milling rate, the prediction of a particle size based on milling time (the method used in ball milling) has become difficult. Also, dynamic milling variables such as agitator and ball wear change rapidly enough to significantly affect the milling rate, making a particle size distribution prediction based on time even more difficult. Finally, as the milled particle size distribution is one of the major factors determining the properties of the finished product, any changes in the as-milled particle size distribution brought about by attritor milling must be determined, and their effect on the compaction and sintering behaviour quantified.

With these problems in mind, this project was begun with the following objectives:

- To develop a process model for the attritor milling of WC + 6 wt. % Co powder mixtures to a specific final median WC particle size based on the integrated energy input to the mill.
- To examine attritor milling efficiency as influenced by variations in selected milling parameters.
- To assess the changes in bulk behaviour of attritor milled WC + 6wt.% Co mixtures related to changes in milled median particle size and size distribution.

Specific attention was focused on apparent density and compactibility since these factors are critical in the ultimate control of sintered shrinkage.

### EXPERIMENTAL PROCEDURE

The experimental work performed for this study was in three major areas; milling, compaction, and powder characterization. The section describing milling covers the slurry preparation, operation and monitoring of the attritor mill, and slurry sampling techniques. Powder characterization includes the methods used to determine the particle size distribution, the median particle size, and apparent density of the powders both before and after milling. The preparation of a compact and the determination of its green density is discussed in the section dealing with compaction.

### Materials

In each of the milling experiments performed, the materials used were tungsten carbide powder, cobalt powder, and technical grade acetone. Two grades of tungsten carbide powders were supplied by General Electric Co., Carbology Systems Department, each representing varied size distributions (Table 1). These powders possess distributions which can be generally described as either narrow or broad with a given median particle size. Median particle sizes (MPS) were determined throughout this work with a Fisher Sub-sieve Sizer. A single lot of cobalt powder, having a median particle size of  $1.50 \mu\text{m}$  was used for all of the experiments.



Particle Size Distribution (wt%)

Powder	Size Interval (μm)										MPS (μm)
	0-1	1-2	2-3	3-4	4-5	5-6	6-7	7-8	8-10	10-20	Bal.
A	20	49	19	5	2	-	-	-	-	-	5
B	5	12	16	18	14	12	7	6	6	3	1

TABLE 1 Characteristics of tungsten carbide powders before milling.

**Attritor Milling**

A Union Process Inc. model 1-ST attritor was used for all of the milling experiments. This machine is a stirred ball mill of 9.5 litre (2 1/2 gallon) capacity, capable of batch operation (Fig. 1). During milling, a motor driven agitator shaft imparts a stirring action to the grinding medium and the slurry creating stresses sufficient for particle fracture. In all experiments, the grinding medium consisted of 0.64 cm (1/4 inch) diameter tungsten carbide balls. A transducer mounted on the agitator shaft allowed the monitoring of rotational velocity in revolutions per minute and torque in inch pounds. Both torque and velocity were observed throughout the milling cycle in order to determine the energy input to the mill.

The specific energy input was calculated using the equation:

$$\bar{E} = \frac{E}{M} = 1.18 \times 10^{-5} \frac{T N t}{M}$$

where;  $\bar{E}$  = the specific energy consumption for the material being ground (kwh/kg),  $E$  = the energy provided to the material being milled (kwh),  $M$  = the mass of solids being ground (kg),  $T$  = torque (in.lb.),  $N$  = rotational velocity (rpm), and  $t$  = the milling time (hr). The unit has a variable speed drive which permits the mill to be run at speeds ranging from 65 to 500 rpm. For all of the work undertaken in this study the rotational velocity was held constant at 330 rpm.

The calculation of the amounts of WC, Co, and acetone needed for an experimental run was generally based on the total recommended slurry volume, the slurry solids

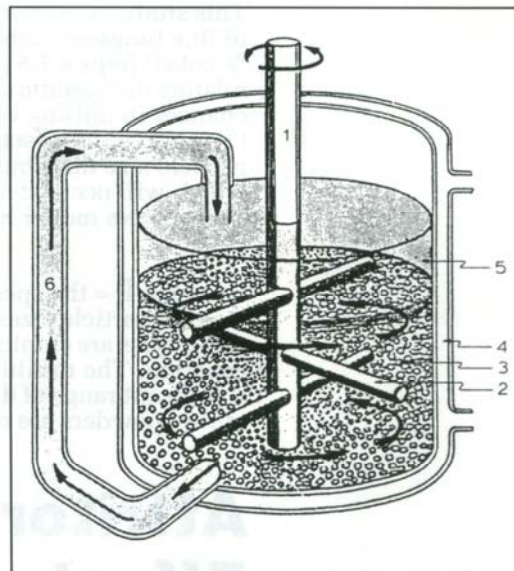


FIG. 1 Schematic drawing of the grinding chamber of an attritor. Components: 1. main agitator shaft, 2. agitator arms, 3. grinding medium, 4. water cooled jacket, 5. slurry, 6. slurry recirculating system.

concentration, and the alloy composition desired. The standard operating volume for the attritor is four litres. The amount of acetone required and the total solids volume for a given slurry solids concentration were calculated based on this slurry volume, using the density of acetone and a rule of mixtures density for the WC-Co blend desired. The only exceptions to this were experiments involving the variation of the slurry solids concentration, where the solids volume was maintained constant at 500 cm<sup>3</sup>, and the total slurry volume was allowed to vary.

The milling work consisted of seven experimental runs which were designed to examine the following:

- The effect of changes in agitator arm length on milling behaviour.
- The effects of variation of slurry solids concentration on milling behaviour.
- The influence of a large variation in the pre-milled particle size distribution on the milling behaviour.

In addition, the data generated from these runs were used to formulate and test a process model relating the specific energy input to the milled particle size. A listing of the milling parameters for this study is given in Table 2. Several of these parameters were kept constant throughout the experimentation. All runs were made with a tungsten carbide - 6 wt.% cobalt powder mix to stimulate a common grade of commercial material. A slurry volume of 3.8 litres was maintained throughout the work with the exception of experiments in which solids concentrations were varied from 10 to 30 vol. % (runs 3, 4, and 5). In these runs, a solids volume of 500 cm<sup>3</sup> was chosen because at this value, the least dense slurry occupied the maximum volume which could be held in the attritor mill. As a result, the slurry volumes varied from 1.67 litres to 5.00 litres for this set of experiments.

