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## Fracture toughness evaluation of WC–10 wt% Co hardmetal sintered under high pressure and high temperature

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### ABSTRACT

This work focused on fracture toughness studies of WC–10 wt% Co hardmetal fabricated through the high pressure/high-temperature technique. A powder mixture of WC–10 wt% Co was sintered at 1500–1900°C under a pressure of 7.7 GPa for 2 and 3 min. Vickers hardness test at two different loads of 15 and 30 kgf was done and fracture toughness of the sintered bodies was measured using the indentation method to obtain the effect of sintering parameters. Structural analyses were also performed via X-ray diffraction to investigate structure-related properties. Full density was achieved for high sintering temperature along with abnormal grain growth that reduced hardness. High hardness was observed ranging from 1200 to 1670 HV and fracture toughness increased with increasing sintering temperature up to the highest value of 17.85 MPa/m<sup>1/2</sup>.

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### KEYWORDS

Hardmetals; high pressure; high temperature; fracture toughness; grain growth; WC

## 1. Introduction

WC–Co cemented carbides are widely used as cutting, machining and rock drilling tools due to their excessive hardness and strength characteristics, good fracture toughness and wear resistance over a wide range of temperatures [1]. It has become the workhorse for hardmetal component applications today with 98% of hardmetal components made of cemented WC–Co [2]. The conventional production route is through powder metallurgy, where the main steps are: ball milling mixtures of WC and Co powder in proper media; drying, pressing, debinding and liquid phase sintering in the temperature range 1400–1500°C [3,4]. Effects of Co content, WC mean grain size and origin of WC powders on mechanical and performance properties of cemented carbides were examined in numerous publications and these results are summarized in [5,6].

WC grain growth is a common issue in cemented carbide sintering due to its effects on mechanical properties such as fracture toughness. This problem is discussed in various studies, some of them including carbides as grain growth inhibitors to suppress WC coarsening [7–10]. During sintering, the average carbide grain size increases due to coarsening or Ostwald ripening, that is, large grains grow while small grains dissolve, which leads to an

increase in average grain size. Abnormal grain growth may also occur, that is, a few larger grains consume all small grains, leading to an abnormally large grain size. In cemented carbides, where normal WC grain size is of the order of  $\mu\text{m}$  or less, abnormal grain growth can, sometimes, lead to grain sizes of several hundred  $\mu\text{m}$ . In cemented carbides, the diffusion distances are very short, and the common faceted shape of WC particles indicates that the obstacle in forming new atomic layers is the rate-controlling mechanism, rather than long-range diffusion [11].

While many cemented carbides rely primarily on the hardness, a number of application-relevant properties, such as strength and wear resistance, strongly depend on the crack resistance; hence easy access to fracture toughness is desirable for alloy development and quality control. While measuring other interesting properties such as microstructural parameters, Young's modulus, hardness or bending strength has become a routine matter, conventional toughness testing requires considerable effort; in particular, pre-cracking of specimens has remained a serious obstacle [12].

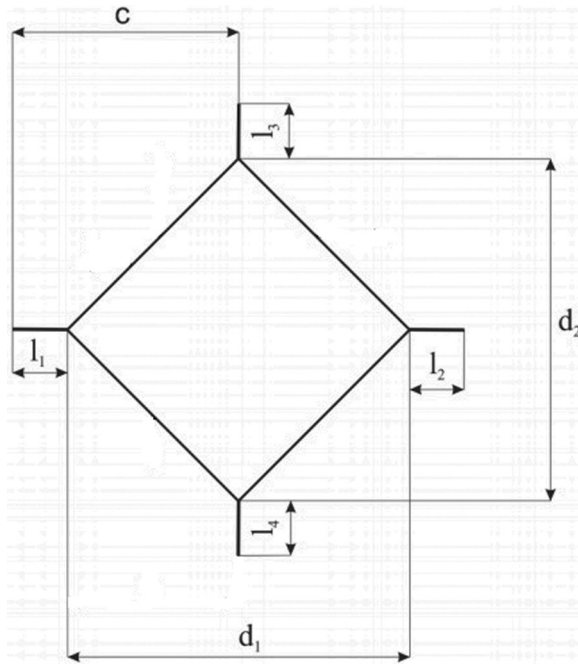
The indentation fracture toughness test has been considered an attractive method for assessing the toughness of ceramic materials because of the ease and low cost of conducting experiments. The predominant method to date has involved using a Vickers diamond hardness indenter to induce radial cracks in the material. Such radial cracks are thought to emanate from the indent as a result of residual tensile stresses that develop during unloading; arresting when the near-tip stress intensity,  $K_{\text{tip}}$ , equals the material toughness,  $K_{\text{IC}}$  [13].

