# NEW ROUTE FOR PRODUCTION OF HARD METAL COMPOSITE POWDER

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***Abstract.*** *The aim of this is work to investigate the mechanism of densification of WC-Co at different temperatures. The obtaining hard metal alloy in the percentage of 10, 15 and 20 per cent of cobalt from a commercial alloy of WC-6% Co, by mass balance. It was a procedure for obtaining the binary alloys, by calculation of balancing determining the amount of cobalt necessary to transform a commercial alloy 94WC-6Co in alloys with 10, 15 and 20 percent of cobalt. After the calculations made added to the commercial cobalt alloy with 1.5% of oil in a blender on "Y". The alloys with 10, 15, 20 percent of cobalt obtained by mixing mass were sintered at their respective temperatures of 1420ºC, 1400°C and 1360°C. To analyze the efficiency of the mixture was tested for density, metallography and microhardness.*

***Key-words:*** *Hardmetal, Powder metallurgy, Sintering, Mass balance.*

## 1 INTRODUCTION

The term "hard metal" indicates a composition consisting essentially of two constituents. The carbide, high hardness and high wear resistance, and the principal is the tungsten carbide, is often added to titanium carbide, tantalum and niobium. A metal binding agent can be iron, nickel or cobalt. The more metal used as binder phase is cobalt. (Chiaverini, V. 2001). The advent of hard metal at the decade end 20 in Germany, where Karl Schröter in the laboratory produces WC powder, causing the second major thrust in the area of material for cutting tool. Krupp Organization was pioneer in the development of hard metal called Widia (Diamont Wie in German, as diamond), according to its high hardness and wear resistance, a reference to the similarity between the properties of both materials. (Kim Chang-Soo. 2004)

This evolution combined with a need for high performance material, the hard metal transpose the exclusivity of its use in machining. This is used extensively in cold forming in general, such as extrusion, forging, stamping, lamination. Also used in mechanical seals, dies punctures and compaction of powder metallic or ceramic, and oil drilling tools, showing that the hard metal is an composite for many uses. (Brito F. I. G. *et al.* 1997)

In 40 years, almost half this sintered carbide was intended for the manufacture of tools for mining. At that time, the hard metal was taxed as a material of high cost, but has not been discarded due the excellent wear resistance, but over time its use was feasible. The fixations interchangeable with simple forms gaining market, because the inserts were flat on both sides and with provision for fixing, added output of chips, and improving the geometry of chip-breaking. (Rodrigues, M.F., 2006) The technique for the production of hard metal can be described briefly; the powder is compressed to pressures ranging from 80 to 200MPa. (ASM Metals Hanbook v.7, 2004)

The mixture is compressed, resulting in pieces in the desired format or briquettes with sufficient strength to be manipulated. The size of the piece are not the end, because 40 to 50% of the volume compressed is porosity which should disappear in the sintering, causing a linear contraction from 18 to 21%, depends basically the amount of binding agent and the sintering parameters. (Albert, C.H., 2000)

The sintering of hard metal with WC-Co occurs at temperatures between 1360 to 1600ºC, depending on the percentage of binders, which can vary from 6 to 25%. The lower the carbon presence in the composite, the greater the amount of tungsten dissolved, which contributes to increased resistance to breakage by traction, hardness and elongation (4 to 10%). The large contraction of the hard metal during sintering is due to the excellent wettability of WC by the liquid phase, around 1.300ºC, (Albert, C.H., 2000)

Developed by means mass balance of three hard metal composite, with 10, 15, and 20 percent cobalt. After the mass balance determined was the physical and mechanical properties for each mixture of the composite as: apparent density, green density, shrinkage, linear contraction, metallography and microhardness. (Shi, X.L., 2005)

The mass balances are used to explain quantitatively, the efficiencies, profitability, etc.. To determine an efficient mass balance, should consider the operation's principles, as a system, as a process, as a border, and basis of calculation. The system is defined as an area selected, which may be subject to definition and assessment of physical properties. ( College, W. and Chang, R. ,2002) In the quality control of hard metal, it is customary to determine the values of density, porosity to assess and examine the microstructure.( Kolaska, H. and Wheith, W.; 1992)

