

Study on Properties of Heat-treated WC-6Co with Different Wt. % of TiC Powder

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ABSTRACT

The WC-6Co cemented carbide with different composition of Titanium Carbide (TiC) powder (0.5 wt. %, 0.75 wt. %, 1.0 wt. %) were fabricated by powder compaction method. The effect of different heat treatment holding time and TiC addition on the properties of the treated sample of WC-6Co were studied. WC-6Co metal powder were mixed together with the TiC powder using ball mill mixer before proceeding with the hydraulic compression process to produce a green body. The compacted sample then being sintered at 378°C with a different holding time (15 min, 30 min and 45 min). Based on the result obtained, it was concluded that the optimal sintering holding time is 30 min. At 30 min, WC-0.75TiC-6Co produced the best properties as the value of density, hardness and grain size were 10.2185 g/cm³, 102.3966 HV and 33.61 μm respectively. The TiC addition appeared to be beneficial up to 0.75 wt. %.

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1. Introduction

WC-6Co cemented carbides are widely used in heavy industry as drilling tools, metal cutting tools and wear-resistant parts owing to their superior properties such as high hardness level, high corrosion resistance and low thermal expansion coefficients [1-4]. Among these applications, wear resistance is very essential, and it has been proved that wear resistance improved with increasing hardness [5-7]. As a result, previous researcher has been thoroughly investigated on hardness as a function of the composition and microstructure and of WC-Co [8-11]. Besides, the WC grains growth during sintering of WC-Co is important to be control since the size distribution of grain have a critical effect on many properties. For example, as the grain size decreases, most of the mechanical properties of the alloy increase [12-14].

Since carbides are effective grain growth inhibitors (GGI), some researchers reported that the addition these carbides such as VC, Cr₃C₂, NbC, TaC, TiC and ZrC on cemented carbide significantly improve the hardness and flexural strength based on ability to strengthen the binder by solid solution strengthening [15,16]. So far, the most successful way to control the grain growth of WC is

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the addition of small amounts of these grain growth inhibitors into the original powder mixture typically less than 1.0 wt. % of a metallic carbide [17,18]. Among these alloys, TiC particle reinforced tungsten is the most superior GGI owing to its high bending strength, desirable impact ductility, excellent high temperature tensile strength, and outstanding irradiation resistance [19-21]. Previous study by [22] investigated the effectiveness of TiC as a grain growth inhibitor in the WC–VC–TiC–Co stated that the average grain size of the cubic carbides decreased significantly from 2.0 μm to 1.7 μm and the grain size distribution became narrower with the addition of TiC GGI, and the HV₃₀ hardness improved from 1519 to 1549.

In this work, WC-TiC-6Co hard alloys with different composition of TiC content were prepared under heat treatment process via the powder metallurgy method. The objective of present work is to establish the influence of the TiC addition and heat treatment time on the mechanical properties and grain size of WC-TiC-6Co hard alloys.

2. Experimental

In this study, various heat treatment holding time were examined in order to find the optimal parameters of WC–6Co, as well as to compare the different TiC composition in the cemented carbide. The ratios of the powder mixtures are listed in Table 1. The powders WC–0.5TiC–6Co, WC–0.75TiC–6Co and WC–1.0TiC–6Co were mixed using ball milling machine for an hour under 100rpm. To form a sample with a diameter of 14mm, the mixed powders were poured into a stainless steel die before being pressed by hydraulic at 6 tons for 5 min. After compaction, the specimens were heat treated at 378°C by using different holding time (15 min, 30 min and 45 min) according to Figure 1. Samples were polished and tested for mechanical properties and microstructure. Density of the cemented carbide was measured by the Archimedes method using the formula in the Eq. (1). The hardness test was performed on the polished surface using a Vickers diamond pyramid indenter with a load of 980.7mN (0.1HV) and a loading holding time of 10 s. The Vickers hardness (HV), were calculated using Eq. (2). In addition, phase identification of the heat-treated material was done through X-ray diffraction (XRD). The average grain size was statistically analyzed based on 500-800 grains. Chemical composition was examined by an energy dispersive spectroscope (EDS) using SEM microscopy.

