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Compaction of AISI M2+10%vol. NbC Processed by High Energy Mill

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ABSTRACT A mixture of AISI M2 + 10%vol. NbC was processed in an Atrittor high energy mill to obtain a composite powder. The milling time was varied during the process. The equation $\ln\left(\frac{1}{1-D}\right) = A\sqrt{P} + B$ was used to fit the compacting experimental data from the composite milled powders. The linear correlation coefficients were better than 0.985 for all the composite powders. The parameters A and B from this equation were used to show how the composite powder characteristic changed after attritional milling.

INTRODUCTION

In a previous work the compacting equation (Eq. 1) was developed with the following conditions [1]:

1- A good fit of the experimental data;
2- Easy applicability; and
3- It allows a quantitative description of the compacting process.

$$\ln\left(\frac{1}{1-D}\right) = A\sqrt{P} + B \qquad \text{Eq.1}$$

In this equation, P is the applied pressure, D is the compact relative density, and *A* and *B* are parameters related to the powder characteristics in the compacting process.

It has been established that the parameter *A* (slope of the curve resulting from Eq. 1) provides the powder plastic deformation capacity during the compacting process. It was also postulated that the parameter *B* (interception of the curve resulting from equation A at zero pressure) expresses the density without pressure application. The value of B depends on how the system is charged and the powder characteristics such as particle size distribution and shape.

The purpose of this work is to apply Eq. 1 to composite powders produced by milling AISI M2 + 10%vol. NbC particles in a high energy mill and then to analyze the processing effects by using parameters A and B of Eq. 1.

EXPERIMENTAL PROCEDURE AND RESULTS

The materials used in the present work were AISI M2 powders with 46 µm mean particle size and NbC powders with 6 µm mean particle size. The mean particle sizes before and after processing were measured according to the ASTM E20-85.

The AISI M2 + 10%vol. NbC powder mixture was processed in a Molinex PE 075 Atrittor mill (Netzsch) under a N_2 protective atmosphere with a flow rate of 0.3 l/min. The grinding media were 52100 steel spheres of 7.5 mm diameter. The spheres to the powder mass ratio was 5:1 and the total mass of the system was 1000 g.

Samples were prepared at different milling times (1, 2.5, 5, 7.5, 12.5 and 15 hours) and compacted to obtain the compressibility curves. Eq. 1 was used to fit the compacting experimental data. Fig. 1 shows the mean particle size as a function of milling time [2].

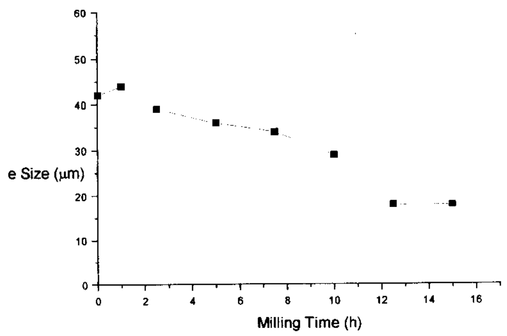

Fig. 1. Mean particle size as a function of milling time

Figs. 2, 3 and 4 show representative scanning electron micrographs of selected powders.

Fig. 2: 1h milled powder, loosed, showing lamellar morphology.

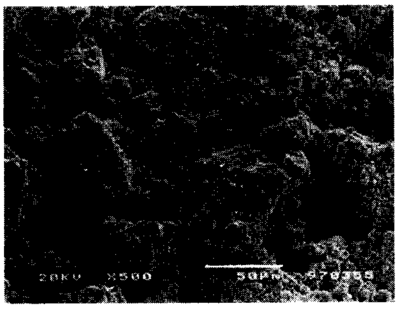

Fig. 3: 15h milled powder, loosed, showing irregular morphology.

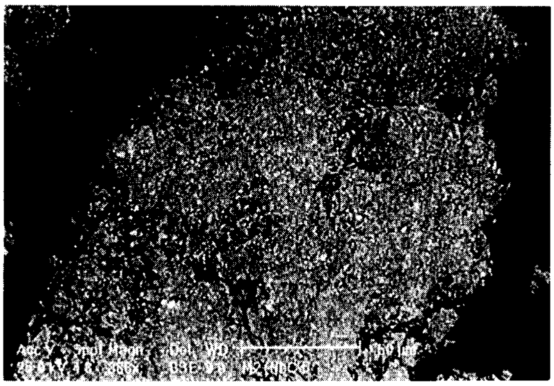

Fig. 4: 1h milled powder, metallographically prepared, showing NbC particles entrapped in an AISI M2 matrix, (backscattering).

Table 1 shows the relative geometric density of compacts as a function of applied pressure for different milling times. The results presented in this Table correspond only to the milled powders because NbC particles are already entrapped in the steel particles (Fig. 4) after 1 hour milling. Therefore, after 1 hour milling, the powder can be considered a composite.

Table 1: Relative density of compact as a function of applied pressure for different milling times

Composite M2+10%vol. NbC	Applied Pressure (MPa)				
Milling Time	260	365	474	567	655
1h		5.32	5.58	5.80	5.97
2.5h		5.20	5.41	5.61	5.76
5h	4.95	5.18	5.33	5.52	5.66
7.5h		5.15	5.29	5.41	5.50
12.5h		4.83		5.26	
15h	4.96	5.10	5.20	5.32	5.39

Data shown on Table 2 were resulted from the fitting of Eq.1 to the experimental data presented in Table 1. *R* is the linear correlation coefficient.

Table 2: Compressibility values for powder samples.