Table 1: Density and microhardness of the carbide. ( Kolaska, H. and Wheith, W.; 1992)

| **Compósite** | **Density g/cm³** | **Microhardness HV 50** |
|---|---|---|
| 94WC-6Co | 14.9 | 1580 |
| 90WC-10Co | 14.6 | 1410 |
| 85WC-15Co | 14.0 | 1150 |
| 80WC-20Co | 13.6 | 1050 |

## 2 MATERIAL AND METHODS

In this work we used the powder of 94WC-6Co with 99% of the composition, supplied by ALFA AESAR, -325 Mesch, which served as raw material, together with the powder of cobalt -400 Mesch the same company. In this composite was added zinc stearate (1.5% by weight) as a lubricant. Figure 1 shows the commercial powder of the composite WC-6Co, while Figure 2 shows the powder of cobalt.

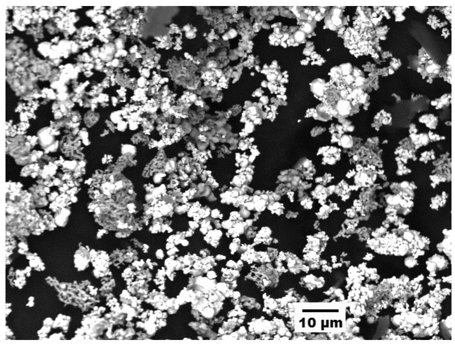


Figure1: Commercial powder WC-6Co 1.000X

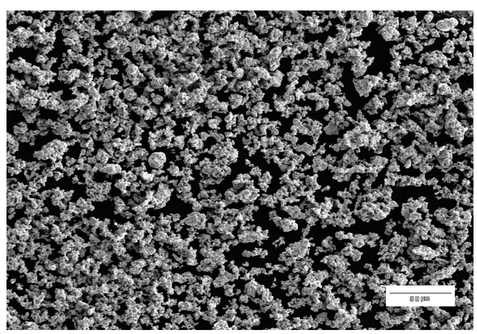

Figure 2: Cobalt powder -400Mesh 300X

Used is a balance of precision to determine the exact weight of powder, and a mixer in "Y" to homogenize the commercial composite mixture, and the cobalt powder with a particle size less than 37µm, for 30 minutes at 25rpm, aiming greater integration of the particles. Using 100 grams of the commercial composite 94WC-6Co by be an quantity satisfactory for tests. From the stoichiometric calculation (mass balance) determined the amount of cobalt to be added in the commercial composite 94WC-6Co. After mixing the powders, using the scanning electron microscope mark JEOL model 5800 to evaluate the homogeneity of the commercial composite hard metal WC-6Co and cobalt. The powders were placed in the die cavity to determine the apparent density (g/cm3) of the composite obtained by mass balance. Knowledge this characteristic determines the real volume occupied by a mass of loose powder. Consequently, was determined the depth cavity of the matrix. Was used a die with 22,20 mm in height and diameter of 14,00 mm, with a fixed volume of 3,41 cm3 in a cylindrical shape. To compression matrix used was a cylindrical steel tempered and Eka hydraulic press with a maximum capacity of 40 tonnes. In the compaction pressure was used 200MPa.

Traced the sintering curve of WC for the samples with 6, 10, 15 and 20% of cobalt, with a heating rate equal the typical curve of the furnace exceeding 10°C/min. (maximum heating rate shown in the literature). One aspect kept of the conventional curve was the effective sintering time of 60 min. From ambient temperature to the heat level of 1000°C was used a heating rate of 21,75ºC per minute. From 1000°C by the stage thermal sintering was used to heat a heating rate of 12,3ºC per minute. The average rate of heating was 13°C/min. The average cooling rate of parts in the furnace was 9,21° C /min until the ambient temperature.

