

## CERAMIC PROCESSING OF NBC NANOMETRIC POWDERS OBTAINED BY HIGH ENERGY MILLING AND BY REACTIVE MILLING

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**Abstract:** Production of nanometric ceramic powders is one of the most recent advances in materials science. However the large scale production of some materials is still a challenge. There are two approaches to the fabrication of nanomaterials that results in powders with distinct characteristics. In high energy milling the particle size is reduced by mechanical forces to achieve nanosized particles. Another technique is reactive milling in which nanometric particles are synthesized by mechanically activated reactions. In this work NbC nanoparticles were produced by high energy milling of commercial NbC and by self sustained high energy reactive milling of Nb<sub>2</sub>O<sub>5</sub>-Al-C powder mixture. The NbC particles were desagglomerated for 1h in a planetary mill. The obtained powders were characterized by X-ray diffraction, scanning electron microscopy and laser diffraction. The objective of this study was to compare the efficiency of two employed techniques to determine the most of producing nano-scale NbC.

### Introduction

One challenge of materials development is increase the production of nanoscale materials. These materials are the center of several researches due to the potential application in materials field. <sup>[1,2]</sup> The use of nanometric powders as reinforcement in ceramic matrix is one special application due to the good properties that these materials can provided to ceramics, such as mechanical reinforcement and enhancement on fracture toughness. <sup>[3,4,5, 6]</sup>

There are many paths to produce for production of materials at nanoscale, be used complex assembly process or traditional routes <sup>[7]</sup>. High energy milling is one traditional route widely applied on nanoceramics production. On this milling the energy transferred to materials is achieved by shearing action or impact of the high velocity balls<sup>8</sup>. This mechanical process leads to repeated welding and fracture of powder particles. <sup>[5,9]</sup> Due to high energy the process of obtaining nanometric crystalline structures can be separated in two routes. <sup>[1,10]</sup> One consists in starting with rough, large scale material and gridding it into a nanoscale shape by mechanical milling. This method is efficient in producing bulk quantities of nanocrystalline powder by the comminution of brittle materials. However this route provides a limited powder size in final products. <sup>[11]</sup> Other method is to create nanostructures through the synthesis of element reactive powders using the repeated impact of balls to create self propagating high temperature synthesis which can produce particles with smaller size. <sup>[3, 12]</sup> In the two cited methods there are variations not only in size but also the morphology of obtained particles.

The aim of this work was study the differences between niobium carbide produced using high energy milling as a method for particle size reduction of commercially available micrometric NbC, the material obtained by means of reactive milling of niobium oxide with carbon black and alumina powder to produce nanometric NbC.

### Experimental procedure

Present investigation was separated into two experimental groups. In one group a commercial available Niobium Carbide (H.C. Starck) was used to produce nanometric powders by means of mechanical milling using a high energy milling. Commercial powders of aluminum (ALCOA), Nb<sub>2</sub>O<sub>5</sub> (CBMM), High purity alumina (AKP-53, Sumitomo) and carbon black was the reactants used to produce NbC by reactive milling process, following the Equation 1.



The milling equipment used on this work was a shaker Mix Type SPEX, with hardened steel vial and balls. The balls diameter was 10mm and the ball-to-material ratio fixed on 4:1. Grinding was performed in 240 minutes for high-energy milling process and 280 minutes for reactive milling powders. Obtained powders were deagglomerated in planetary mill, for 1 h, in alcohol suspension with 0,2% of deflocculant PABA (4-aminobenzoic acid).

After drying the powders the characterization of obtained powders was carried out using X-ray diffraction to identify crystalline phases and to calculate the crystallite size of samples. This calculation was carried out in according to Scherrer method<sup>[13]</sup>, using the width of the Bragg peak profiles at half of the maximum peak intensity. Particle size distribution was obtained by laser diffraction method and the morphology of NbC powders was observed using scanning electron microscopy.