After preparing a slurry as previously described, the attritor mill started at a speed of 330 rpm. As the run progressed, torque, rotational velocity, and slurry density were monitored at 5, 10, 15, and

Run No.	Slurry Solids Conc.	Slurry Volume	Solids Volume	Milling Time	Arm Length	Slurry Make-up
1	85 wt. %	3.87 l	885 cm <sup>3</sup>	6 hrs.	7 1/2 in.	12,477 g. WC (A) 796 g. Co 2.980 l. acetone
2	85 wt. %	3.87 l	885 cm <sup>3</sup>	6 hrs.	6 1/2 in.	12,477 g. WC (A) 796 g. Co 2.980 l. acetone
3	10 vol. % (68 wt. %)	5.00 l	500 cm <sup>3</sup>	6 hrs.	6 1/2 in.	7,050 g. WC (A) 450 g. Co 4.500 l. acetone
4	20 vol. % (83 wt. %)	2.50 l	500 cm <sup>3</sup>	6 hrs.	6 1/2 in.	7,050 g. WC (A) 450 g. Co 2.000 l. acetone
5	30 vol. % (89 wt. %)	1.67 l	500 cm <sup>3</sup>	6 hrs.	6 1/2 in.	7,050 g. WC (A) 450 g. Co 1.167 l. acetone
6	85 wt. %	3.87 l	885 cm <sup>3</sup>	10 hrs.	7 1/2 in.	12,477 g. WC (A) 796 g. Co 2.980 l. acetone
7	85 wt. %	3.87 l	885 cm <sup>3</sup>	6 hrs.	6 1/2 in.	12,477 g. WC (B) 796 g. Co 2.980 l. acetone

Note: The initial ball charge consisted of 50 kg of 0.64 cm. (1/4") WC balls. A weight loss of approximately three percent occurred throughout the milling study. No new grinding balls were added to compensate for this loss.

TABLE 2 Parameters controlled in attritor milling runs.



30 minutes, and at thirty minute intervals thereafter. The slurry density was determined by drawing a 70 - 90 ml sample from the recirculation system, into a 100 ml graduated cylinder which was then weighed on a balance tared for the cylinder. Acetone additions were made if significant evaporation had occurred. The slurry samples removed at 1/4, 1/2, 1 hour, and each subsequent hour were retained for future particle size analysis. All other samples were returned to the mill after the density was measured. The slurry samples were allowed to dry by evaporation. Upon completion of milling, the remaining slurry was removed from the machine, and the mill cleaned by repeated acetone rinses.

#### Powder Characterization

Milled powder samples weighing 35 grams were leached with HCl to remove the cobalt, allowing particle size analysis of the milled tungsten carbide powder alone. A previous set of experiments (2) determined that the leaching procedure used has no effect on the tungsten carbide particle size distribution measurements.

Median particle sizes were obtained using a Fisher sub-sieve sizer in accordance with ASTM standard B 330. (3) Particle size distributions were determined using a WAB turbidimeter interfaced with a Hewlett Packard HP-85 desktop computer for data acquisition and calculations. The practice specified in ASTM standard B 430 (4) was followed in performing the turbidimetric analysis.

The apparent density of the milled powders was determined with the Carney apparatus as described in MPIF standard 28 (5). This procedure is similar to that described in ASTM standard B-417 (6).

#### Compaction

For the compaction studies, several fifty gram samples of milled powder were taken from each of four milling runs. In three of these runs, type A powder was milled, while the fourth involved type B powder. The milling times represented ranged from 15 to 600 minutes, having median particle sizes which varied from 2.90  $\mu\text{m}$  to 0.4  $\mu\text{m}$ . Prior to compaction, each powder sample was laboratory rod milled for 15 minutes to break up the cake formed upon drying.

Fifteen gram samples of the deagglomerated powders

were compacted using a floating action, 1.80 cm (0.707 in.) square die at a compaction pressure of 207 MPa (15 tsi). Zinc stearate was used as a die wall lubricant. Compaction speed was not controlled, however full loading of the die typically took about 30 seconds. Following compaction, mass and volume measurements were made to calculate green density.

## RESULTS AND DISCUSSION

The results of this study are presented in three parts. First, the effects of changes in selected milling variables on the milling kinetics and the specific energy - median particle size relationship are examined. Secondly, the development and testing of a process model relating the specific energy input to the median particle size is discussed. Finally, the effects of changes in the as-milled particle size distribution on the bulk powder properties and compactibility is examined.

#### Effects of Changes in Milling Variables on Energy Input Requirements

The effects of selected milling variables on attritor milling behaviour were examined in order to:

- Determine how their variation would change the milling efficiency of the system.
- Observe the effects of these parameters on the rate of particle size reduction.
- Gather data which would provide the information necessary for the formulation of a mathematical process model relating the energy input to the median particle size.
- Discern the effects of changes in the pre-milled distribution on the bulk behaviour and compactibility of the milled powders.

**Agitator Arm Length:** The agitator arm is that part of the attritor which extends perpendicularly from the motor

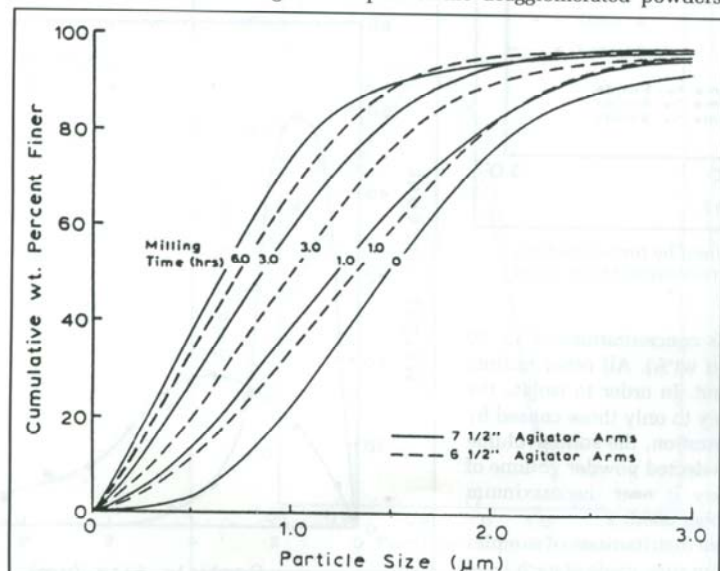


FIG. 2 A comparison of particle size distributions, as determined by turbidimetry, at 1, 3 and 6 hours of milling for two agitator arm lengths. The data shown were taken from Runs 1 and 2, details of which are listed in Table 2.