Table 1
 The nominal compositions in %wt. of the sample

Sample	WC-6Co	TiC
WC-6Co	100.0	0
WC-0.5TiC-6Co	99.5	0.5
WC-0.75TiC-6Co	99.25	0.75
WC-1.0TiC-6Co	99.0	1.0

$$\text{Bulk Density} = \frac{W_d}{(W_d - W_s)} \quad (1)$$

where

W_d = Weight of specimen in air

W_s = Weight of specimen in liquid

The Vickers micro hardness calculation formula

$$\text{Vickers Hardness} = 0.102 \left(\frac{0.2 F \sin \frac{136}{2}}{d^2} \right) \quad (2)$$

where F (N) is the indentation force, d (m) is the average diagonal length.

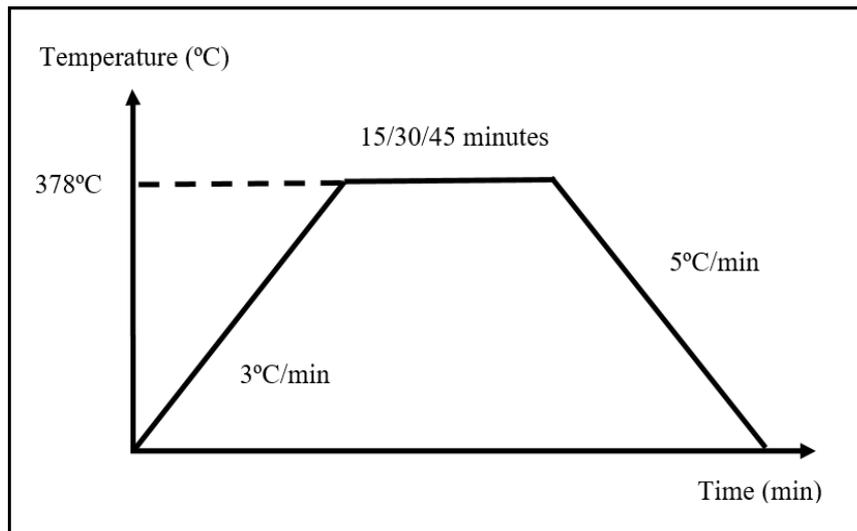


Fig. 1. The heat treatment profile for the preparation of WC-TiC-6Co

3. Results and Discussion

The density was done to get the value of density of the sample by applying the Archimedes principle. Optical microscopy analysis was conducted to measure the grain size of the sintered samples and XRD and EDS was conducted to determine the element present in the samples after finished sintered. All the test result obtained was recorded and discussed.

3.1 Mechanical Properties

Figure 2 shows the density result versus holding time which represents the result of density at different composition. Based on the Figure 2, the WC-0.5TiC-6Co metals shows the highest density at the sintering holding time of 45 min with the value of 10.6561 g/cm^3 . While at sintering holding time of 15min, WC-1.0TiC-6Co shows the lowest value of density of 9.3896 g/cm^3 . Study by [23] on microstructure and mechanical properties with Cr_3C_2 and TaC addition on ultrafine WC-10wt%Co on cemented carbides shows that the decrease of strength at the grain boundaries is due to the excessive Cr enrichment at WC/Co and WC/WC grain boundaries. However, each of the sample have the lowest value of density when the sintering holding time is 15 min. That is because of the densification of the sample was not fully complete due to the presence of pores. The highest density of the cemented carbide was 10.6561 g/cm^3 slightly lower than that observed in several WC-Co cemented carbides (approximately $14.2 \pm 0.4 \text{ g/cm}^3$) process by powder metallurgy [24]. The sample with composition of 1.0 wt.% shows the lowest density value compared to the composition of 0.5 wt.% and 0.75 wt.%. This result was equivalent from the previous researcher that using Cr_3C_2 as a GGI stated that when the content of Cr reaches up to 0.8 wt.%, the density of WC-10 wt.%Co cemented carbides starts to decrease. It is found that when the Cr content exceeds its saturated concentration in binder phase, not only the solution/re-precipitation of WC is hindered but also excessive Cr reduces the particle rearrangement and wetting behaviour at the grain boundaries. This will subsequently reduce the density of the alloy.