### Results and discussion

In Fig. 1(a) is showed X-ray diffraction pattern of high energy milled NbC powder. Only the peak corresponding to NbC phase is observed. The pattern showed on Fig. 1 (b) is the diffraction data for NbC powders obtained by reaction milling. Presence of NbC and Al<sub>2</sub>O<sub>3</sub> phases can be observed, these materials are expected due to the reactant products added in the mill according to Equation 1.

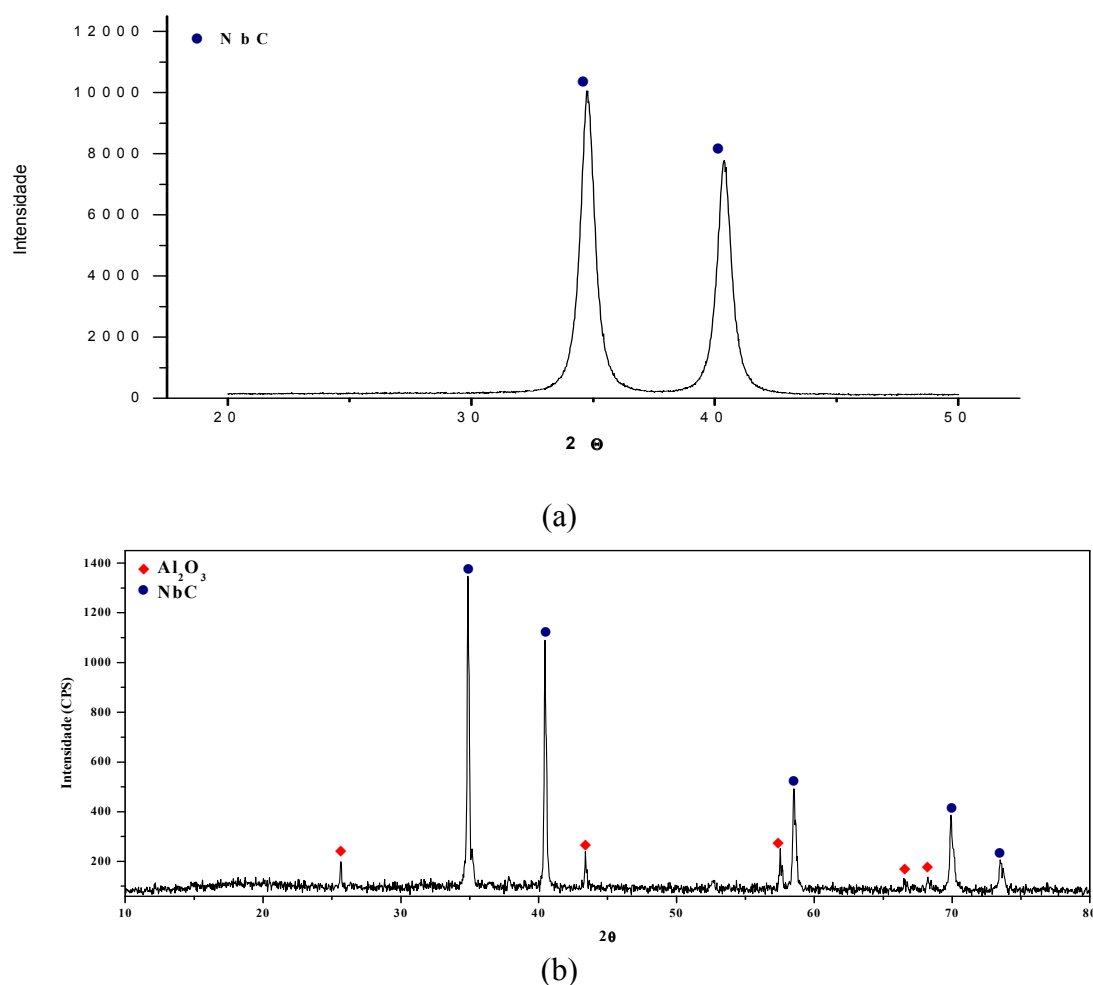


Fig. 1: Diffraction Pattern of obtained NbC powders obtained by (a) high energy milling (b) Reactive Milling

Peaks related to NbC were used to measure the crystallite size; obtained values were listed in Table 1. Differences can be observed in the size of reactive milled and high energy milled NbC, reactive milled NbC is smaller than the one obtained by mechanical breakage. Moreover, the Spex milled powder presents agglomerates formed with the fine powder compaction that occurs due the high-energy compressive impact between the balls.