The green density was determined by the method of Archimedes, as MPIF-95 standard, based on the thrust exerted on the sample during its immersion in a container with water, connected to a balance of precision. The diameters of the sintered samples were measured with a Mitutoyo micrometer mark of 0 - 25mm, together with the height. Was found the linear contraction of the parts. Measurement of Vickers microhardness, according to ASTM E 384 - Standard Test Method for Microhardness of Materials, defines the microhardness test,the hardness of microindentações performed with calibrated equipment to penetrate specific geometry of diamond. The test was conducted in microdurometro of LAFUN - Casting Laboratory - UFRGS. Using a load 1000gf on the surface of the test material to measure the diagonals of the indentations optics. The samples were analyzed in the scanning electron microscope brand JEOL 5800 coupled with EDS, where were realized the metallography and EDS.

## 3 RESULTS

From the ready mix was found apparent density of composites of hard metal, obtaining values in g/cm3, as shown in Table 2.

Table 2: Apparent density of hard metal composite

| hard metal composite | Density g/cm³ |
|---|---|
| 94WC-6Co | 2.57 |
| 90WC-10Co | 2.63 |
| 85WC-15Co | 2.55 |
| 80WC-20Co | 2.45 |

Using the mass balance if defined the percentage of the composite, adding 4.5, 10.5, 17.5 grams of cobalt, with granulometry of -400Mesh in the composite of 94WC-6Co by transforming the composite at 90WC-10Co, 85WC, 80WC-15Co and 20Co, respectively, as shown in Table 3.

Table 3: Percentage of cobalt from hard metal composites

| Hard metal composite | Percentage Cobalt | Mass additional of Cobalt (g) | Mass total of Cobalt (g) |
|---|---|---|---|
| 94WC-06Co | 6 | 0 | 6 |
| 90WC-10Co | 10 | 4.5 | 10.5 |
| 85WC-15Co | 15 | 10.5 | 16.5 |
| 80WC-20Co | 20 | 17.5 | 23.5 |

The samples were compressed in size, geometry and composition of the material requested, with sufficient integrity to be handled and moved. The pressure of compaction was obtained through the curve of compressibility of the alloy, as in Figure 3, drawn based on ASTM B331.

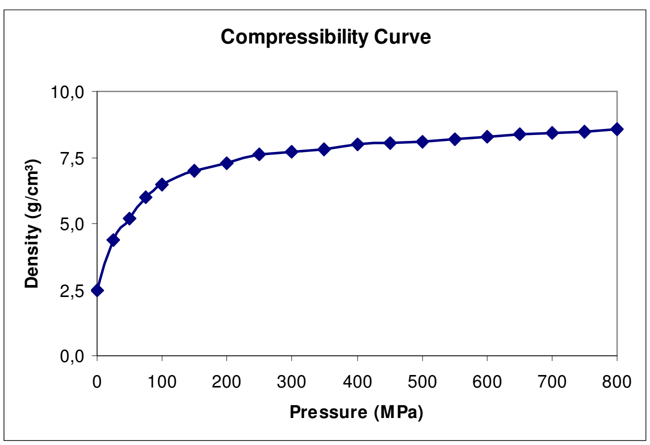


Figure 3 – Compressibility curve of the material WC-6%Co

The densities of the green composites of hard metal with 6, 10, 15 and 20% obtained in the tests are shown in table 4:

Table 4: The green density of hard metal composites

| Sample | Hard metal composte | Volume of compressed (cm$^3$) | Mass (g) | green density (g/cm$^3$) |
|---|---|---|---|---|
| 01 | WC-06%Co | 1.21 | 9.57 | 7.91 |
| 02 | WC-06%Co | 1.40 | 11.29 | 8.02 |
| 03 | WC-06%Co | 1.44 | 11.39 | 7.91 |
| 01 | WC-10%Co | 1.52 | 11.11 | 7.31 |
| 02 | WC-10%Co | 1.48 | 11.10 | 7.50 |
| 03 | WC-10%Co | 1.47 | 10.80 | 7.34 |
| 01 | WC-15%Co | 1.39 | 10.05 | 7.22 |
| 02 | WC-15%Co | 1.52 | 11.03 | 7.25 |
| 03 | WC-15%Co | 1.52 | 10.84 | 7.13 |
| 01 | WC-20%Co | 1.42 | 9.99 | 7.03 |
| 02 | WC-20%Co | 1.47 | 10.35 | 7.04 |
| 03 | WC-20%Co | 1.47 | 10.00 | 6.80 |