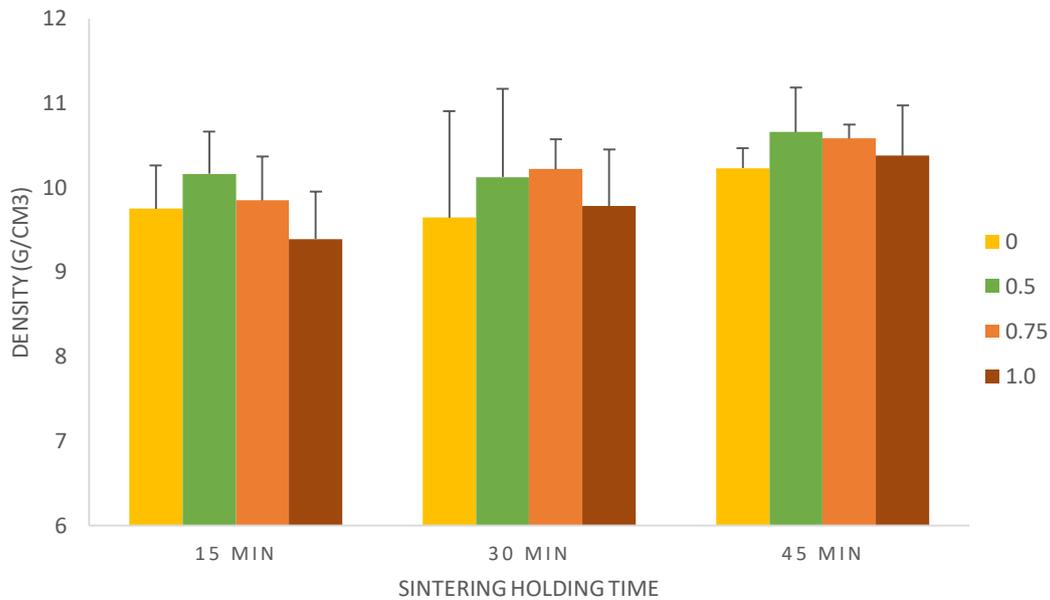


Fig. 2. The density result versus holding time

From the Figure 3, WC-0.5TiC-6Co shows the highest value of hardness with the value of 133.65 HV followed by WC-0.75TiC-6Co with the value of 106.387 HV and WC-1.0TiC-6Co with the value of 104.631 HV. The pure WC-6Co obtained 102.68 HV of hardness value which is lower compared to other three composition. Previous study by Lin *et al.*, [25] shows that the addition of TiC in hard metals could enhance the hardness and wear resistance of WC-Co cemented carbide while [26] claimed that the hardness and wear-resistance of WC-TiC-Co alloy are generally better than WC-Co cemented carbide due to the fact that resistance properties of titanium carbide is higher than that of WC-Co. However, the result shows the lower value in hardness with further increasing of TiC. This might be happened due to nonuniform segregation of the TiC particles within the sample. The value of hardness obtained for the WC-1.0TiC-6Co which is lower compared to WC-0.5TiC-6Co and WC-10.75TiC-6Co. Therefore, the amount of added GGI should be in optimized range in order to get better hardness sample [27]. TiC addition appeared to be still beneficial, up to 0.75 wt. %.

