HIGH ENERGY MILLING EFFECT IN PARTICLE SIZE DISTRIBUTION AND CELLS PARAMETERS OF THE WC-10wt.%CO COMPOSITE

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Resumo: Nanocrystalline WC-10wt.%Co powders were prepared by high energy milling and liquid phase sintered. The powders with different milling time were characterized by particle size distribution, mean diameter and cells parameters. The milling time caused a particle size reduction and mean diameter reduction. The cells parameters of the phases WC, Co-hcp, Co-fcc in WC-10wt.%Co composite powders decreasing at milling time increase. For the composites sintered the cells parameters of the phases WC, Co-hcp and Co-fcc it is practically constant for the axial relations “a”. For the axial relations “c” at milling time increase the cells parameters in WC-10wt.%Co composite powders for the WC and Co-hcp phases increase until milled at 2 hours compared with starts materials, after 2 hours at milled until 300 hours decrease. At milling time increase the cells parameters in WC-10wt.%Co sintered composite for the WC phase decrease until milled at 300 hours compared with starts materials The cells parameters of the Co-hcp phase until milled at 2 hours increase compared with starts materials, after 2 hours at milled until 100 hours decrease. After 100 hours at milled until 200 hours decrease.

Key Words: High energy milling, particle size distribution, mean diameter and cells parameters
1.0 INTRODUCTION


THIS WORK, A STUDY THE EFFECT OF HIGH ENERGY MILLING IN PARTICLE SIZE DISTRIBUTION, MEAN DIAMETER AND CELLS PARAMETERS.

2.0 EXPERIMENTAL PROCEDURE

The start materials were WC powders with particle size of 0.57 μm, 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 was the same for all samples.

The nanostructured powders of WC-Co were prepared in a planetary mill Fritsch Pulverisette 7, at different times. After this process the powders were dried through at Rota vapor. Dry mechanical mixed was carried out in planetary ball mill at 1/6h. The X-ray diffraction was carried out in Shimadzu-XD600 difractometre, with CuKα (1,5418Å) radiation with tension 30kv and 20mA. 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 ºC by 5 min. The particle size distribution and mean diameter analysis was carried out in a Granulometro Cilas model 920 L and 1180. The cells parameters were calculated though Rietveld method analyzes.

3.0 RESULTED AND DISCUSSION

The Figure 1 show the particle size distribution of WC powder and Co powder when can be see that for the particle size of Co is minor than 10 μm, with a mean diameter of 3.90 μm and in WC the particle size is minor than 10 μm, with a mean diameter of 1.46 μm.

Figure 1. Particle size distribution of a) WC powders and b) Co powders.

The figure 2 show the particle size distribution of the WC-10wt.%Co mechanical mixed and milled at 2h. The particle size distribution in powder mechanical mixed is minor than 17 μm, with a mean diameter of 2.05 μm. Comparison the figures 1 and 2 can be affirmation that have a amount of WC and Co agglomeration, this effect can be justified though the pick in 10 μm of the Co in figure 2. The particle size
distribution in powder mechanical mixed is minor than 30 µm, with a mean diameter of 3.79 µm. This values are major than the powders mechanical mixed because the milling time caused a minor cold welding and the appearance the little agglomerations of WC.

![Figure 2. Particle size distribution of WC-10wt.% Co powders a) mechanical mixed and b) milled at 2h.](image)

The figure 3 show the particle size distribution of the WC-10wt.%Co milled at 100h and milled at 200h. The particle size distribution in powder milled at 100h is minor than 16 µm, with a mean diameter of 2.91 µm. This values are major than the powders milled at 2h this effect came from of the particle reduction at milling time increase. The milling time caused the cold welding and amount of the agglomeration of WC e Co that also influenced these values. The particle size distribution in powder milled at 200h is minor than 12 µm, with a mean diameter of 1.51 µm. This values are minor than the powders milled at 100h this effect came from the particle size reduction at milling time increase. The amount of the cold welding, fracture and agglomeration of WC at milling time increase influenced in value of mean diameter.

![Figure 3. Particle size distribution of WC10wt.%Copowders c) milled at 100h and d) milled at 200h.](image)

The figure 4 show the particle size distribution of the WC-10wt.%Co milled at 300h when can be see that for the particle size is minor than 12 µm, with a mean diameter of 1.76 µm. The mean diameter of the composite powder milled at 300h is major than 200h, this effect is related the a possible cold welding between particles.
The figure 5 shows the variation of mean diameter of the composite powder milled at different times. The same figure shows that the milling time caused a reduction of the mean diameter of the particles. The composite powder milled at 300h have a mean diameter major than milled at 200h, this effect is caused by a cold welding between particles. The table 1 shows the variation of mean diameter of the particles in composites powders at milling time increase.

Figure 5. Variation of mean diameter of the particles in composite powders milled at different times.
Table 1. Variation of mean diameter of the particles in composite powders milled at different times.

