EFFECT OF SINTERING TEMPERATURE ON TUNGSTEN CARBIDE DENSITIES AND POROSITY

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Abstract— *The manufacture of tungsten carbide begins with mixing WC with Co in the milling process and then the compaction process is carried out using a compacting machine. After that, the sintering process is carried out to increase the strength of the material. From various studies it was found that the sintering temperature had an effect on the density of the tungsten carbide produced. The problem is what is the best sintering temperature for WC (Tungsten carbide). This study was intended to analyze the effect of temperature on the density and porosity of tungsten carbide. The research was carried out with a compaction press load of 20 tons with 4 variations of sintering temperature and heating electric current strength, namely 50A -600°C, 60A -800°C, 70A -1000°C and 80A -1200°C. The results of this study prove that the WC sintering temperature has a significant effect on density and porosity. The highest average density of 9.4763 g/ml was achieved by specimens with a sintering temperature of 1.000°C and a current of 70A. Microstructural observations using SEM-EDX also proved that this specimen had the best density compared to other specimens.*

Keywords: *Arc Plasma Sintering, SEM-EDX Test*

1. INTRODUCTION

Tungsten carbide (WC) is a hard and wear-resistant material, having a high melting point [1]. In its most basic form, Tungsten carbide (WC) is a fine gray powder, but it can be pressed and formed into shapes through a process called sintering for use in industrial machinery such as cutting tools, grinding wheels, bearings, textile machinery components, oxidation-resistant gas burners, and many others [2][3].

The most popular cutting tool material, WC is usually produced with an admixture of cobalt (Co) (the binder that holds WC together), with many variations in carbide grain size and carbide to binder ratio [4]. Tungsten carbide is most commonly used for cutting tools with the addition of 3 to 12 percent by mass Co. Iron and nickel can also be used as alternatives to Co, but Co is more suitable due to the higher solubility of WC in it. It is the nature of cobalt that allows WC to dissolve well in the cobalt matrix. This means that when WC is mixed with cobalt in the manufacturing process of materials such as hard carbides, the WC particles can be better dispersed and bonded in the cobalt matrix. The result is a strong and wear-resistant material. Nickel is most often used only as an additive to Co, as it stabilizes the relatively ductile cubic face-centered phase. It also prevents the transformation of this phase into the less ductile hexagonal phase during deformation. Chromium is often added to inhibit the growth of WC crystal grains during sintering [5][6].

WC materials are mostly recognized for their application as tool materials for machining applications [7] [8]. Pure WC is attractive for a wide range of applications (cutting tools, molds, special indentation items etc.) due to a combination of physical properties (high melting point, high hardness, low friction coefficient, and chemical stability against corrosion and oxidation) [9]. WC has a very high hardness due to the presence of carbide particles. With sufficient amount and proper binder distribution, they also have quite good toughness. [5] .

In recent years innovations in the manufacture of WC with tungsten carbide nano powder composition on density, structure, and mechanical properties made by the SPS (Spark Plasma Sntering) method have encouraged researchers and industry to develop materials / composites with better properties [10]. For this reason, I would like to try to develop the above by using a sintering tool called Arc Plasma Sintering.

Arc Plasma Sintering (APS) technology is a new sintering technology with the use of plasma generated by the ionization of Argon gas between the potential difference between the anode and the cathode with an impulse current, the temperature that can be established on it can reach more than 3000°C. The use of Argon as a process gas shares certain advantages, which is that it can coincidentally share protection against materials from the influence of oxidation, so APS does not require special efforts to protect the workpiece from the threat of oxidation or other air influences [11] [12] [13].

In this study, we will discuss the effect of sintering temperature on the densities of tungsten carbide. This can be seen from the characteristics of WC material on microstructure.

2. METHODOLOGY

After material preparation, the research phase continued by reading literature related to the research. Then the grinding / milling process, after that it is compressed and then the material is heated using APS (600-1200)°C. testing which includes SEM-EDX test. Then the test data is analyzed, and a discussion of the test data is carried out. The last step is to draw conclusions from the research that has been done.

In this study, WC material was used from Alfa Aesar Thermo Fisher Scientific Chemicals, Inc. And Co Metal Powder (LAB) Extra Pure LOBA CHEMIE PVT. Ltd. A total of 16 g of powders (WC-8.8 g; Co-7.2 g) were milled in the presence of 10 balls with a mass of 21.1 g. The milling was carried out at 700 rpm for 5 minutes. Milling was carried out at 700 rpm for 5 hours.

This process is the WC-Co material that has been mixed in the grinding process, then the next process is the compaction process. In this study, WC and Co materials were compressed which were previously weighed with a composition of 55% WC and 45% Co, then the powder was pressed using a press with a pressure of 20 tons. The pressed results (pellets) were then heated using APS with 4 current conditions, the first 50A - 600°C, the second 60A - 800°C, the third 70A - 1,000°C and the fourth 80A - 1,200°C for 1 minute..