Since Palmqvist [14–16] initially exhibited the potential significance of indentation-induced cracking to characterize the toughness of brittle materials, indentation fracture toughness (IFT) testing in determining the fracture toughness ( $K_{\text{IC}}$ ) of brittle materials on small scales has been the research hotspot over the past half century. Due to the requirements of sample size and shape in conventional fracture testing, there are few choices other than IFT when the test sample is small [17].

Palmqvist fracture toughness can be inferred from the total length of cracks produced at opposite corners of a Vickers indent and from the hardness of the specimen. The sum of crack lengths values, into Palmqvist fracture toughness values, is used. Indentation and crack length measurement are the distance from crack tip to crack tip for both diagonal directions. The total crack length is the sum of both these values minus the sum of the indentation diagonals (Figure 1) [18].

Equations for fracture toughness calculation can be obtained in different ways. Different equations relate to different crack types such as semi-elliptical cracks, median cracks or curve-fitting technique was developed. Fjodor and Maksim [19] listed some of the available equations for calculation of fracture toughness ( $K_{\text{IC}}$ ) values from Vickers indentation crack systems.

In this study, WC–10 wt% Co hardmetals have been investigated for their relationship between sintering parameters in the high pressure/high-temperature (HPHT) sintering method and hardness and fracture toughness determined via IFT. Such a study is of scientific interest because most of the previous fracture toughness studies and their conclusions are based on the conventional sintering method of WC–Co hardmetals. The HPHT method allows sintering WC/Co hardmetal at a higher temperature than the conventional sintering method and also decreasing sintering time to avoid WC grain growth that can affect the properties of hardmetals. Furthermore, this study is technologically



**Figure 1.** Schematic diagram of Palmqvist indentation characteristics [18], where  $l_n$  (mm) is crack length at indent,  $d_1$  and  $d_2$  (mm) are indentation – diagonal individual values.

important as the insight derived from this work can be utilized by both the scientific community and the industry.

## 2. Experimental procedure

Commercial Co powder and WC powders were used as the primary materials. WC powder and Co powder had an average particle size of 12.80 and 11.50  $\mu\text{m}$ , respectively. Powders were weighted with the nominal composition of WC–10 wt% Co and were mixed via ball milling in cyclohexane media. Milling speed and time were 200 rpm and 2 h, respectively, with ball to powder ratio of 10 to 1 by weight. Hardmetal balls and vessel were used to prepare the powders. Finally, WC–10 wt% Co powder mixture was obtained after drying under vacuum.

Mixed powders were encapsulated in a cylindrical graphite capsule of 5 mm diameter with graphite cap. Sintering was performed using an industrial HPHT machine (Razianse Pressmach – 630 tons capacity). In order to study the effects of sintering temperature and morphology evaluation, compacted capsules were subjected to five different temperatures of 1500°C, 1600°C, 1700°C, 1800°C and 1900°C under a constant pressure of 7.7 GPa and 2 and 3 min of heating time.

Relative density of sintered samples was measured using Archimedes' principle according to ASTM B962, and the morphologies of sintered samples were discerned by a scanning electron microscope (Hitachi Tm3000 desktop SEM) equipped with energy dispersive detector, and X-ray diffraction (XRD) analysis (Shimadzu PDA 7000) was used

to study the structured feature. The samples were ultrasonically cleaned for 30 min and then sectioned, ground and polished to mirror finish using diamond paste, up to a grit size of 1  $\mu\text{m}$ . Microstructures were observed in the mode of backscattered electron and XRD was performed using Cu  $K\alpha$  radiation and Ni filter with scanning step of  $0.3^\circ$  in the  $20\text{--}90^\circ$  ( $2\theta$ ) range for acquisition of complete spectra.