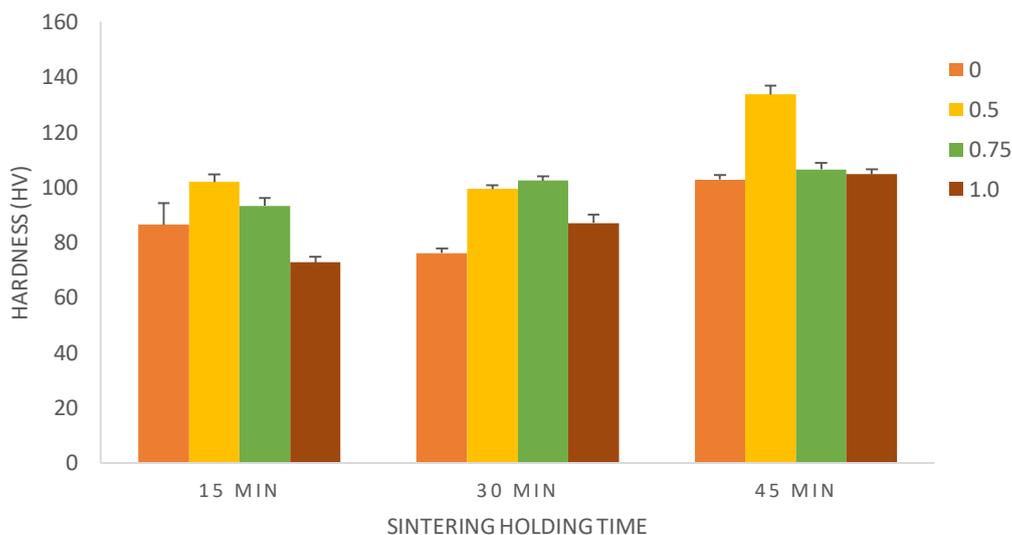


Fig. 3. The hardness value versus holding time

3.2 Phase Analysis

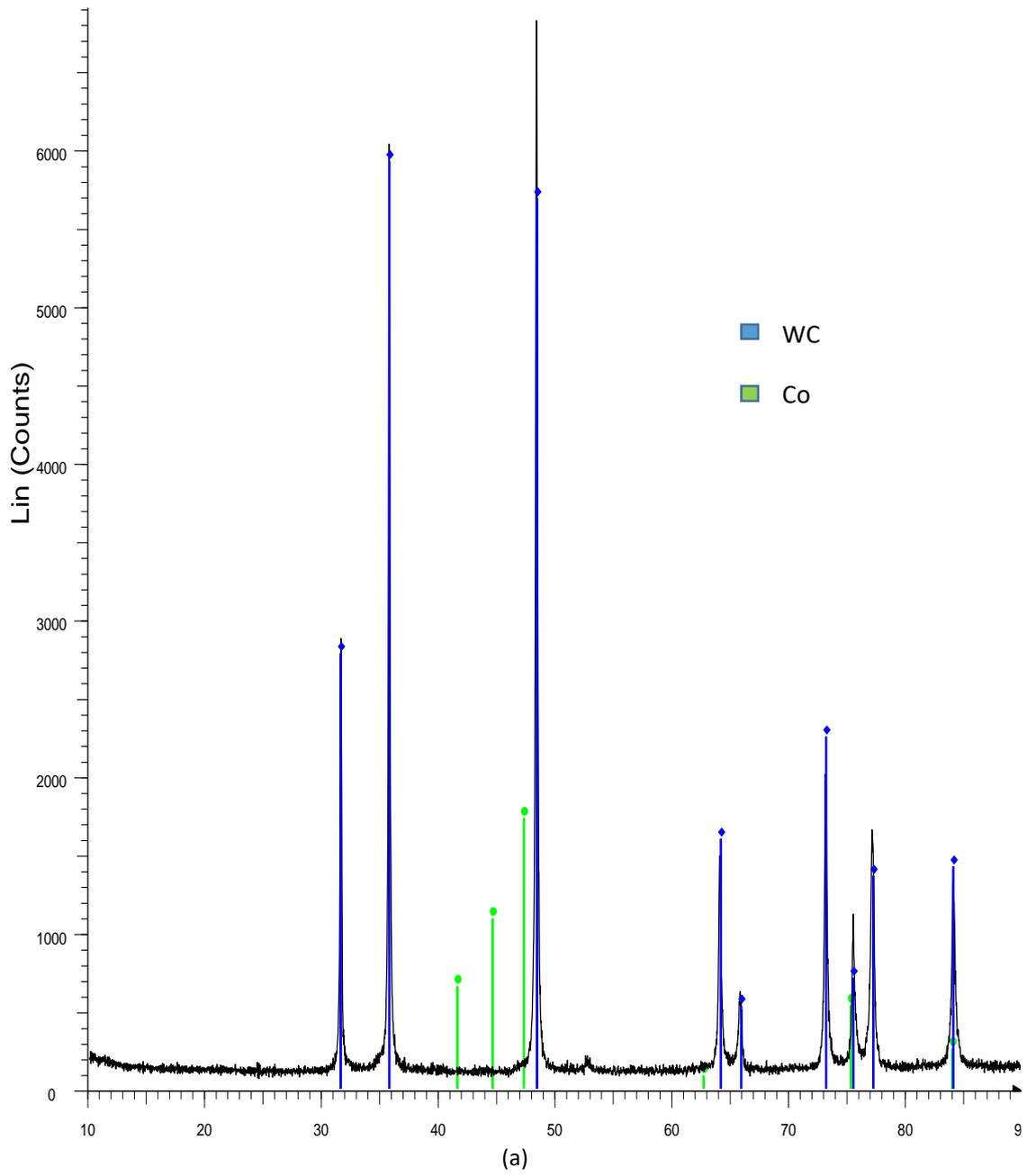
The XRD patterns of heat-treated sample with different holding time are shown in Figure 4(a)-(c). Only the diffraction peaks of WC (PDF 00-061-0244) and Co (PDF 00-001-1278) phases present in the XRD patterns and no carbon or η phases appearing in the specimens. All the samples contain only two observable primary phases. The phase related to trace element (<1 wt. %) such as Ti and C observed in the EDS analysis are not found in the X-ray diffraction pattern present in Figure 4 as could be that the amount of grain growth inhibitors and secondary phases in samples, if fall below the detection limit of the XRD[28]. A previous study by Chang and Chen [29], pointed out that during the high-temperature heating process, specimens are prone to the free carbon and η phase that result from escaping and accumulating carbides. However, in this study, no generation of any impurities or η phases in the heating process since the temperature is not too high.