Time	Equation (1)	*R*
1h	$\ln\left(\frac{1}{(1-D)}\right)=0.0425\sqrt{P}+0.2765$	0.999
2.5h	$\ln\left(\frac{1}{1-D}\right)=0.0346\sqrt{P}+0.3821$	0.998
5h	$\ln\left(\frac{1}{(1-D)}\right)=0.0272\sqrt{P}+0.5177$	0.985
7.5h	$\ln\left(\frac{1}{1-D}\right)=0.0202\sqrt{P}+0.6454$	0.998
12.5h	$\ln\left(\frac{1}{1-D}\right)=0.0170\sqrt{P}+0.6866$	1.000
15h	$\ln\left(\frac{1}{(1-D)}\right)=0.0145\sqrt{P}+0.7176$	0.996

DISCUSSION

Table 2 shows that a good linear correlation coefficient can be reached when experimental data from composite powder is fitted by Eq. 1. This fact confirms that Eq. 1 is a suitable compacting equation as it was shown by the authors for other powders [1].

Table 2 also shows that the parameter A decreases and the parameter B increases for longer milling times. These results can be discussed based on cold welding and fracturing phenomena associated with metal work hardening and powder morphology and size changes that occur during High Energy Mill processing [2, 3].

The parameter A from Eq. 1 provides the powder densification capacity by plastic deformation in the compaction [1]. Therefore a decrease of the parameter A as the milling time goes on would be expected. There is a progressive metal work hardening in the attrition processing that is caused by the continuous impact of the grinding media to the composite powder particles.

Fig. 5 shows a plot of the parameter A versus milling time. A decrease of the work hardening rates as the milling time increases is noted. This effect would also be expected because the hardening by plastic deformation should be more difficult if the metal has been deformed previously. By fitting the experimental data shown in Fig. 5 by an exponential function, the Eq. 2 is obtained, which also shows a lower decrease of the parameter A for longer milling times, leading to a stable condition.

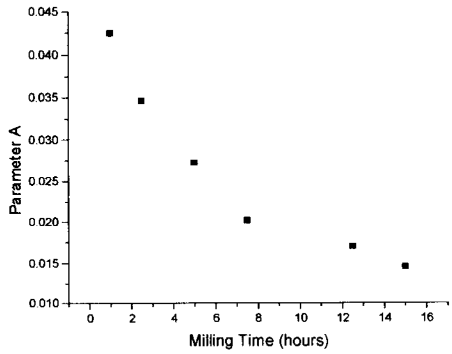

Fig. 5: Parameter A as a function of milling time.

$$A=0.046\,t^{-0.400} \quad R=-0.9815 \qquad \text{Eq. 2}$$

Besides work hardening, there are other contributions to the plastic deformation during the compacting process, such as the size and shape of composite particles which are dynamically changing.

The parameter B of Eq.1 gives an estimation of the density before starting the compaction. However, this parameter should be carefully used due to an error caused by the regression analysis. The parameter B is determined at null pressure but experimentally, pressures are very far to be null so B values have a large imprecision, and they can vary over a wide range[1, 4]. Nevertheless, the parameter B increases for longer milling times and this behavior could be explained based on powder characteristic changes.

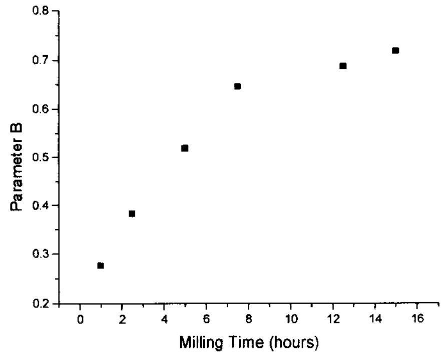

Fig 6: Parameter *B* as a function of milling time.

At the beginning of High Energy Mill processing the powder assumes a lamellar morphology (Fig. 2) changing to an irregular one as the processing goes on (Fig. 3). Meanwhile, the mean particle size decreases causing a change in the powder packing and leading to an increase of *B*, as shown in Fig. 6. If the data in Fig. 6 are fitted by an exponential function, the Eq. 3 is originated. The parameter B also tends to a stable value.

$$B = 0.281\, t^{0.365} \quad R = 0.9901 \qquad (3)$$

In Eqs. 2 and 3 the exponents of t are similar in module. That should lead to a correlation between the parameters A and *B* induced by the high energy mill processing. By plotting *B* versus *A* (Fig. 7), this relationship is confirmed. The metal work hardening causes a predominance of fracture of the material during the processing and changes in the powder size and morphology. Therefore the work hardening leads to a decrease of the parameter *A* and the changing in the composite powder characteristics leads to an increase of *B*.

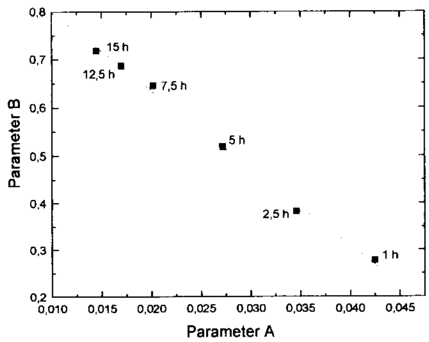

Fig. 7. Parameter *B* as function of parameter *A*.

The dots in Fig. 7 tend to be closer for longer milling times. Changes of parameters A and B are less evident as milling time increases, indicating that the powder tends to a stable condition during the attrition processing, as previously shown in other works related to High Energy Mill processing [5].

CONCLUSIONS

1-The compaction of AISI M2 +10% NbC composite powders can be quantitatively described by the compacting equation $\ln\left(\frac{1}{1-D}\right) = A\sqrt{P} + B$;

2-The parameters A and B of the compacting equation (Eq.1) characterize the changes of the composite powder processed by High Energy Ball Mill. The observed alterations of the parameters A and B indicate that the powder characteristics tend to a stable condition during the attrition processing; and

3-There is a correlation between the parameters A and B induced by High Energy Mill processing.

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