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MAGNETIC PROPERTIES AND HARDNESS OF NANOSTRUCTURED WC-CO CEMENTED CARBIDE

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Abstract. Nacrystalline WC-10wt.%Co powders were prepared by high energy milling and liquid phase sintered. The powders with different milling time were characterized by X-ray diffraction and SEM. After sintered the WC-10wt.%Co cemented carbides exhibits ultra fine grain sizes. Coercitive field and Vickers hardness measurements on the consolided samples detected a significant increase and decrease Vickers hardness with the milling time increase in sintered samples.

Introduction

The main class of hard metal is a composite, consisting of a ceramic part, tungsten carbides (WC) and metallic part, cobalt (Co). It is used for cut tool, it have good mechanical properties as: high hardness, breaking good resistance and heat resistance. All of these properties depend of the material microstructure that it comes from the material processing and sintering, for example: the tenacity and hardness grow with refinement powder of WC and Co. The high energy milling is an important contribution for this process, Zhang et al [1]. The milling is a sufficiently process to used to prepare nanocrystalline, hard metal, amorphous alloy, metallic alloy and nitrites metal. With the high energy milling is possible to get in 32 minutes nanocrystallines particles with grain size around 35 to 25 nm for the composite WC-10%Co [1].

Nanocrystalline WC-Co powders are presently used for producing cemented carbides with finer microstructure and excellent mechanical properties such high hardness, strength and toughness. Although nanocrystalline WC-Co powders have been developed by spray conversion process (SCP) and by high energy ball milling, some controversial persist about their mechanical properties. In this work, the hardness and magnetic properties of nanocrystalline WC-Co cemented carbide with different milling times were investigated.

At the same time, the grain size and structure evolution of the powders were monitored by Rietveld analysis of X-ray diffraction patterns. The relations among the microstructures, hardness, chemical compositions and magnetic properties were discussed.

Experimental procedure

The start materials were WC powders with particle size of 0,57μm, manufactured by WBH in Austrian and Co powders with particle size of 0,95 μm, manufactured by HC Stark in Germany. In the powders was added graphite powder to supply carbon loss during the processing. It also was added paraffin to serve as lubricant during compacting process. The Composition for all samples were 89,93 wt.% of WC, 10 wt.% of metallic Co, 0,07 wt.% of graphite and 2 wt.% of, paraffin. The composition was the same for all samples.

The nanostructured powders of WC-Co were prepared in a planetary mill Fritsch Pulverisette 7, at the times: 2 h, 100 h, 200 h and 300 h. After this process the powders were dried through at 70°C Rota vapor temperature. Dry mechanical mixed was carried out in planetary ball mill at 1/6h. The samples of the WC-10wt.%Co were compacted in stainless steel die with cylindrical county of 10 mm of diameter, at a pressure of 200 Mpa. The compacted samples were sintered in a resistive furnace at argon atmosphere at 1400℃ by 5 min. The X-ray diffraction analysis was carried out in a Shimadzu-XD600 diffractometer, with CuKα (1.5418°*A*) radiation. A scanning electronic microscope model Philips-XL30 was used to determine the distribution of pores and average diameters of the sintered samples and powders respectively.

The magnetization measurements were carried out on a vibrating sample magnetometer in the Magnetic Materials Laboratory UFRN, equipped with applied fields up to 2T.

Results and discussions

At milling time increase the WC-10wt%.Co presented the formation of the particle composite this effect came from of the cold welding between particles. The table 1 shows the crystallite size of the composite at different milling times.

Does not have a significant reduction in the average particle size of the material milled at 2h compared with the starting material. However, significant decrease in particle sizes are obtained in the materials milled at 100, and 200h. The reduction of the particle size is very important to get better properties of the hard metal.

Table 1. Crystallite size.

Milling time (h)	crystallite size (nm)
Mec.Mixture	70,3
2h	30,1
100h	27,4
200h	18,4
300h	13,2

The figure 1 show X-ray diffraction patterns of the WC-10wt.%Co powders mechanical mixed and milled at 2h 100h 200h and 300h. The starting WC-10wt.%Co powder contain a Co phase composed of fcc(α) and hcp (Є) phases. After 100h of ball milling, diffraction peaks of α-Co disappear. This is because that fcc-Co transforms into hcp-Co due to mechanically induced transformation in the ball milling. This kind of allotropic transition in Co was reported in previous work [6]. It can also found that with increase milling times the WC diffraction peaks became short and broad, which due to refining of grain size and increasing of internal strain resulted from ball milling. The decreased tendency to nanoscale grain sizes estimated from the widths of the X-ray diffraction peaks. The peak variation of cobalt phase for all the samples is not very clear because the intensity is low.