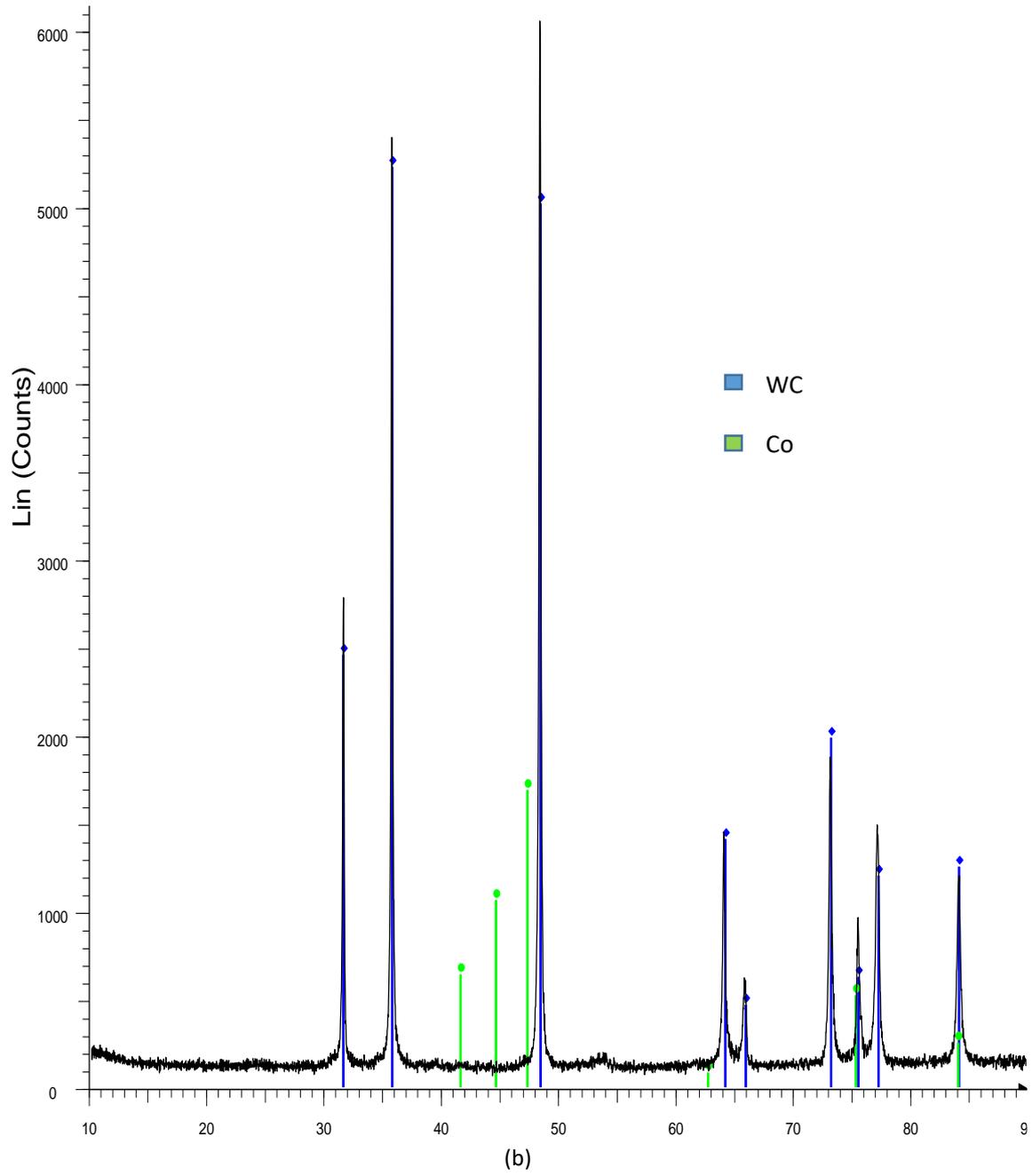
All the elements that presence to the sample was compiled in Table 2 which represent the analysis at each holding time. The picture of the EDS analysis was set to the magnification of 1.0KX for each sample. From the result obtain, it was found that all the samples were oxidize during the sintering process because of the presence of the Oxygen at all samples. This is because of the sample was sintered by using Box Furnace, which the Oxygen was exposed to the samples and react so that oxidation occurs. In addition, the oxidation will affect the strength of the samples which can cause of the damage and defect of the samples [30]. There are also impurities elements contained in the sample such as Aluminium (Al) but the presence was not significant, since the percentage is too small and can be ignored. This element is present due to the contamination while handling the samples and at the atmosphere during sintering process.

3.3 Grain Size Measurement

The total grain size that measured in each sample was in between 500 to 800 with the total percentage area of 90%. The average result of the grain size was plotted at the graph in order to compare are shown in Figure 5. Based on Figure 5, the average grain size of the sintered samples in all composition were in the range of 30 μ m to 60 μ m.