Figure 4 shows the curves of sinterizações measures, based on a conventional curve, measuring up to every 15 minutes, thus showing the behavior of the furnace. The temperature of sintering is shown in the figure for the composite of WC-20Co, WC-15Co, WC-10Co and WC-6Co respectively. It is important that evince the sintering cycles are high heating rate, which is not common in the sintering of hard metal.

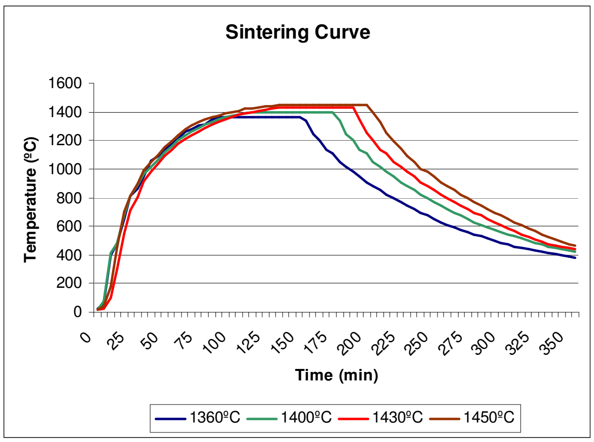


Figure 4: Sintering curve dos hard metal composites

The measurement of the sintered density is very important to be evaluated, get together the value of densification. After the sintering were measured the densities of the samples using the principle of Archimedes, as MPIF Standard-95. Table 5 shows the densities of composites in cylindrical shape. Figure 5 shows the relative density of the compositions.

Table 5: Density of the sintered hard metal composites

| Sample | Hard metal composite | Sintered volume (cm$^3$) | Mass (g) | Sintered density (g/cm$^3$) |
|---|---|---|---|---|
| 01 | WC-6%Co | 0.772 | 11.38 | 14.75 |
| 02 | WC-6%Co | 0.647 | 9.53 | 14.74 |
| 03 | WC-6%Co | 0.765 | 11.24 | 14.70 |
| 01 | WC-10%Co | 0.747 | 10.60 | 14.19 |
| 02 | WC-10%Co | 0.737 | 10.40 | 14.11 |
| 03 | WC-10%Co | 0.762 | 10.68 | 14.01 |
| 01 | WC-15%Co | 0.721 | 9.89 | 13.72 |
| 02 | WC-15%Co | 0.803 | 10.91 | 13.59 |
| 03 | WC-15%Co | 0.787 | 10.72 | 13.63 |
| 01 | WC-20%Co | 0.750 | 9.84 | 13.12 |
| 02 | WC-20%Co | 0.792 | 10.29 | 13.00 |
| 03 | WC-20%Co | 0.764 | 9.96 | 13.03 |

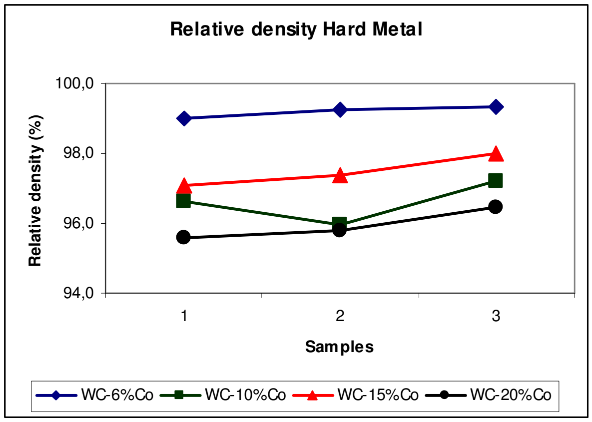


Figure 5: Relative density of hard metal composites

Table 6 shows the average linear and volumetric contraction of composites for mass balance, after the sintering. Figures 6 and 7 show the volume reduction and linear reduction in two different composites.