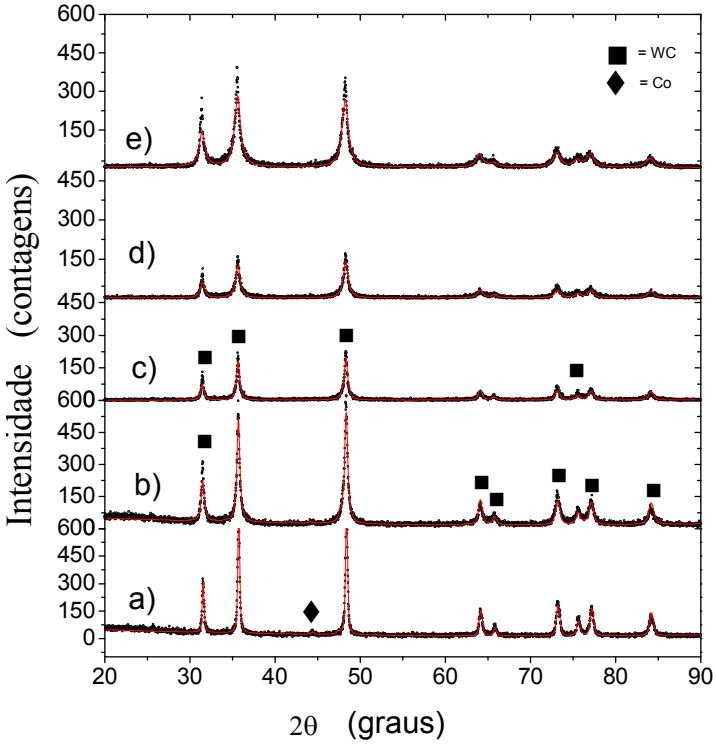

Figure 1. X-ray diffraction of WC-10wt.%Co powder, a) mechanical mixed, b) milled at 2h, c) milled at 100 h, d) milled at 200 h and e) milled at 300h.

The X-ray diffraction pattern of the WC-10wt.%Co sintered samples, planetary ball milled at 2h, 100h, 200h, 300h and mechanical mixed, are showed in figure 2. The samples mechanical mixed and milled at 2h shows the similar phase, hexagonal WC and Co phase, The sample milled at 100h appearance of an additional Co_3C, and Co_2W_4C phases, for sample milled at 200h, disappear the Co_3C phases. For sample milling at 300h appearance more phase of Co_2W_4C. These transformations of phases are in accordance with WC-Co ternary phase's diagram, Nerz et al [8]. The appearance this phases it must be related with the variation of the stoichiometric of the carbon in the WC phase, caused by a provable deficiency of the argon atmosphere used during the sintering. Also he is frequent used to locate the carbon deficiency, as well of the presence of the eta-phase.

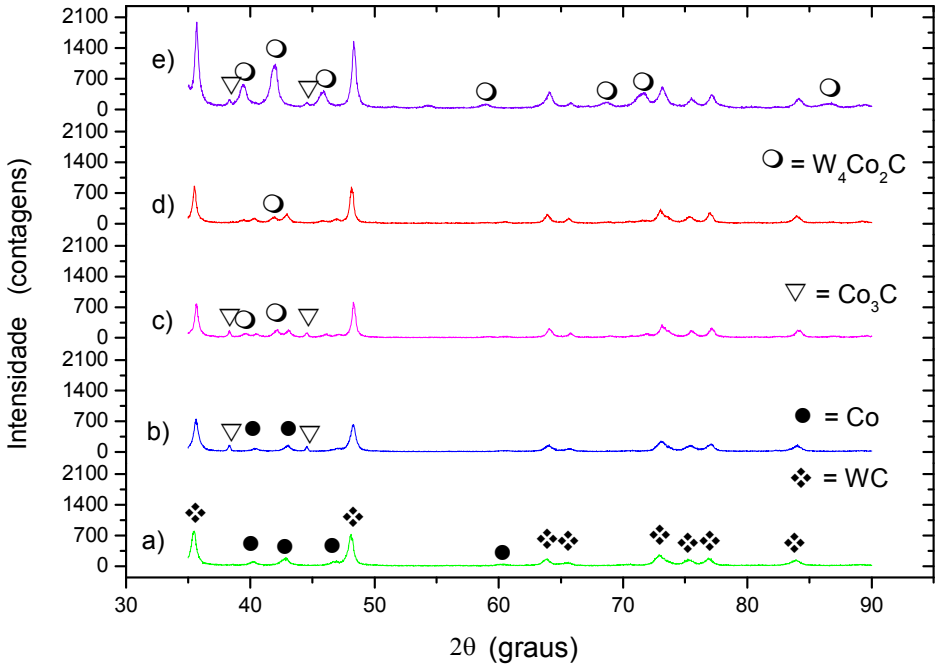

Figure 2. X-ray diffraction of WC-10wt.%Co sintered sample, a) mechanical mixed, b) milled at 2h, c) milled at 100h, d) milled at 200h and e) milled at 300h.