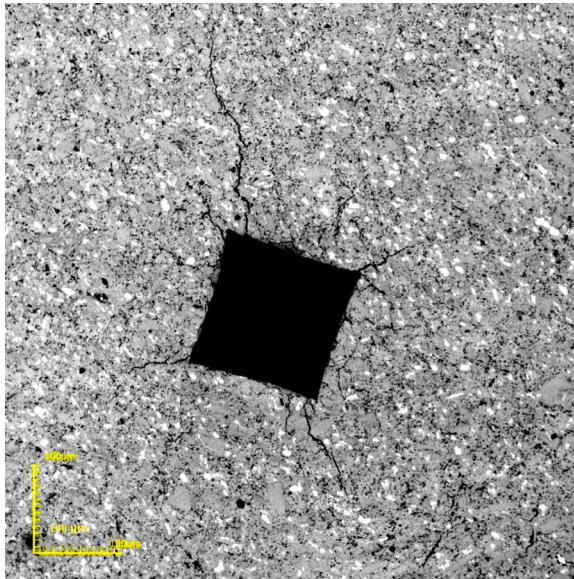
Hardness was measured with 15 and 30 kgf load and 15 s dwell time according to ISO 3878. Palmqvist indentation toughness was determined on cemented carbide specimens and the indentations for each load were carried out on diamond polished surfaces. The lengths ( $l$ ) of cracks, starting at the corners of indentation, were measured by light optical microscopy at  $500\times$  magnification (Figure 2). The length progression of cracks was observed with increase of the applied load. Palmqvist fracture toughness was assessed from Shetty et al.'s equation [20], according to

$$K_{IC} = \beta \left( \frac{PH}{\Sigma l} \right)^{1/2}, \quad (1)$$

where  $H$  is the hardness,  $P$  is the applied load,  $\Sigma l$  is the sum of crack lengths,  $\beta$  is a constant with a value of 0.0988 and  $K_{IC}$  is fracture toughness given as  $\text{MPa}/\text{m}^{1/2}$ . For  $HV_{15}$  and  $HV_{30}$  values expressed in  $\text{kgf}/\text{mm}^2$ , Palmqvist fracture toughness can be calculated as

$$K_{IC} = 0.383 \left( \frac{HV_{15}}{\Sigma l} \right)^{1/2}, \quad (2)$$

$$K_{IC} = 0.541 \left( \frac{HV_{30}}{\Sigma l} \right)^{1/2}. \quad (3)$$



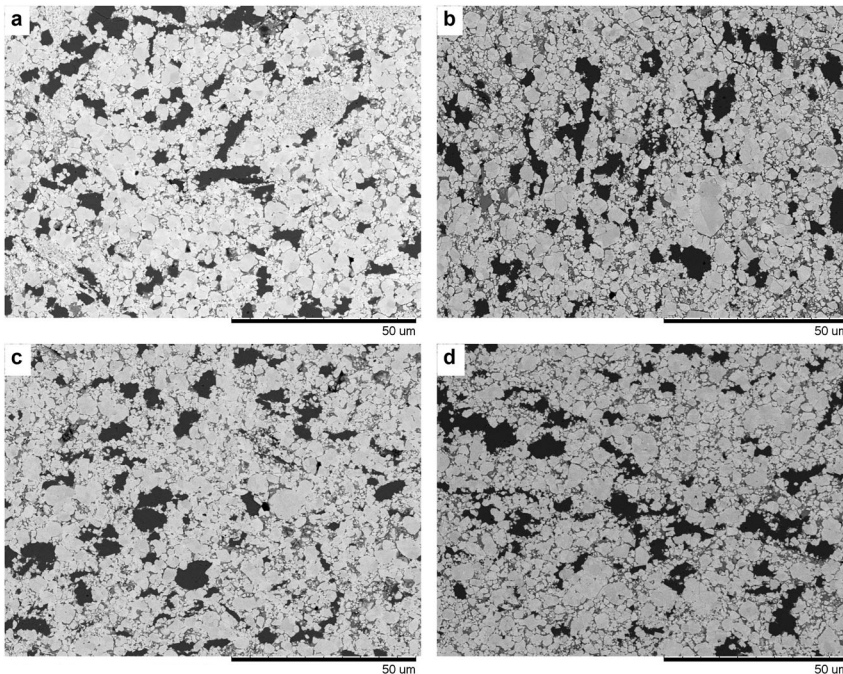
**Figure 2.** Photographs of Vickers (30 kgf) indentation of the typical sample sintered at 7.7 GPa/1900°C/2 min.