Table 6: linear and volumetric contractions of hard metal composites

| Hard metal composite | Volumetric contractions (% ) | Linear contractions (height) (%) | Linear contractions (diameter) (%) |
|---|---|---|---|
| 94WC-6Co | 45.9 | 17.4 | 19.7 |
| 90WC-10Co | 49.8 | 21.9 | 19.7 |
| 85WC-15Co | 48.2 | 18.0 | 19.7 |
| 80WC-20Co | 47.1 | 18.7 | 19.7 |

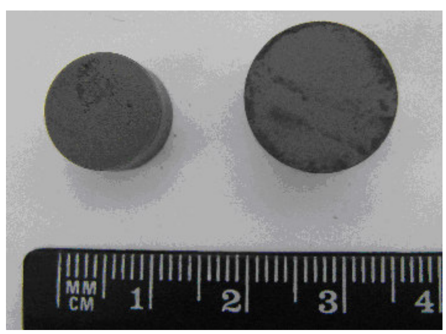


Figure 6: 1st sample sintered and 2nd sample compacted WC15-Co

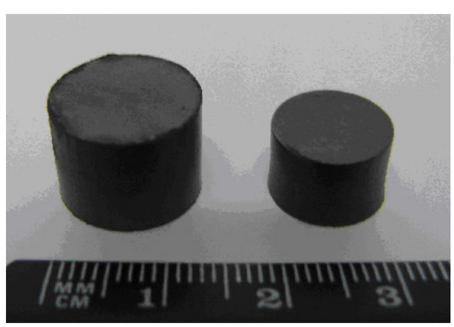


Figure 7: 1st sample sintered 2nd sample compacted WC20-Co

Obtaining measures of Vickers microhardness (HV) according to ASTM E 384 is shown in table 7.

Table 7: Microhardness of hard metal composites.

| Atmosphere and temperature | Hard metal composite | Load (gf) | Indentation Time (seg) | Microhardness (HV) |
|---|---|---|---|---|
| **$H_2$ 1450 ºC** | 94WC-06Co | 1000 | 15 | **1663 ± 20** |
| **$H_2$ 1420 ºC** | 90WC-10Co | 1000 | 15 | **1437 ± 20** |
| **$H_2$ 1400 ºC** | 85WC-15Co | 1000 | 15 | **1150 ± 20** |
| **$H_2$ 1360 ºC** | 80WC-20Co | 1000 | 15 | **1055 ± 20** |

Figure 8 shows a magnification of 300x showing a homogeneous mixture of WC-10Co composite, Figure 9 shows a magnification of 1500X, showing also the homogeneity of it, where is possible see the cobalt added in the mixture.

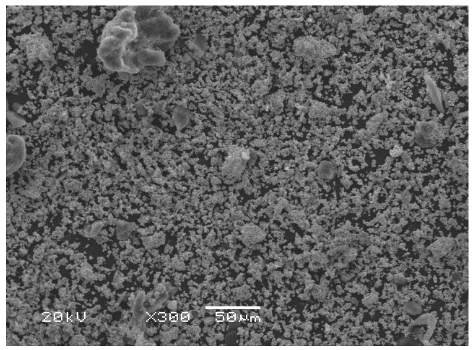


Figure 8: WC-10Co mixture (300X).

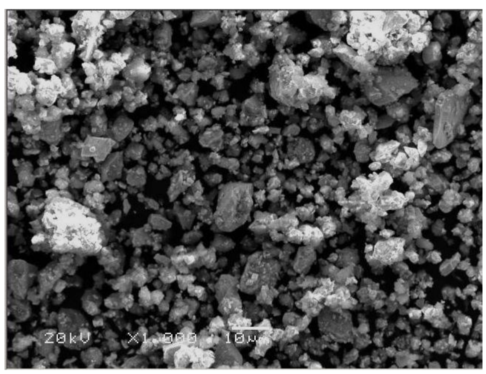

Figure 9: WC-10Co mixture (1000 X).