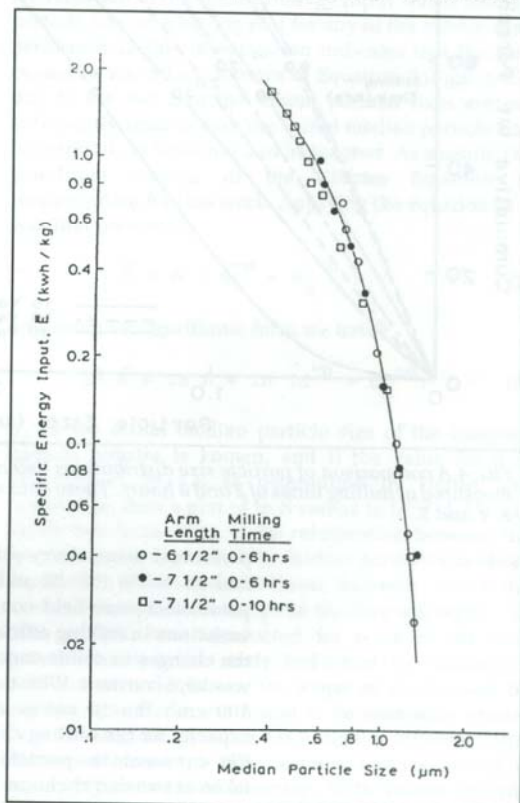


FIG. 3 Specific energy input vs. median particle size for attritor runs using agitator arm lengths of 16.5 and 19 cm (6 1/2 and 7 1/2 inches). The data shown represents samples taken at intermediate milling times during Runs 1, 2, and 6, details of which are listed in Table 2.



driven main shaft, providing the stirring motion in the grinding chamber (Fig. 1). At the outset of the experimentation, agitator arms of 16.5 cm (6 1/2 in.) and 19 cm (7 1/2 in.) in length were available. Two six hour milling runs were made, one with each agitator configuration. In addition, a ten hour run was undertaken using the longer agitator arms to observe the effects of extended milling times.

An examination of the particle size distributions shown in Fig. 2 reveals that milling occurred more rapidly with the longer agitator arms. This increase in the milling rate was accompanied by an increase in torque of approximately 70 % throughout the run. Thus, the specific energy input per unit time was greater in the run using the longer agitator arms.

Analysis of the relationship between the specific energy input and median particle size (Fig. 3), showed that the data from all three runs described a single curve. This would indicate that, although the rate of particle size reduction and the rate of energy input were increased with the longer arms, an equal amount of energy was required to achieve a given median particle size regardless of the agitator configuration. Assuming that agitator wear can be approximated by the gradual shortening of the agitator arms, this wear would appear to have no effect on the milling efficiency from an energy input standpoint.

**Variation of Slurry Solids Concentration:** To investigate the effect that changes in the slurry solids concentration have on the milling efficiency, three experiments were

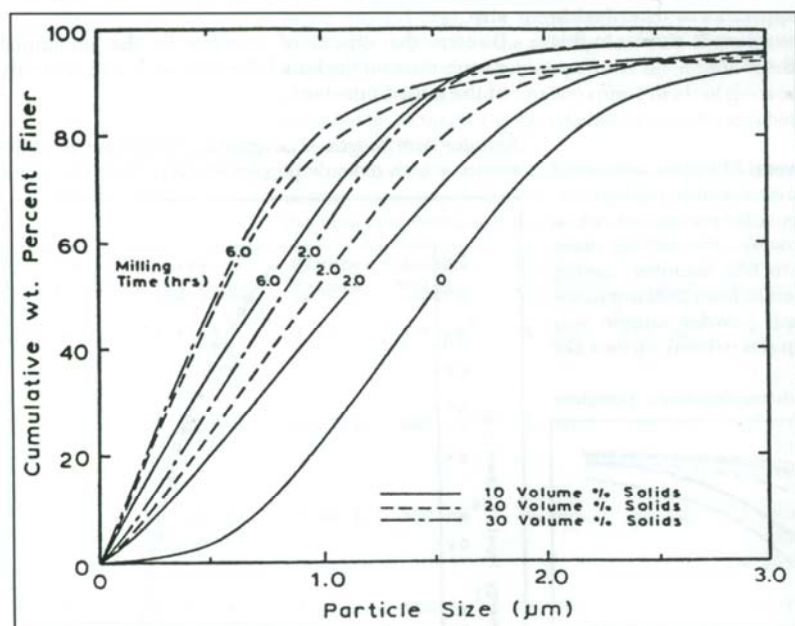


FIG. 4 A comparison of particle size distributions determined by turbidimetry, measured at milling times of 2 and 6 hours. These data were obtained from Runs 3, 4 and 5.

performed using slurry solids concentrations of 10, 20 and 30 vol/% (68, 83 and 89 wt%). All other milling parameters were held constant. In order to isolate the variations in milling efficiency to only those caused by the changes in solids concentration, the solids volume was kept constant. With the selected powder volume of 500 cm<sup>3</sup>, the 10 vol/% slurry is near the maximum capacity for the milling chamber used.

Fig. 4 presents the particle size distributions of samples taken at two and six hours from runs made at each level of solids concentration. The curves show an increasing rate of size reduction with increasing solids concentration.

Inspection of the specific energy - median particle size

relationship shown in Fig. 5 suggests that the data from all three runs may describe a single curve. However, the scatter in the data is greater than was observed in the experiments comparing agitator configurations.