### 3. Results and discussion

Figure 3 shows the typical micrograph of sintered hardmetal samples at different sintering parameters. As found in SEM images, in samples sintered at higher temperature, there are some grain growths in microstructure for sintering time of both 2 and 3 min (see Figure 3 (b,d)). It can be explained by the effect of higher temperature on diffusion rate. At higher temperature, because of higher diffusion rate, it is easier for bigger particles to dissolve the smaller one and grow. Due to short sintering time, these abnormal growths were only found in samples sintered at higher temperatures (1800°C and 1900°C); however, some evidence of grain growth at lower temperatures were also found, but it seems that sintering time was not sufficient for severe growth.

Table 1 represents the achieved results including relative density of sintered samples at different sintering times and temperatures. The results show that the samples sintered at lower temperatures had some fine porosity and did not achieve full density. Full density at higher temperatures can be related to higher diffusion rate at higher temperature. Also, as temperature increases, porosities decrease due to higher liquid phase flux during sintering, and could be a reason for increasing relative density [21]. It seems that increasing sintering time from 2 to 3 minutes has no considerable effect on density. It suggests that temperature is a more important factor than time on diffusion processes.

Figures 4 and 5 show the X-ray diffractograms of sintered samples in different temperatures for 2 and 3 min, respectively. There are some peaks that cannot be indexed assuming the primary powders. Sample sintered at 1500°C, 1600°C and 1700°C for



**Figure 3.** SEM metallographic images of sintered WC-10Co at different temperatures and times. (a) 1600°C/2 min, (b) 1900°C/2 min, (c) 1600°C/3 min, and (d) 1900°C/3 min.

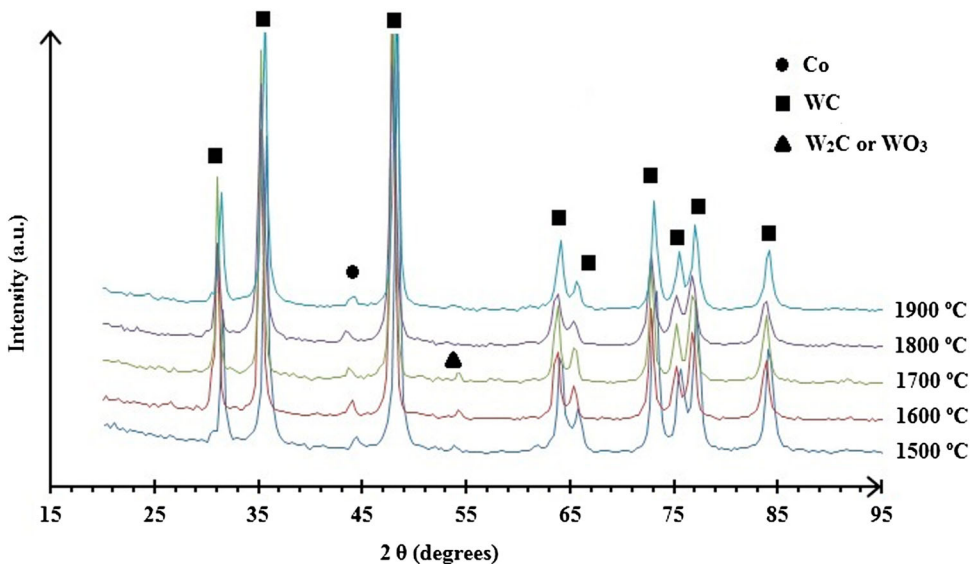
**Table 1.** Sintering parameters, relative density, hardness and fracture toughness of HPHT-sintered samples.