Figure 10 shows a magnification of 300x showing a homogeneous mixture of WC-15Co, Figure 11 shows a magnification of 4000X, showing also the homogeneity of the composite, where is possible see the cobalt added in the mixture. Also shows the point where we performed the EDS and Figure 12 shows the result of EDS.

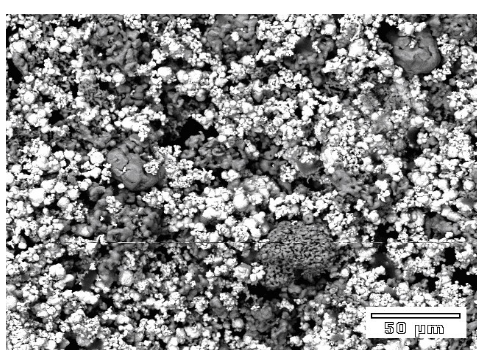


Figure 10: WC-15Co mixture (300X).

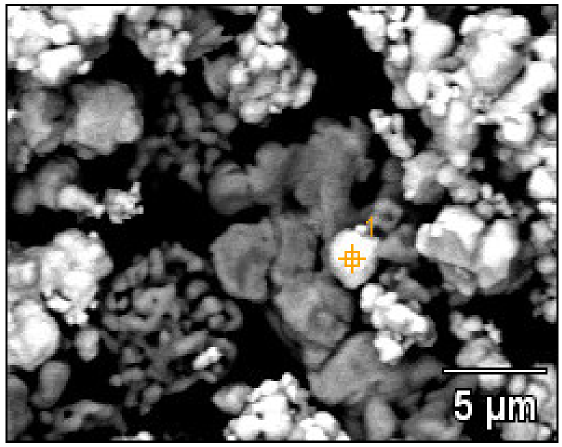


Figure 11: WC-15Co mixture (4000 X).

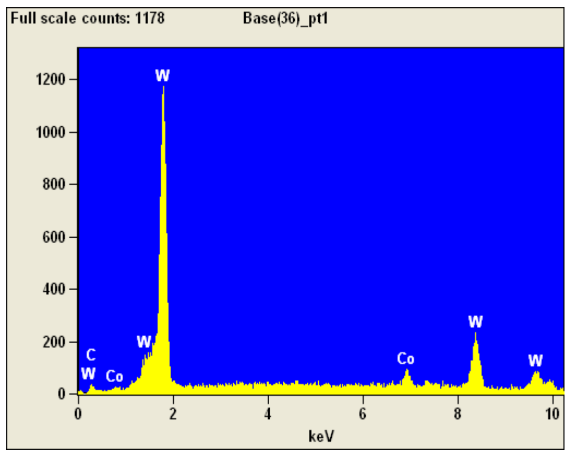


Figure 12: WC-15Co composite EDS analyses.

Figure 13 shows a magnification of 500x showing a homogeneous WC-20Co mixture, Figure 14 shows a magnification of 2500X, showing also the homogeneity of the composite, where is possible see the cobalt added in the mixture.

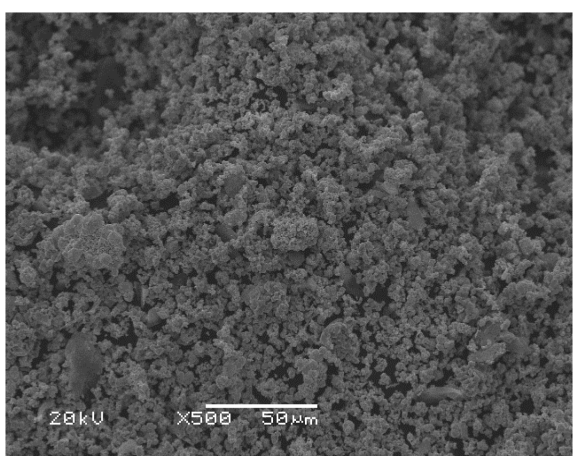


Figure 13: WC-20Co mixture (500 X).