**Variation of the Initial Particle Size Distribution:** In order to study the effects that changes in the pre-milled particle size distribution have on the milled particle size distribution and the specific energy - median particle size relationship, the relatively coarse (initial mps = 3.60 μm) type B powder was milled and the milling data compared to a milled type A powder (initial mps = 1.47 μm). In addition to the difference in median size, the powders display a significant difference in particle size

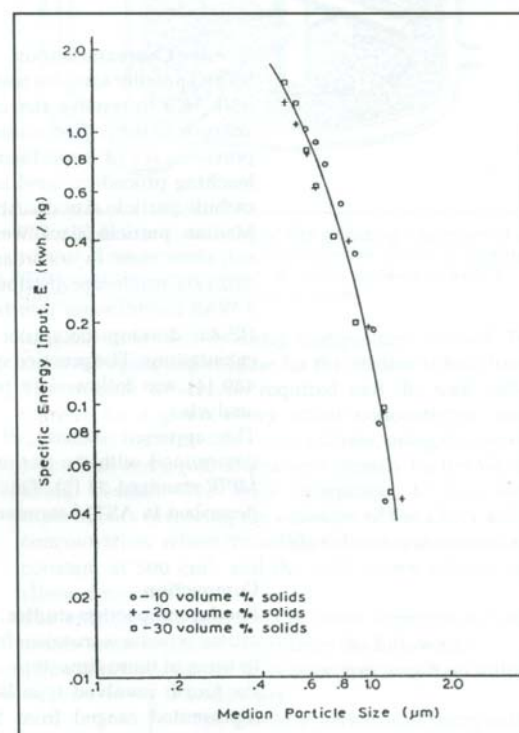


FIG. 5 Specific energy input vs. median particle size for three attritor milling runs at varying WC + 6% Co slurry solids concentrations. The initial solids volume was kept constant at 500 cm<sup>3</sup>.

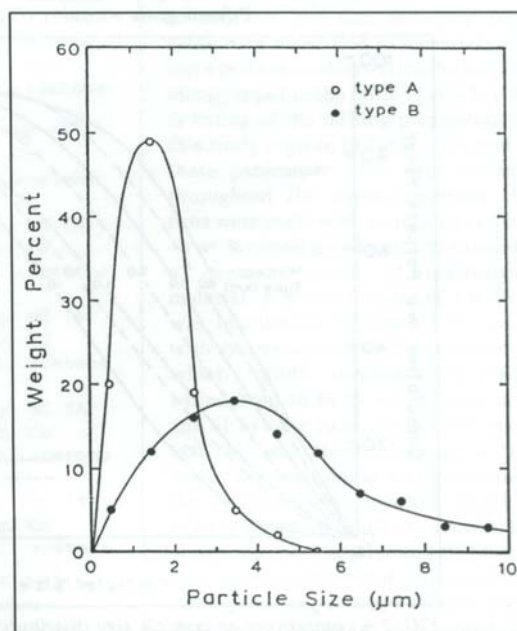


FIG. 6 Particle size distributions of the type A and type B powders prior to milling.



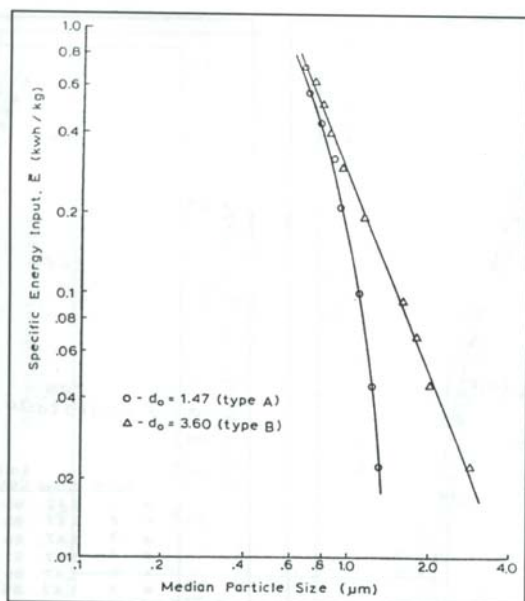


FIG. 7 Specific energy input vs. median particle size for milled type A and B powders. Samples were taken at intermediate milling times from runs 1 and 7.

distributions, the type B powder having a broad distribution, while the type A powder possesses a narrow distribution as shown in Fig. 6.

Noting the changes in the median particle sizes these powders with continued milling (Fig. 7), it can be seen that as energy input (and therefore milling time) is increased, the curves describing the milling data for both runs tend to converge.

A comparison of the size distribution of the type B powder after six hours of milling with a milled type A powder having the same median particle size (after two hours of milling) is shown in Fig. 8. While the type B powder has a slightly greater percentage of particles in the 0 - 0.5 μm range, the distributions are very similar. This clearly suggests that, with sufficient milling, powders with quite different initial distributions will approach similar distributions at the same median particle size.

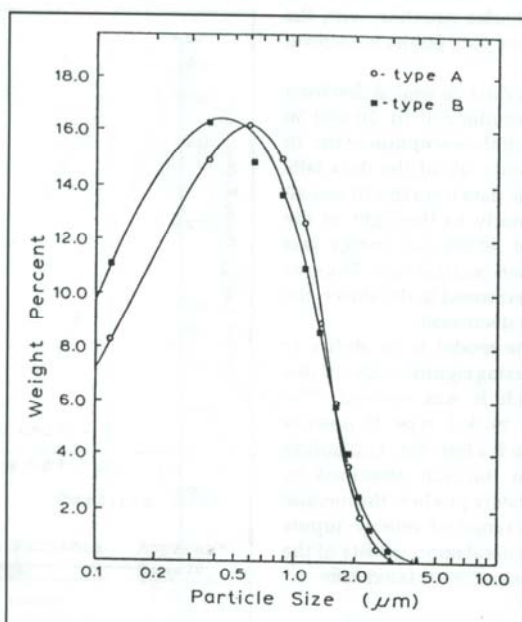


FIG. 8 Particle size distributions of type A and B powders milled to a median particle size of 0.75 μm. Samples represented here were taken after milling the type A (run 5) and type B (run 7) powders for 2 hrs. and 6 hrs. respectively.

### Development of a Mathematical Model Relating Specific Energy Input to Median Particle Size

A major objective of this work was to evaluate the feasibility of accurately predicting the median tungsten carbide particle size resulting from milling to a given energy input. The development of such a process model began with the Charles equation (7). In the form applicable to attritor milling, this equation can be written;

$$\bar{E} = A (d^{-\alpha} - d_0^{-\alpha}) \quad [2]$$

where,  $\bar{E}$  = the specific energy input,  $d$  = the milled median particle size,  $d_0$  = the initial median particle size,  $A$  and  $\alpha$  are constants. Often, if the milled median particle size is sufficiently smaller than the initial median particle size, the equation is simplified to the form;

$$\bar{E} = A d^{-\alpha} \quad [3]$$

Logarithmic transformation of this simplified equation yields;

$$\ln \bar{E} = \ln A - \alpha \ln d \quad [4]$$

providing a linear plot when the specific energy is compared to the median particle size on a log-log scale (power law behaviour), and allowing the graphical determination of the constant  $\alpha$ . This method has been employed by Herbst (8) and Sepulveda (9), and also by Goodson, Sheehan, and Larson (10) in previous work involving attritor milling. Herbst and Sepulveda's findings indicate that the constant  $\alpha$  has a value of 1.8 for all attritor milling (although they worked with materials other than tungsten carbide). Goodson et. al. generally supported this value for  $\alpha$  in work associated with coarser grades of tungsten carbide than were employed in the present study.