The table 2 shows the coercitive field and hardness measurement parameters. The reduction of the coercitive field with milling time increase can be related with the reduction of free cobalt in composite. The reduction of density can be related with the appearance of the non magnetic phases and reduction the hardness is related the reduction of density. However the relationship between coercitive field and microstructure of WC-Co is complex, because of the interaction of domain walls in a two-phase material with non-magnetic particles. [6,7]. The coercitive field measurement of the all sintered samples detected a significant increase and a reduction in the Vickers hardness, with milling time increase, as shows in table 2. The sample mechanical mixed and milled at 2h present a similar hardness and coercitive field. The sample milled at 300 hours had less hardness and not had coercive field, because the sample presented a paramagnetic behavior.

The hardness increase with the reduction of the particle size (pores reduction), this also originates an increase in the coercitive field.

The increase coercitive field and the hardness reduction are related with the appearance of the non-magnetic phases. On the other hand the appearance of the no-magnetic phases as is truth that reduces the hardness, but would have to also reduce the coercitive field. As separate the effect with the simultaneous graph of hardness, coercitive field and milling time, as shows in figure 3.

Magnetic hysteresis measurements on the sintered samples detected significant increase in the coercitive field and a decrease on the saturation magnetization with milling time increasing [5]

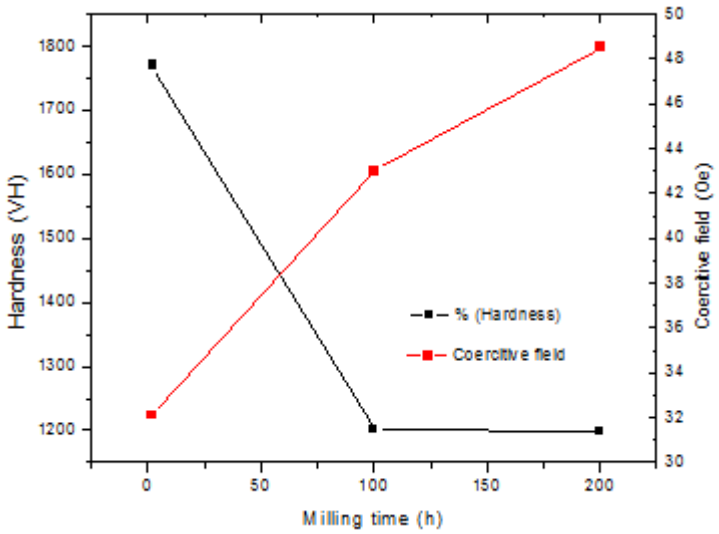

Figure 3. Relate between hardness, coercitive field and milling time.

Table 2. Measurement of the hardness and coercitive field.

Samples	Hardness [VH]	Coercitive field [Oe]
2h	1770	32,1
100h	1202	43,0
200h	1199	48,5

The magnetization measurements of the WC-Co powders are shows in table 3. The variation in saturation magnetization in the powders mechanical mixed and milled at 2h, 100h, 200h and 300h are relate with bulk variation of wt.% Co and allotropic transformation of Co-hcp for Co-fcc.

Table 3. Magnetization measures.

Sample	HC [Oe]	Ms [emu/g]
Mec. mixed	0,016	7,76
2h	0,016	12,18
100h	0,029	2,12
200h	0,028	2,75
300h	0,030	5,00

Conclusion

The particle size reduction had an influence in coercitive field and less influence in hardness. The appearance the non magnetic phase less influence in hardness. The appearance the non magnetic phase less influence in coercitive field, but had vary influence in hardness reduction. The coercitive field increase with decrease the particle size in the WC-Co powders. The magnetization

saturation varies with the variation of free Co wt.% in WC-Co powders. With milling time increase the value of magnetization saturation decrease and decrease also the free Co wt.%. The allotropic transformation of cobalt have also influence in magnetization saturation.

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