In SEM, the signal generated does not only come from the electrons it fires, but can also come from other interactions that occur within the sample close to the surface. SEM is capable of producing images with very high resolution. The image magnification ranges from 15 times to 200000 times. SEM was first invented by Manfred von Ardenne in 1937 [14]. Further preparations must be made in advance to observe the metal surface metallographically:

1. Cutting test specimens At this stage it is expected that the test specimens are in a flat state so as to facilitate observation.
2. Specimen mounting is only done on thin or small materials at this mounting stage, if needed. While thick materials do not need to be installed.
3. Grinding and polishing the purpose of this stage is to flatten the entire surface of the test specimen by shaping it during the grinding and polishing process. The specimen is ground by rubbing it on scouring paper with increasingly finer grid sizes (grid 320 to 320) on a hand grinding machine. During the process, the specimen is polished by rubbing it on a polishing tool that has a wool cloth coated with alumina powder with a fineness of 1-0.05 microns. Alumina powder is added to the specimen to further smoothen its surface and make
4. Mengalami korosi atau menjadi tidak rata kembali pada proses etsa. Permukaan metallography easier..

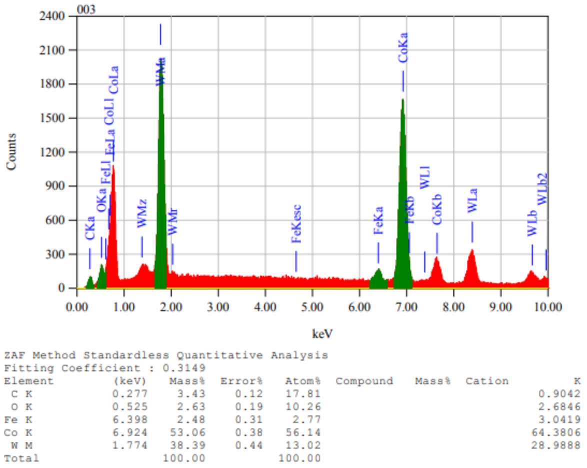
The surface of a specimen that becomes flat as a result of the grinding and polishing process is essentially an uneven specimen because different microstructures dissolve at varying rates, leaving surface marks with varying angular orientations. Actually the etching procedure is carried out by dipping the specimen into an etching liquid.

3. RESULT AND DISCUSSION

In this research, WC material is used from Alfa Aesar Thermo Fisher Scientific Chemicals, Inc. And Co Metal Powder (LAB) Extra Pure LOBA CHEMIE PVT. Ltd. Then it was done by mixing the powders using a High Energy Milling (HEM) tool on a ball mill with a diameter of 10 mm. The composition of 55% WC powder and 45% Co was ground in the presence of 10 balls with a mass of 21.1g. Milling was carried out at 700 rpm for 5 hours.

WC Co materials that have been HEM then compressed weighing 4 grams per sample, then the powder is pressed using a press with a pressure of 20 tons. The pressed results (pellets) were then heated using APS with 4 current conditions, the first 50A - 600°C, second 60A - 800°C, third 70A - 1,000°C and fourth 80A – 1.200°C for 1 minute.

Elemental analysis and elemental distribution tests were carried out using SEM-EDX at the Puspiptek/BRIN Serpong laboratory. Chemical element composition analysis is the result of EDX analysis discussed in Figure 1 cross-section of the sample surface in WC and Co..



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ZAF Method Standardless Quantitative Analysis
Fitting Coefficient : 0.3149
Element         (keV)   Mass%  Error%   Atom%  Compound   Mass%  Cation          K
 C K            0.277    3.43    0.12   17.81                               0.9042
 O K            0.525    2.63    0.19   10.26                               2.6846
Fe K            6.398    2.48    0.31    2.77                               3.0419
Co K            6.924   53.06    0.38   56.14                              64.3806
 W M            1.774   38.39    0.44   13.02                              28.9888
Total                  100.00          100.00
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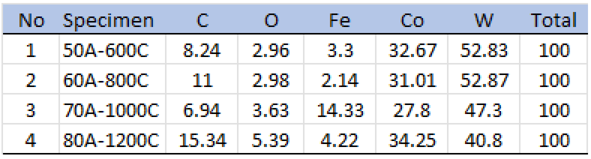
Figure 1. SEM-10,000X and EDX spectra of WC-Co alloy material, temperature 1200 oC

SEM-EDX test The elemental composition on the surface of the samples obtained is shown in Table.1 successively influenced by sintering temperature and sintering holding time. :