Examination of the specific energy input versus median particle size on a log-log plot for any of the attritor runs performed in this investigation indicates that the data cannot be described in terms of Equation 3 (Figs. 3,5,7) due to the fact that the milled particle sizes are not sufficiently smaller than the initial median particle size to permit  $d_0$  in Equation 2 to be ignored. As a result, the simplified version of the Charles Equation is inappropriate for this work. Applying the equation as it was first presented;

$$\bar{E} = A (d^{-\alpha} - d_0^{-\alpha}) \quad [2]$$

and using its logarithmic form we have;

$$\ln \bar{E} = \ln A + \ln (d^{-\alpha} - d_0^{-\alpha}) \quad [5]$$

Since the initial median particle size of the tungsten carbide powder is known, and if the value for  $\alpha$  is assumed to be 1.8 as determined by Herbst and Sepulveda, then a plot of  $\ln \bar{E}$  versus  $\ln (d^{-1.8} - d_0^{-1.8})$  will verify two facts. First, if the relationship between the specific energy input and the median particle size obeys Equation 2, the data must be linear. Secondly, if 1.8 is the correct value for  $\alpha$ , the slope of the line will equal 1.0. This analysis was conducted for some of the data generated early in this study, and while the relationship was shown to be linear, the slope as determined by regression analysis was found to be somewhat greater than 1.0, suggesting that  $\alpha$  has a slightly different value than the assumed 1.8. The constants  $A$  and  $\alpha$  can still be accurately determined however. With known specific energy values, computer curve fitting techniques can be employed to vary the value of  $\alpha$  within the variable  $\ln (d^{-\alpha} - d_0^{-\alpha})$ , until a slope of 1.0 is achieved and accurate values for  $A$  and  $\alpha$  found. This method of analysis was



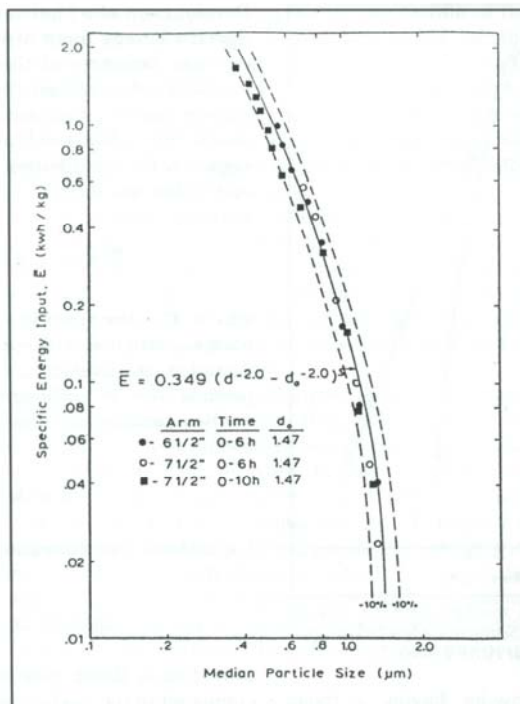


FIG. 9 A comparison of the process model to the specific energy - median particle size behaviour exhibited by three milling runs using type A powder in a 85 wt% solids slurry. The solid line represents the milled median particle size predicted by the equation for a given energy input. A deviation of  $\pm 10\%$  from the predicted particle size is represented by the broken lines.

applied to all of the data generated using type A powders. Values for the constants A and  $\alpha$  were found to be 0.349 and 2.0 respectively, providing that specific energy was measured in kilowatt hours per kilogram and median particle size is measured in micrometers. Equation 2 then becomes;

$$\bar{E} = 0.349 (d^{-2.0} - d_o^{-2.0}). \quad [6]$$

Fig. 9 compares the data generated for the milled type A powders at a slurry concentration of 85 wt.% solids to the behaviour predicted by the Charles equation with the derived constants. In all cases the data points are within  $\pm 10\%$  of the predicted values.

The data from all type A milling runs, as well as that from runs with slurry solids concentrations of 10, 20 and 30 vol.% are shown in Fig. 10. With the exception of the 10 and 20 vol.% solids experiments, all of the data falls within the  $\pm 10\%$  band. That the data from the 10 and 20 vol.% solids runs lie consistently to the right of the predicted curve indicates that additional energy was required to reach a given median particle size. Thus, as the concentration of solids is decreased in the slurry, the energy efficiency of the mill is decreased.

A more demanding test for the model is its ability to predict data from milling runs using significantly coarser powder than that from which it was derived. The information gathered for the milled type B powder provides the opportunity for such a test. Fig. 11 displays the type B milling data with the line described by Equation 6. The equation accurately predicts the median particle size over the entire range of energy inputs studied, thus supporting the general applicability of the model over the particle size ranges considered here.

#### Effects of Variations in the Milled Particle Size Distribution on Compaction Behaviour

After milled particles have been consolidated into a green compact, they are sintered to achieve full density

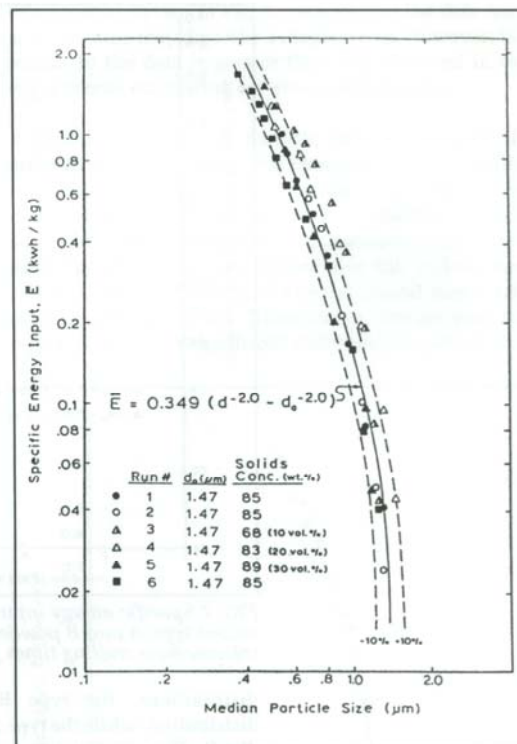


FIG. 10 A comparison of the process model to the specific energy - median particle size behaviour exhibited by all milling runs using type A powders at various slurry solids concentrations.

and develop the desired microstructure. During sintering considerable shrinkage of the compact takes place (typically around 40% by volume), the amount of shrinkage being dependent on the particle size distribution.