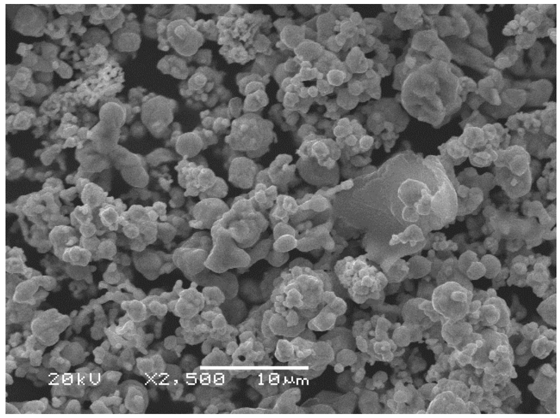


Figure 14: WC-20Co mixture (2500 X).

After the sintering process has been made micrograph analysis with increased 3000x to examine the microstructure of the WC-6Co composite sintered and new composites processed in the blender "Y". Figure 15 show the micrograph of the WC-6Co composites sintered to 1450ºC. Figure 16 shows the micrograph of the WC-10Co composite sintered to 1420°C.

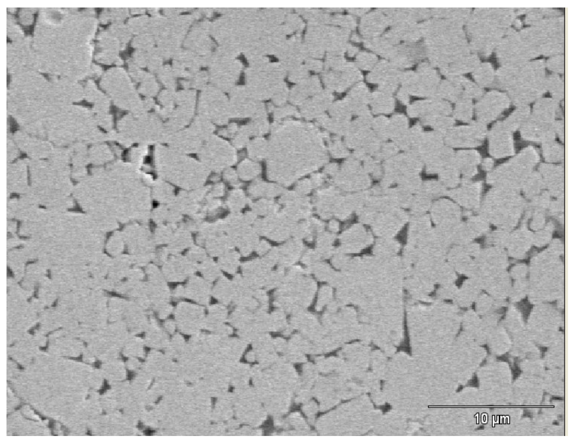


Figure 15: WC-6Co microstructure (3000 X).

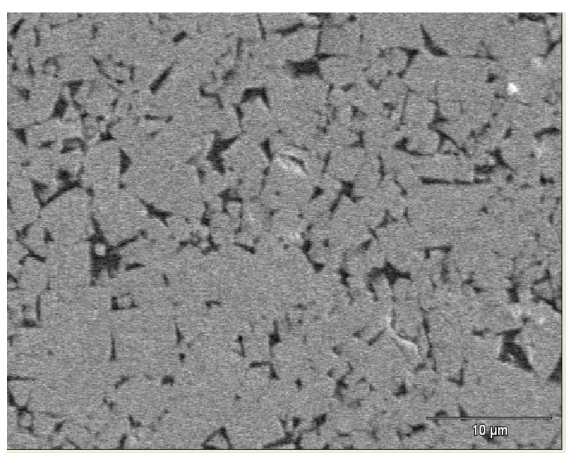


Figure 16: WC-10Co microstructure (3000 X).

Figure 17 shows the micrograph of the composite sintered WC-15Co to 1400°C. Figure 19 shows the micrograph of the WC-20Co composite sintered to 1360°C.

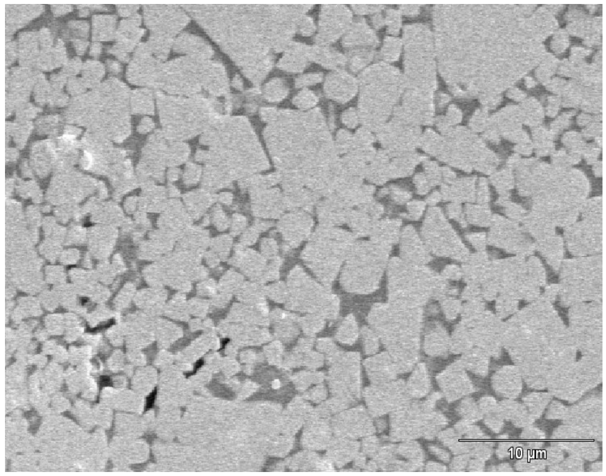


Figure 17: WC-15Co microstructure (3000 X).