<table>
<thead>
<tr>
<th>Milling time (h)</th>
<th>$D_{\text{min}}$ (µm)</th>
<th>$D_{\text{max}}$ (µm)</th>
<th>$D_{\text{med}}$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.48</td>
<td>9.72</td>
<td>3.79</td>
</tr>
<tr>
<td>100</td>
<td>0.50</td>
<td>7.27</td>
<td>2.91</td>
</tr>
<tr>
<td>200</td>
<td>0.27</td>
<td>4.02</td>
<td>1.51</td>
</tr>
<tr>
<td>300</td>
<td>0.11</td>
<td>5.81</td>
<td>1.76</td>
</tr>
</tbody>
</table>

The theory of the crystalline systems establish the following axial relations: cubic system $a = b = c$ and hexagonal system $a = b \neq c$, R. A. Young (1965). In this context the figures 6 e 7 analyzing the axial relations “a” for the phases WC, Co-hcp and Co-fcc in WC-10wt.%Co composite.

In the composite powders the cells parameters for the phases WC, Co-hcp and Co-fcc decrease at milling time increase as show in figure 6. This decreasing is directly influenced for particle size reduction at milling time increase. This reduction caused the micro deformation in crystalline structure and decreasing crystallite size.

In the sintered composite the cells parameters for the phases WC, Co-hcp and Co-fcc are practically constant at milling time increase as show in figure 7. The variation existing in thousandth order it must be related with the formation of the substitutional solid solution during the sintering, influenced directly with the particle size reduction. At milling time increase caused allotrophic transformations in Co phase, Y. Huang, et al (1994). The table 2 and 3 shows the cells parameters measurement of the composite powders and sintered composites, axial relations “a”.

Table 2. Cells parameters measurements of the composite powders, axial relations “a”.

<table>
<thead>
<tr>
<th>Milling time</th>
<th>Cells parameters (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>WC</td>
</tr>
<tr>
<td>Start mat.</td>
<td>2.900</td>
</tr>
<tr>
<td>Mec. mix.</td>
<td>2.9064</td>
</tr>
<tr>
<td>2h</td>
<td>2.9057</td>
</tr>
<tr>
<td>100</td>
<td>2.9057</td>
</tr>
<tr>
<td>200h</td>
<td>2.9040</td>
</tr>
<tr>
<td>300h</td>
<td>2.9031</td>
</tr>
</tbody>
</table>

Table 3. Cells parameters measurement of the sintered composite, axial relations “a”.

<table>
<thead>
<tr>
<th>Milling time</th>
<th>Cells parameters (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>WC</td>
</tr>
<tr>
<td>Start mat.</td>
<td>2.900</td>
</tr>
<tr>
<td>Mec. mix.</td>
<td>2.907</td>
</tr>
<tr>
<td>2h</td>
<td>2.909</td>
</tr>
<tr>
<td>100</td>
<td>2.906</td>
</tr>
<tr>
<td>200h</td>
<td>2.909</td>
</tr>
<tr>
<td>300h</td>
<td>2.968</td>
</tr>
</tbody>
</table>
In the figures 8 and 9 are analyzing the axial relations “c” for the phases WC, Co-hcp following the theory of crystalline system. At milling time increase the cells parameters in WC-10wt.%Co composite powders for the WC and Co-hcp phases increase until milled at 2 hours compared with starts materials, after 2 hours at milled until 300 hours decrease as show in figure 8.

The table 4 and 5 shows the cells parameters measurement of the composite powders and sintered composites, axial relations “c”.

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**Figure 6.** Variation of the cells parameters versus milling time the phases WC, Co-hcp and Co-fcc in composites powders.

**Figure 7.** Variation of the cells parameters versus milling time the phases WC, Co-hcp and Co-fcc in composites sintered.
This decreasing is directly influenced for particle size reduction at milling time increase. This reduction caused the micro deformation in crystalline structure and decreasing crystallite size.

At milling time increase the cells parameters in WC-10wt.%Co sintered composite for the WC phase decrease until milled at 300 hours compared with starts materials. The cells parameters of the Co-hcp phase until milled at 2 hours increase compared with starts materials, after 2 hours at milled until 100 hours decrease. After 100 hours at milled until 200 hours decrease The variation existing in thousandth order it must be related with the formation of the substitutional solid solution during the sintering, influenced directly with the particle size reduction.
4.0 CONCLUSION

The milling time caused a particle size reduction and mean diameter reduction. In the composite powder the cells parameters of the phases WC, Co-hc, Co-fcc decreasing at milling time increase. This decreasing is directly influenced for particle size reduction at milling time increase in axial relations “a”. This reduction caused the micro deformation in crystalline structure and decreasing crystallite size.

For the composites sintered the cells parameters of the phases WC, Co-hcp and Co-fcc it is practically constant in axial relations “a”. The variation existing in thousandth order it must be related with the formation of the substitutional solid solution during the sintering, influenced directly with the particle size reduction.

In the WC-10wt.%Co composite powders for the WC and Co-hcp phases increase until milled at 2 hours compared with starts materials, after 2 hours at milled until 300 hours decrease in axial relations “c”. This variation is directly influenced for particle size reduction at milling time increase.

At milling time increase the cells parameters in WC-10wt.%Co sintered composite for the WC phase decrease until milled at 300 hours compared with starts materials. The cells parameters of the Co-hcp phase until milled at 2 hours increase compared with starts materials, after 2 hours at milled until 100 hours decrease. After 100 hours at milled until 200 hours decrease in axial relations “c”. These variations are related with the formation of the substitutional solid solution during the sintering, influenced directly with the particle size reduction.

5.0 REFERENCE


Y. Huang, Y. K. WU, and H Q. YE., Phase Transformation of Cobalt Induced by Ball Milling, Laboratory of atomic imaging of solids, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China, 1994.