To better understand how the size characteristics of an attritor milled powder affect its compactibility, two experiments were undertaken. The first dealt with an examination of type A powders. Samples milled for varying amounts of time and thus representing a range of particle size distributions were tested to determine their apparent and green densities. The second part of the

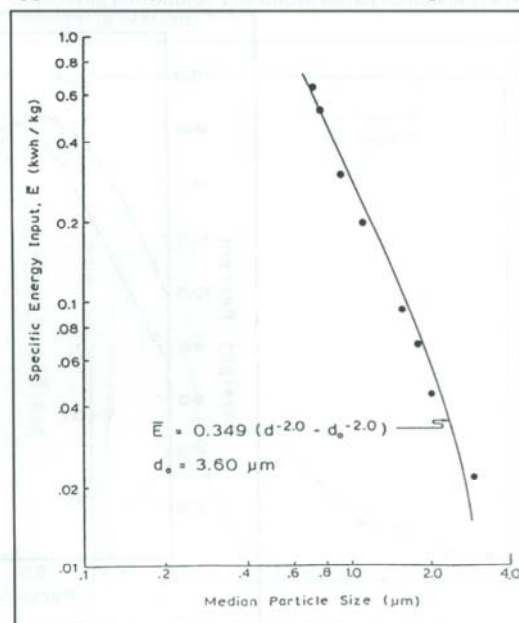


FIG. 11 A comparison of the process model to the specific energy - median particle size behaviour exhibited by the milled type B powder (run 7). The solid line represents the milled median particle size predicted by the equation for a given energy input.

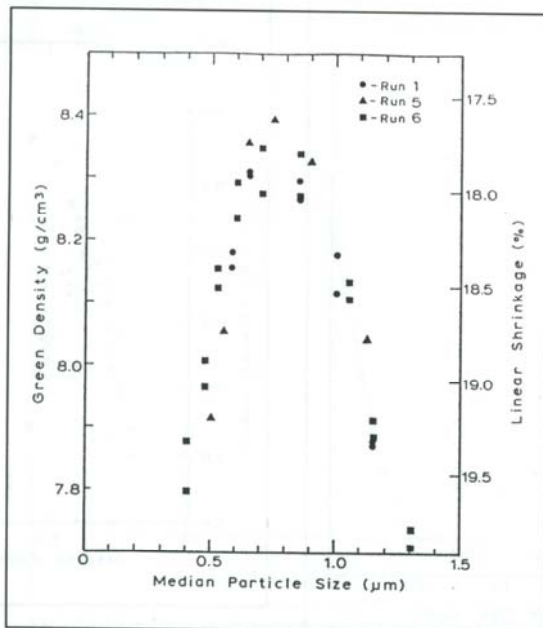


FIG. 12 Green density vs. median particle size for samples taken from three runs of type A powder and compacted at 207 MPa (15 tsi). The data represent milling times ranging from 15 to 600 minutes ( $E = 0.04$  to 1.6 kwh/kg).

study was undertaken to determine the effects that large variations in the as-milled particle size distribution brought about by the milling of a coarser type B powder would have on the compactibility of that powder. In this case the apparent and green densities of a type B powder, having undergone various amounts of milling, were measured.

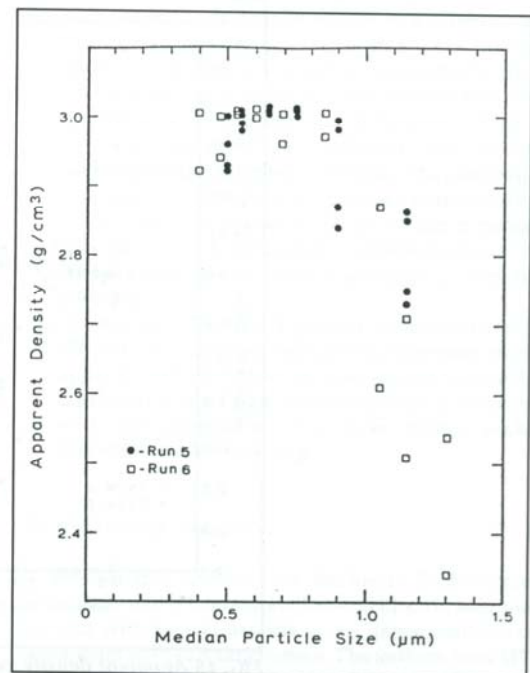


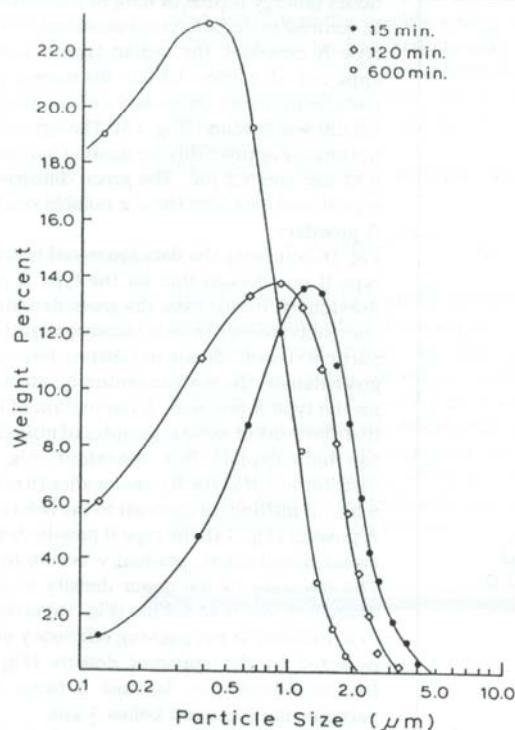
FIG. 13 Apparent density vs. median particle size for samples taken from two milling runs of a type A powder.