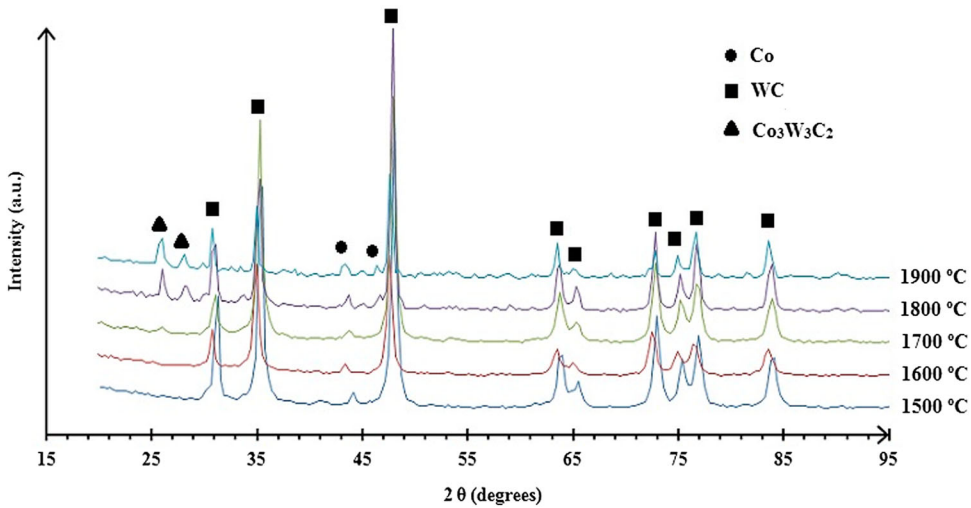
Sample no.	Sintering temperature (°C)	Holding time (min)	Relative density (%)	HV 15 (kgf/mm <sup>2</sup> )	Fracture toughness HV <sub>15</sub> (MPa/m <sup>1/2</sup> )	HV 30 (kgf/mm <sup>2</sup> )	Fracture toughness HV <sub>30</sub> (MPa/m <sup>1/2</sup> )
1	1500	2	90–93	1247	12.60	1195	12.08
2	1600	2	93–97	1364	13.47	1373	12.94
3	1700	2	100	1579	15.36	1545	15.85
4	1800	2	100	1623	16.34	1563	16.04
5	1900	2	100	1478	16.66	1460	16.40
6	1500	3	91–93	1285	12.79	1310	12.39
7	1600	3	93–98	1480	13.73	1434	14.01
8	1700	3	100	1652	15.48	1673	15.89
9	1800	3	100	1583	17.13	1510	16.52
10	1900	3	100	1327	17.85	1250	16.91

2 min have a very small peak at  $2\theta \approx 53^\circ$  related to the  $\eta$  phase that in this case can be  $W_2C$  or  $WO_3$ .

Formation of intermediate phases during WC/Co hardmetal sintering has been reported by various researchers [22–25]. The peaks of intermediate phases are small as compared with those of WC, showing there are only a small amount of these phases in the microstructure. These phases were formed at early stages of the sintering process because sintering has not been done in vacuum and no excess carbon was used. As can be seen in Figures 4 and 5, this peak (at  $2\theta \approx 53^\circ$ ) did not appear either in samples sintered at 1800°C and 1900°C for 2 min, or in all samples sintered for 3 min. It seems that higher sintering temperature and also longer sintering temperature can dissolve these phases.

In Figure 5, there are two peaks at  $2\theta \approx 26^\circ$  and  $28^\circ$  in samples sintered at 1800°C and 1900°C, showing that some intermediate phase ( $Co_3W_3C$ ) was formed during sintering.

**Figure 4.** X-ray diffractograms of samples sintered for 2 min at different temperatures.



**Figure 5.** X-ray diffractograms of samples sintered for 3 min at different temperatures.

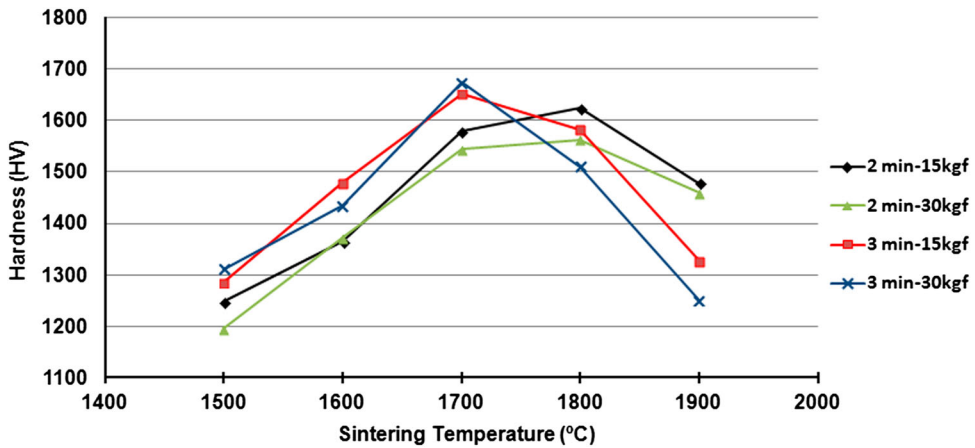
Other studies [22,25] also reported this intermediate phase in hardmetal microstructure. Although there is no peak related to this phase in samples sintered for 2 min at any temperature, and also in samples sintered for 3 min at 1500–1700°C, it seems that this intermediate phase cannot form at a low sintering temperature or short sintering time. In the low sintering temperature and short sintering time condition, there was not enough carbon from WC to diffuse and subsequently form  $\text{Co}_3\text{W}_3\text{C}_2$ . It is important to note that this intermediate phase is formed due to a decrease in carbon content of WC as a result of reaction with adsorbed oxygen, almost instantly during a very quick HPHT sintering [23,25].