At the sintering holding time of 30 min, WC-0.75TiC-6Co produced the smallest grain size compared to the other samples while WC-1.0TiC-6Co produced the largest grain size amongst all. The WC-6Co average grain size while adding 0.5 wt. % of TiC is narrow and the average grain size reduce is also reduce with the addition of 0.75 wt. % TiC in the sample. However, with additional 1.0 wt. % of TiC, the average grain size is increase to 53.38 μ m. It can also be seen clearly that the grain size is larger at the composition of 1.0 wt. % of TiC. The average grain size was almost similar to each other and higher compared to others. During the sintering holding time of 30min, the effectiveness of the grain growth inhibitor can be seen when the average grain size value for 0.5 wt. % and 0.75 wt. % are 37.36 μ m and 33.61 μ m respectively while for WC-6Co, the value of the average grain size is 46.50 μ m which is larger compared to other two.





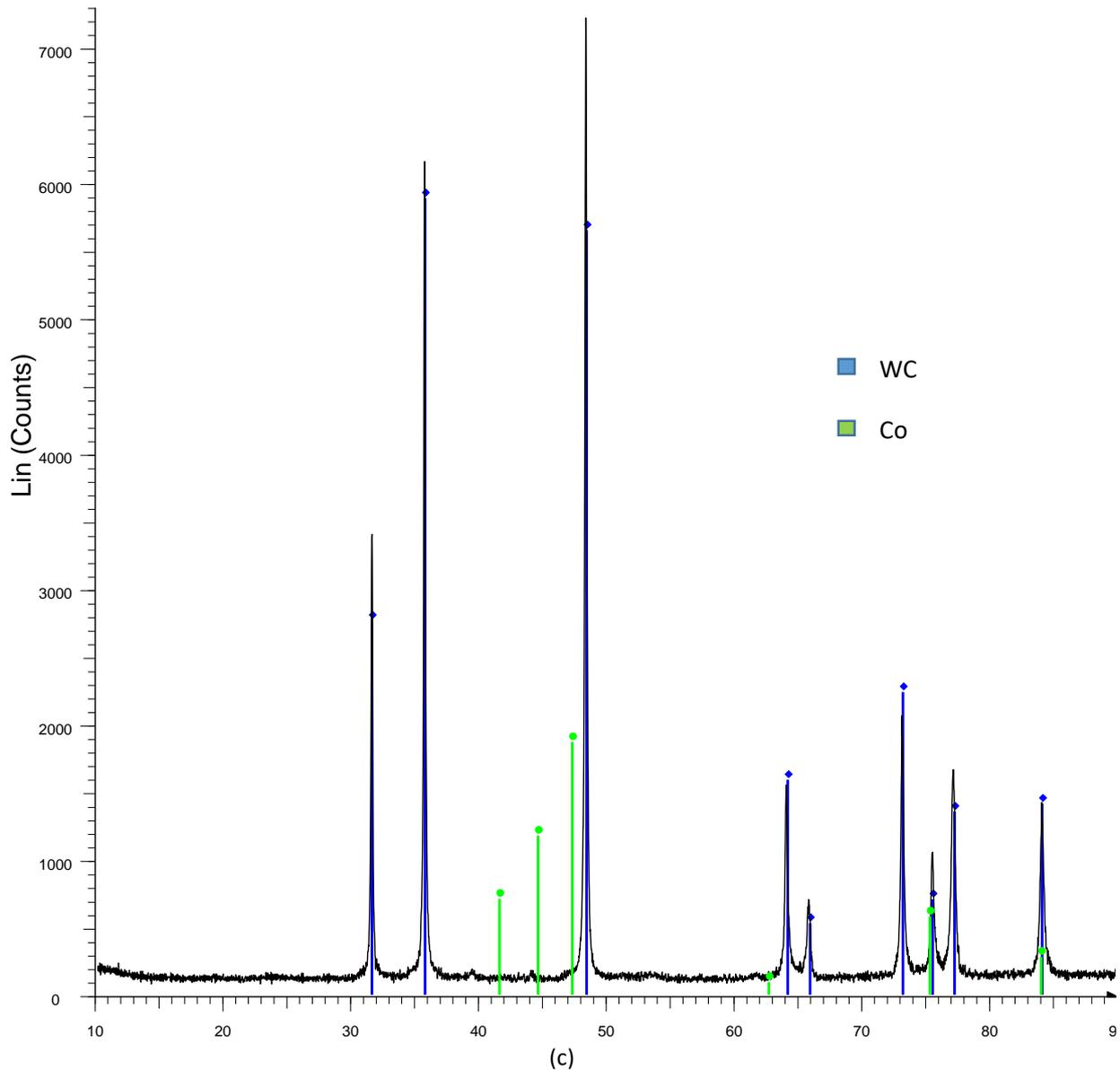


Fig. 4. (a) Result of XRD diffraction phase analysis of WC-1.0 wt. % TiC-6Co during different holding time 15min (b) Result of XRD diffraction phase analysis of WC-1.0 wt. % TiC-6Co during different holding time 30min (c) Result of XRD diffraction phase analysis of WC-1.0 wt. % TiC-6Co during different holding time 45min

Table 2

Element presence at samples for EDS analysis at sintering holding time of 15 min, 30 min and 45min