**Type A Powder:** Samples of milled type A WC with 6% Co were tested for apparent density, then compacted at 207 MPa (15 tsi) and the green densities calculated from mass and volume measurements. These powders possessed median particle diameters ranging from 1.30  $\mu\text{m}$  to 0.4  $\mu\text{m}$ , the results of milling times of 15 minutes to 10 hours (energy inputs of 0.04 to 1.6 kwh/kg). A plot of green density vs. median particle size is presented in Fig. 12. Green density is seen to increase as the median particle size decreases until a median diameter of 0.8  $\mu\text{m}$  to 0.9  $\mu\text{m}$  is reached. The green density then remains fairly constant at approximately 8.3  $\text{g}/\text{cm}^3$  until the median particle size becomes less than 0.65  $\mu\text{m}$ . As the median diameter is further reduced, the green densities of the compacts diminish.

Theoretical determination of the percentage of linear shrinkage expected upon sintering is also displayed in Fig. 12. The right abscissa shows the amount of linear shrinkage to be expected from the corresponding green density, assuming isotropic shrinkage and full densification occurs. It is observed that median particle sizes of 0.6  $\mu\text{m}$  to 0.9  $\mu\text{m}$  will exhibit shrinkages on the order of 17.5 - 18.0 %. However, on either side of this range shrinkage increases rapidly.

The effect of decreasing median particle size on the apparent density of a milled type A powder is shown in Fig. 13. As the median particle size becomes progressively finer, the apparent density increases rapidly until a median diameter of 0.9  $\mu\text{m}$  is reached. Subsequent decreases in the median particle size do not result in significant change in apparent density.

Although no direct observations of the green structures have been made, it appears that the initial increase in both green and apparent densities are due to the generation of fines during the initial stages of milling, allowing interstitial filling. The decrease observed in the green density at median particle sizes smaller than 0.65  $\mu\text{m}$  is most likely the result of increased die wall friction caused by the increasing concentration of very small particles, producing additional surface area in contact with the die. Particle size distributions for a type A powder milled for 15 min., 2 hrs., and 10 hrs. are shown in Fig. 14. A large increase in the concentration of fine particles (less than 0.4  $\mu\text{m}$ ) is apparent. Also, a relative broadening of the initially narrow distribution can be seen, supporting the hypothesis of interstitial filling.



Milling Time (minutes)	Median Particle Size ( $\mu\text{m}$ )	Apparent Density ( $\text{g}/\text{cm}^3$ )	Green Density ( $\text{g}/\text{cm}^3$ )
15	1.30	2.45	7.73
120	0.85	2.99	8.31
600	0.40	2.96	7.84

FIG. 14 Particle size distributions of a type A powder milled for 1/4, 2, and 10 hours. The milling parameters for this run (Run 7) are listed in Table 2, with the related bulk and green properties in the above table.



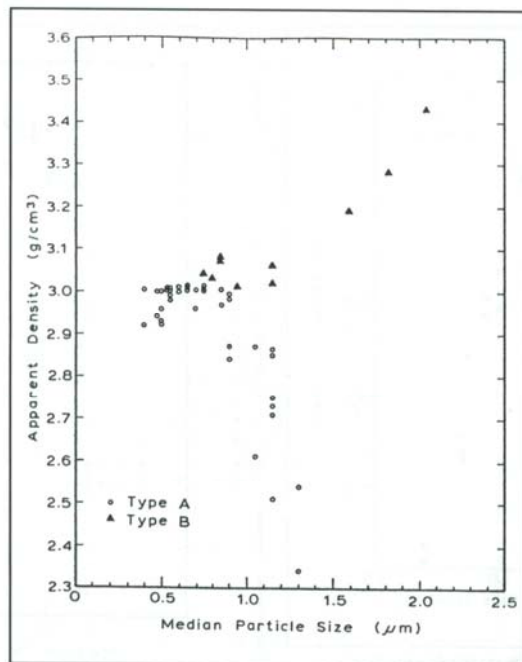


FIG. 15 Apparent density vs. median particle size for samples taken from two milling runs using type A powder (Runs 5 and 6) and one milling run using type B powder (Run 10). The samples were taken at intermediate milling times throughout the runs.

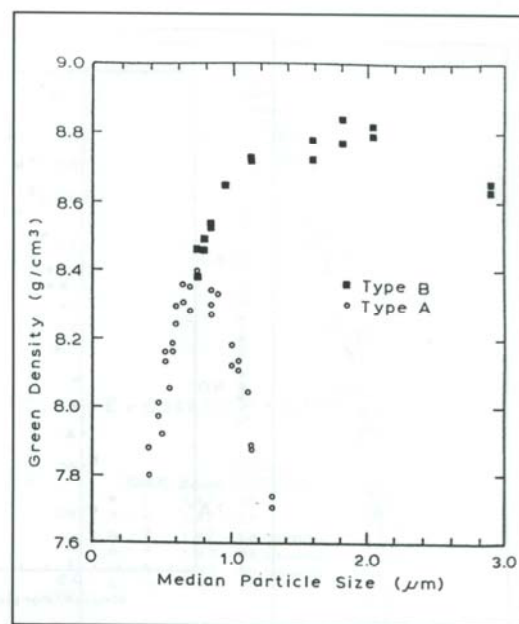
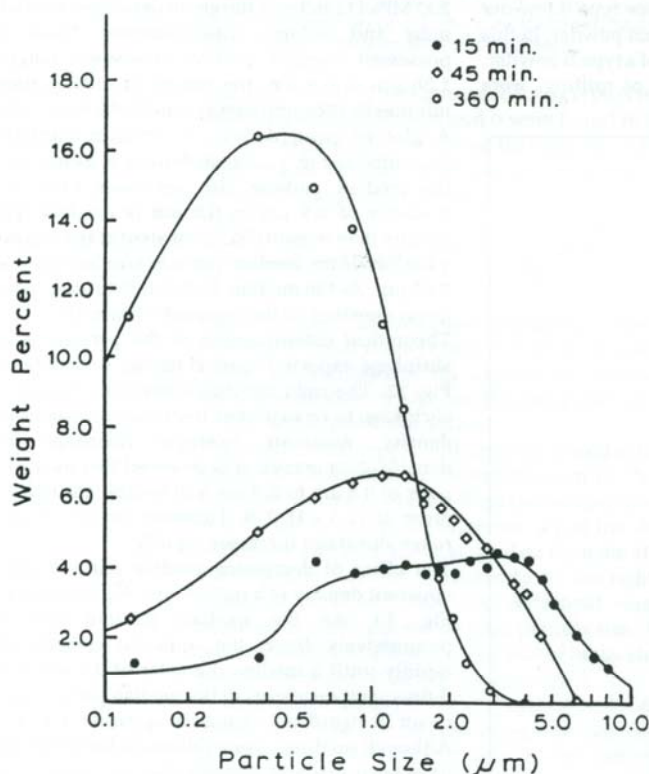


FIG. 16 Green density vs. median particle size for samples taken from three type A milling runs (Runs 1, 5, and 6) having an initial median particle size of 1.47  $\mu\text{m}$ , and one type B milling run (Run 10) having an initial median particle size of 3.60  $\mu\text{m}$ . The data represent milling times from 15 to 600 minutes.