Table 1: Crystallite size measured by XRD of NbC powders

Sample	Crystallite size [nm]
NbC reactive Milling	26,5
NbC High energy milling	45,1

Aggregates and agglomerates are observed in reactive milling process powders these clusters are formed during the synthesis of NbC.

Fig. 2 shows the NbC powder obtained by reactive milling process, the presence of dense clusters of crystallite formed by alumina and NbC. This powder distribution can be explained by the process. Since a reaction is highly exothermic, it can take place abruptly after a certain milling time and, once started, it proceeds in a self-sustained way, similarly to the so-called self-propagating high-temperature synthesis (SHS) process. After reach the reaction temperature all reactant powder is converted into products. The high temperature (2949°C) <sup>[13]</sup> of the process lead to formation of aggregates and dense agglomerates.

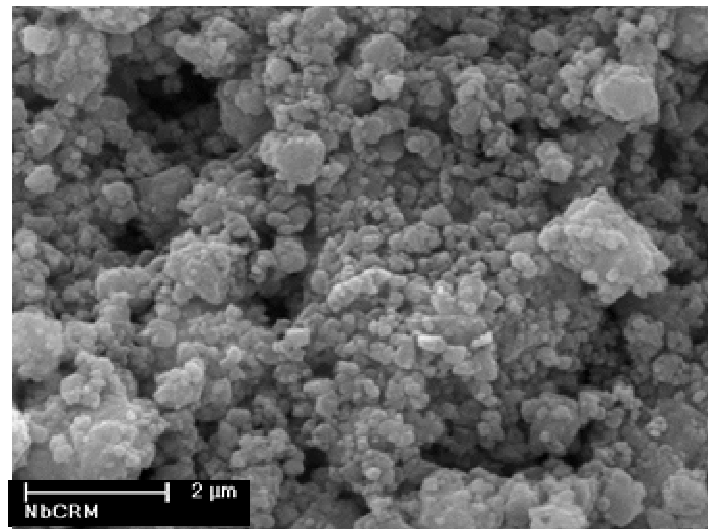


Fig. 2: Micrographs of alumina niobium carbide sample obtained by reactive milling process

When applied only mechanical energy the movement of spheres has a disordered behavior and impacts occur in different ways inside the vial <sup>[14]</sup>, so the breakage is the main process to reduce particles, Figure 3. In this case the mechanical properties are directly related to the efficiency of grinding. <sup>[5]</sup> For many materials the refinement is possible only if the energy transferred to material is enough to produce cold welding and fracture of the particles <sup>[15]</sup>, this means that during milling the impact energy of grinding media to powder should be enough to break hardened particles. However during the movement can occur powder compression, leading to formation of compacted clusters, formed by weak chemical bonds. Added to this effect there is the possibility that some particles do not break completely.