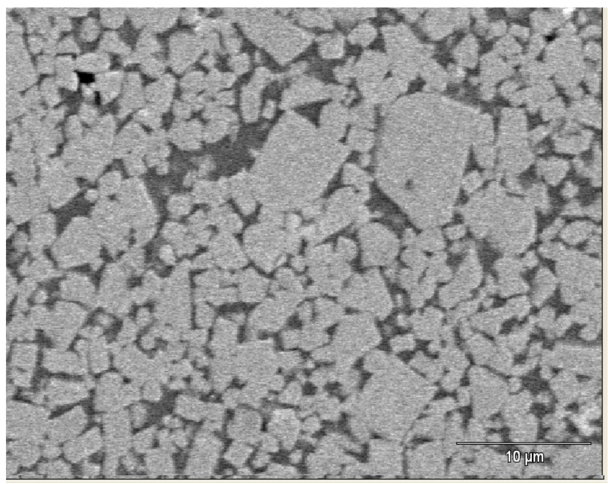


Figure 18: WC-20Co microstructure (3000 X).

**4. DISCUSSION**

Focusing on analyzing the efficiency of mass balance with the addition of cobalt less than 37µm in the 94WC-6Co composite, the apparent density of composites processed in a blender "Y", which noted that the apparent density was between 2,45 and 2,63 g/cm³. Through analysis of the mixture with the scanning electron microscope was observed the homogeneity of the composites with good dispersion of cobalt on the commercial composite. After compaction of the four stoichiometric balanced composite with the same pressure of 200MPa, and masses near, obtained was a green density, which decreases gradually and proportionally with the increase in the percentage of cobalt from hard metal composite.

The green density of commercial composite ranged between 7,91 and 8,06 g/cm ³. The range green density of the composite with 10% of cobalt ranged between 7,31 and 7,50 g/cm³. The WC-15Co composite obtained a green density ranged between 7,13 and 7,25 g/cm³. The green density of the WC-20Co composite varied between 6,80 and 7,04 g/cm³. The composite with 20% of cobalt was sintered at a temperature of 1360°C, and through the sintered density and micro-hardness in Vickers, was observed that the values approaching the values in the literature. For the WC-15Co composite used the temperature of 1400ºC, noting that the values of density and microhardness are also approaching the values of literature.

The temperature of 1420°C was used to sinter the composite with 10% of cobalt, and values of density and microhardness are approaching the values of literature. Consistency was observed comparing the values of the sintered density and microhardness with the literature, thus proving the obtaining process efficiency the composite by mass balance. The micrograph showed that the high rate of heating curve optimized works perfectly. The hard metal composite showed characteristic indicated as literature.

The micrograph analysis of hard metal composite shows the grains homogeneous distribution of the tungsten carbide and cobalt, indicating efficiency in the mass balance according to the temperature of sintering, thus validating the curve with its rate of thermal heating and cooling. Micrograph was also a comparative analysis, showing clearly that the addition of cobalt is well distributed on the samples during sintering, showing clearly that there was a gradual decrease in the amount of cobalt between samples as show the figures 15, 16, 17 and 18. The micrograph shows the efficiency of the process.

## 5 CONCLUSION

The sintered density, the micrograph and microhardness are evidence for the obtaining method validation of a mixture, from a commercial hard metal composite through the balance of mass. The properties of hard metal composites from a mixture processed, with the addition of cobalt in the blender "Y" in a conventional sintering on the commercial composites sintered were equivalent. The cost will be an important factor to validate the process at industrial, taking commercial hard-metal powder and elemental cobalt powders can result in a composite, with any percentage of cobalt over the commercial, not to mention, the reduction of the direct cost of high-energy milling in an industrial process.

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