Milling Time (minutes)	Median Particle Size ( $\mu\text{m}$ )	Apparent Density ( $\text{g}/\text{cm}^3$ )	Green Density ( $\text{g}/\text{cm}^3$ )
15	2.90	3.60	8.63
45	1.83	3.28	8.81
360	0.75	3.04	8.41

FIG. 17 Particle size distributions of a type B powder milled for 15 minutes, 45 minutes, and 6 hours. The related bulk and green properties are listed in the table above.

**Type B Powder:** The effects of changes in the milled particle size distribution on the compaction behaviour of B type powders was observed using the same methods previously described for type A powders. These samples possessed median particle sizes ranging from 2.90  $\mu\text{m}$  to 0.75  $\mu\text{m}$ , the results of milling times of 15 minutes to 6 hours (energy inputs of 0.02 to 0.65 kwh/kg).

In contrast to the behaviour observed in the studies of the type A powders, the milled type B powder displayed apparent densities which decreased as the median particle diameter decreased until a median diameter of 1.2  $\mu\text{m}$  was reached (Fig. 15). The apparent density does not change appreciably for median particle sizes between 0.75  $\mu\text{m}$  and 1.2  $\mu\text{m}$ . The green densities of the milled type B powders also show a notable contrast to the type A powders.

Fig. 16 compares the data generated from the compacted type B powders to that for the type A powders shown previously. In this case, the green densities of the type B compacts are seen to only increase slightly from a median particle size of 2.90  $\mu\text{m}$  to 1.80  $\mu\text{m}$ . Beyond this point the green density decreases eventually intersecting the curve for the type A powders. Examination of the particle size distributions of several samples of milled type B powder can help explain this behaviour. Fig. 17 shows the distribution of a type B powder after 15 min., 45 min., and 6 hrs. of milling. In contrast to the behaviour of the type A powder (Fig. 14), the type B powder begins with a very broad distribution, gradually becoming more peaked. The decrease in the green density at median particle diameters less than 1.8  $\mu\text{m}$  (Fig. 16) is thought to be due to a decrease in the packing efficiency of the powder as reflected by the apparent density (Fig. 15). Die wall friction effects may become a factor as the median particle size decreases below 1  $\mu\text{m}$ .

Comparison of the distributions of both A and B powders with a median particle diameter of 0.75  $\mu\text{m}$  (the region of intersection on the green density plot, Fig. 16), is shown in Fig. 18. The type B powder, which was milled for 6 hours, shows only a slightly higher concentration of fines than the A type powder after 2 hours of milling. The similarity in the particle size distributions clearly supports the close agreement in both the apparent density and green density of these two milled powders.



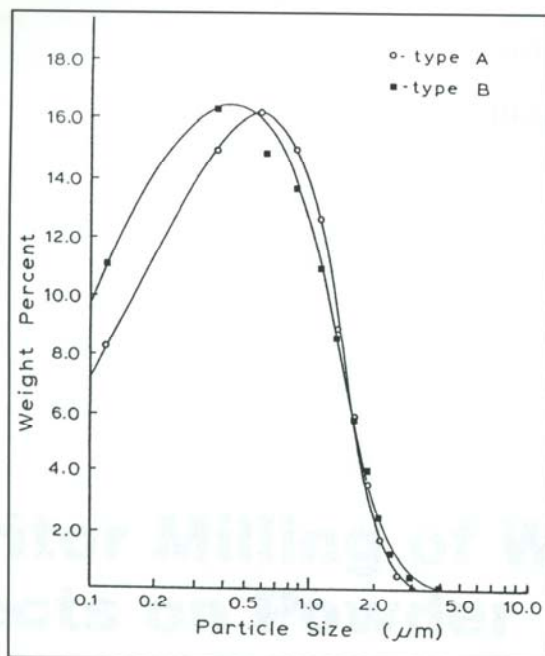


FIG. 18 Particle size distributions of type A and type B powders milled to a median particle size of 0.75 μm. Samples represented here were taken at the intermediate milling times of 2 hours and 6 hours for the A and B powder respectively.

## SUMMARY AND CONCLUSIONS

This research has led to the following observations and conclusions related to the attritor milling of WC + 6 wt. % Co mixtures;

- (1) Increases in agitator arm length resulted in an increased rate of grinding, however, the variation in agitator configuration, over the lengths examined, did not change the specific energy input required to attain a given median particle size.
- (2) At lower slurry solids concentrations, the probability that a given milling event will occur is diminished, thus reducing the milling rate and efficiency of the milling operation.
- (3) The Charles equation, with the constants derived in this study

$$\bar{E} = 0.349 (d^{-2.0} - d_0^{-2.0})$$

(for  $\bar{E}$  measured in kwh/kg and  $d$  in micrometers), accurately describes the energy input - particle size reduction relationship for fine tungsten carbide + 6 wt.% cobalt powders, milled at a solids concentration of 85 wt.%, in a small attritor. For larger scale industrial attritors the coefficient in the Charles equation is expected to change due to variation in the milling efficiency of a larger mill. A similar affect is expected with varying cobalt content due to a change in the level of energy lost to the

deformation of relatively ductile cobalt.

- (4) Both the apparent density and the green density of a type A, WC powder blended with 6% Co, increase upon milling from an initial median particle size of 1.5 micrometers to about 0.7 - 0.9 micrometers. This is attributed to the generation of significant fines in the early stages of milling, resulting in more efficient packing because of interstitial filling. The progressive decrease in green density of this powder upon milling to median particle sizes less than 0.7 μm is thought to be the result of significantly increased particle - die wall friction from excessive numbers of very fine particles.
- (5) In contrast to the type A powder, the coarser type B, WC + 6% Co blend, shows a general decrease in both apparent and green density upon milling to a median particle size of 0.75 μm. This behaviour is consistent with the generation of a more highly peaked distribution over this range.

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