Table 1. Elemental mapping using SEM-EDX of the effect of sintering temperature in WC-Co alloy



No	Specimen	C	O	Fe	Co	W	Total
1	50A-600C	8.24	2.96	3.3	32.67	52.83	100
2	60A-800C	11	2.98	2.14	31.01	52.87	100
3	70A-1000C	6.94	3.63	14.33	27.8	47.3	100
4	80A-1200C	15.34	5.39	4.22	34.25	40.8	100

In specimens with a sintering temperature of 50A -600 ° C, the elements that dominate the alloy of WC and Co with the largest value to the smallest is oxygen, carbon and iron. Elements of WC and Co became the most widely distributed elements evenly distributed after the per-spot test. Then the second sample of WC and Co alloy sintering temperature 60A -800 ° C elements formed the same as the sample temperature 50A - 600 ° C elements are the most Oxygen, carbon and iron dominate from the largest. The sample with a sintering temperature of 70A -1000°C which is the third sample, has the same alloy element composition as the previous two samples except in the number of very small percentage values of oxygen. The percentage value of the largest is oxygen, carbon and iron. The sample with a sintering temperature of 80A - 1200 ° C which is the fourth sample, has the same arrangement of alloying elements as the previous sample except in a very small percentage of iron values. The percentage values of the largest are oxygen, carbon and iron..

Figure 2. shows that the inner surface of WC-Co samples sintered using APS has a significant effect of sintering temperature during the change in WC density and especially on the quality of cobalt binder distribution in the sample volume. The lowest sintering temperature chosen (50A - 600°C) leads to good compaction of the sintered surface as evidenced by significant pore and cavity shapes (up to several microns in size). At the same time, the cobalt binder entering the sample limits its entry into the areas between the WC grains. Sintering in the range 70A – 1.000°C - 80A – 1.200°C as a result of the intensification of the solidification of the sintering process, which results in a qualitative change in the microstructure of the alloy, due to a reduction in the number of pores and an increase in density with the achievement of the best indicators of cobalt binder distribution at sintering at 70A – 1.000°C.

Density calculation inspection process. Table.2 shows the WC-Co alloy used in the study with variations of sintering temperature: (a) 50A-600°C; (b) 60A-800°C; (c) 70A-1000°C; (d) 80A-1200°C, the sample is to determine the density and porosity. The process of examining the calculation of density and porosity, WC-Co alloy that has been sintered using APS tetrlihat that sintering at 80A - 1200 ° C has a density of 9.7463 g / ml and porosity of 60.61%. Related to the fact that the mass transfer process aims to reduce the energy of the system by reducing the intergranular boundary. In sintering, the WC-Co alloy powder composition at 80A - 1,200°C is the temperature generated directly by APS automation and also measured from the mold surface using a thermocouple.

Table 2. Specifications of WC-Co samples used in the study

No	Sample	Average density (*gr/ml*)	Percentage
1	600°C	5.2993	69.35%
2	800°C	5.5687	64.45%
3	1000°C	6.1698	63.03%
4	1200°C	9.7463	60.61%

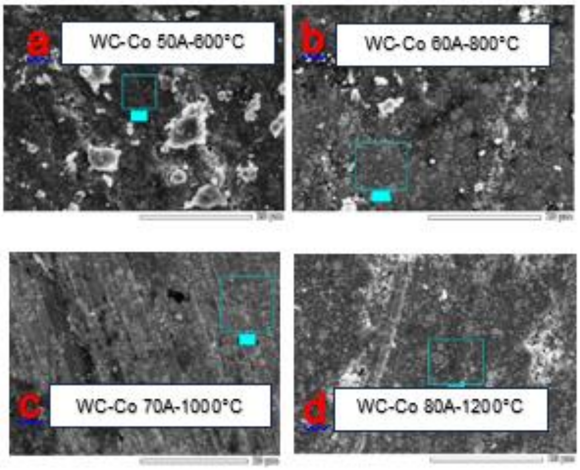


Figure 2. SEM-EDX results of WC-Co alloy obtained via APS at various sintering temperatures: (a) 50A-600°C; (b) 60A-800°C; (c) 70A-1000°C; (d) 80A-1200°C.

4. CONCLUSION

From the test results and analysis of the research data, it can be concluded: WC sintering products with Co mixture using APS through SEM-EDX test by varying the temperature of 50A-600°C, 60A-800°C, 70A-1000°C, 80A-1200°C. that the sintering temperature of WC significantly affects the density and porosity. The highest average density of 9.4763 g/ml and porosity of 60.61% was achieved by specimens with a sintering temperature of 1200°C and a current of 80A. The results of microstructure observation using SEM-EDX also prove that this specimen has the best density compared to other specimens..

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