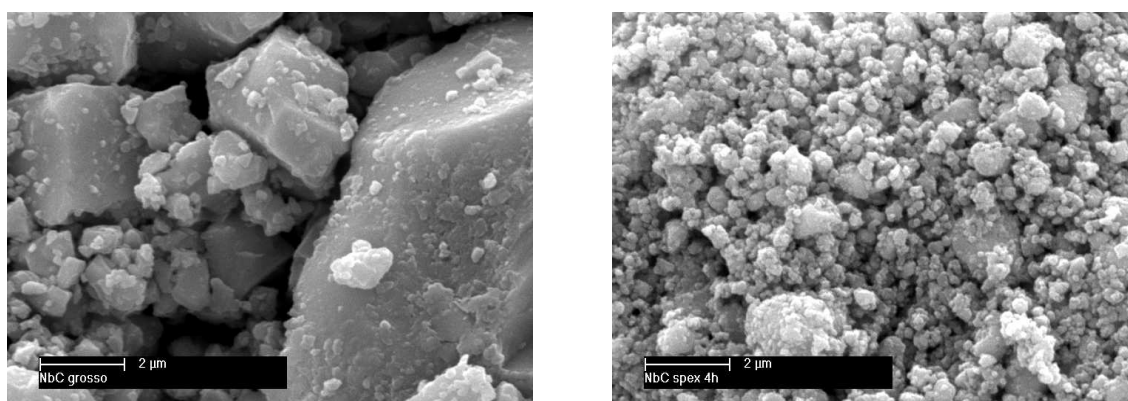


Fig. 3: Micrographs of alumina niobium carbide (a) as received (b) high energy milled

In Fig. 4 is showed the particle size distribution of as received NbC and high energy milled powder measured by laser diffraction, were observed a very large particle distribution for the powders. Can be noted the decreasing in particle size after milling and the presence of smaller particles in the beginning of distribution consistent with the crystallite size measured.

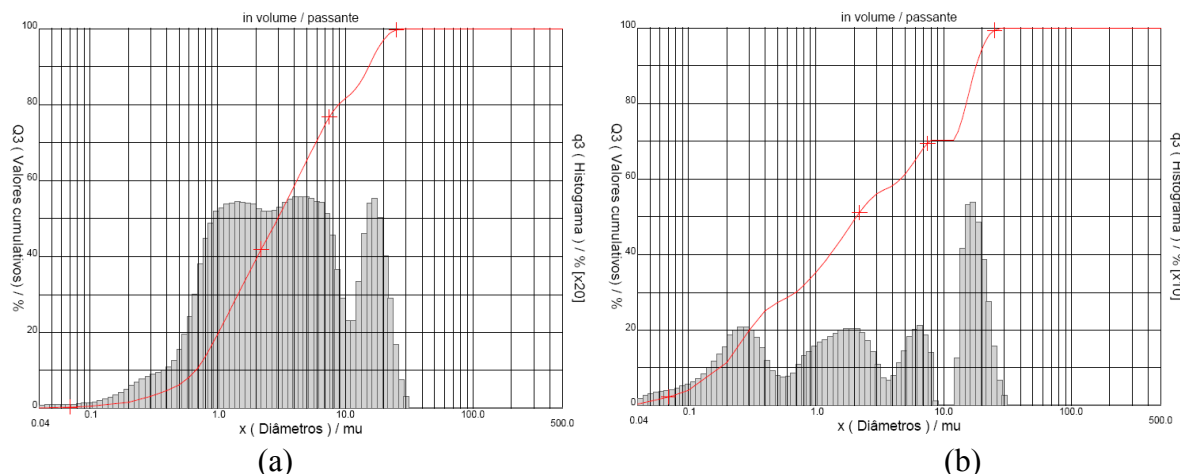


Fig. 4: Particle size distribution of (a) As received NbC and (b) high energy milled NbC

Comparing the obtained powders it is possible to make nano-scale materials using high energy milling but the reactive milling produces smaller particles. These results are due to distinct characteristic of process. To achieve particles by reactive milling process the shock between balls, powder and vial is enough to produce a self sustained reaction and the energy is entirely consumed during the process, which means that in a complete reaction all the reactants inside turn into the final product. On the other hand using only mechanically grinding the comminution of particles depend on the energy transferred from ball to powder.

## Conclusion

In this work was showed the use of conventional technique to produce nanoscale materials by two different routes. The high energy mill of particles leads to formation of strong agglomerates or aggregates of reaction products and grinding NbC. This effect is attributed to welding caused by compression particles against the wall of the jar and was observed on all studied samples. Even with the formation of strong agglomerates the reactive milling process produces smaller particles when compared to high energy milling of rough materials.

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