Element	Composition (wt. %)			
	Pure	0.5	0.75	1.0
Carbide (C)	√	√	√	√
Oxygen (O)	√	√	√	√
Titanium (Ti)	√	√	√	√
Carbide (Co)	√	√	√	√
Tungsten (W)	√	√	√	√

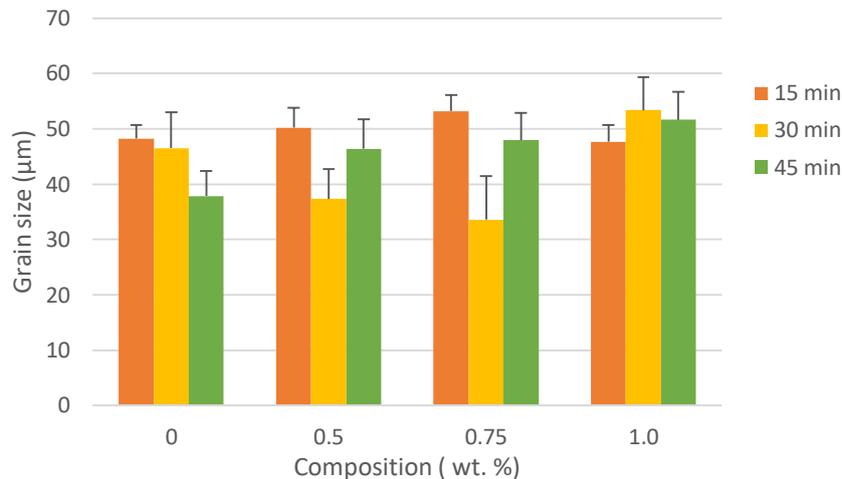


Fig. 5. The average result of the grain size with the composition of 0.5, 0.75 and 1.0 of weight percent (wt. %) at the heating holding time of 15 min, 30 min and 45 min

4. Conclusion

This paper summarizes experimental investigations carried out for heat treated sample of WC-6Co metal powder. From the analysis, the compatible heat treatment holding time for the WC-6Co powder is 30 min to produce a product with a high value in density and hardness followed by smaller grain size. The optimum composition of the grain growth inhibitor in producing WC-TiC-6Co is 0.75 wt. %. This is because by using 0.75 wt. % of TiC at 30 min holding time, the density value obtained is 10.2185 g/cm^3 improved by 5.97% while for hardness, the value is improving about 34.6% which is increased from 76.0764 HV to 102.3966 HV compared to WC-6Co. In addition, at this composition, the grain size obtained is $33.61 \mu\text{m}$. However, when the content of 1.0 wt. % TiC is added, the value of the density and hardness is reduced while the grain size produced is the largest amongst all. An excessive addition of TiC does not give a significant effect towards the blends which can reduce the overall properties.

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