For IFT calculations, it is recommended to take into account the true (load independent) hardness of the material. It is well known that the apparent hardness decreases with the increase in indentation load and approaches a constant value at a relatively high load level [19]. The data resulted from Vickers hardness test at different testing load are listed in Table 1, and were used in IFT calculation. The results are also plotted versus sintering parameters in Figures 5 and 6.

An ideal tool material should have high hardness and high toughness. However, both the properties are inversely proportional to each other. Both properties are structure sensitive, so microstructure of the samples plays a very important role. Past research has also tried to optimize the combination through conventional and advanced sintering techniques. Controlling particle size/microstructure is of great importance for all the sintering processes [26].

The hardness of WC–10 wt% Co hardmetals was almost independent of applied load but was sensitively dependent on the sintering temperature. As can be seen in Figure 6, the hardness increases sharply by increasing the sintering temperature from 1500°C to 1700°C for both sintering times. Increasing sintering temperature up to 1800°C for the sample sintered for 2 min results in a slight increase in hardness but for the sample sintered for 3 min results are contrary (a slight decrease). There is a notable decrease in hardness by increasing sintering temperature from 1800°C to 1900°C for both sintering times.

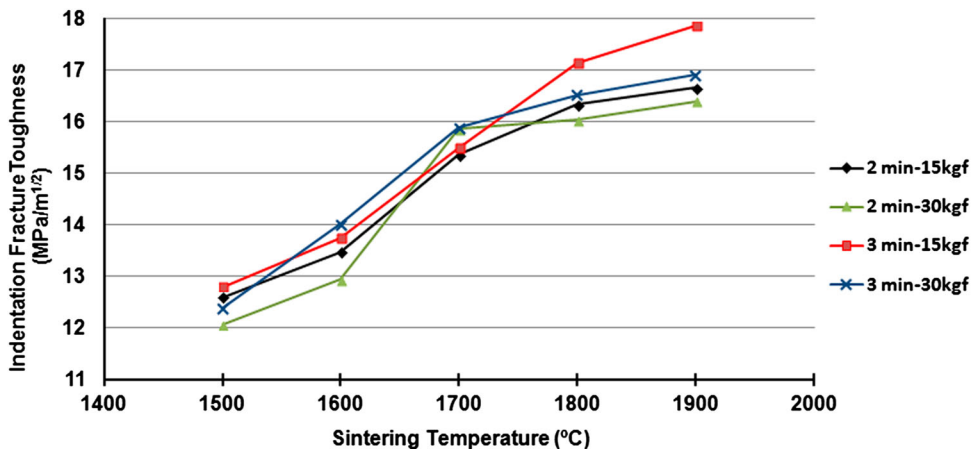




**Figure 6.** Vickers hardness as a function of sintering temperature for different sintering times of 2 and 3 min.

The rapid increase in hardness above 1500°C seems to occur due to the formation of a liquid phase during sintering which leads to an increase in relative density and decrease in porosities. Moreover, the hardness slightly decreases above 1700°C for the sample sintered for 3 min. It can be attributed to the fact that the sintered density reaches the saturated value, but the grain size of WC considerably increases at higher temperatures. For samples sintered for 2 min, it seems that considerable increasing in the grain size of WC started above 1800°C. Hardness decreases dramatically for samples sintered at higher temperature and longer time due to negative effects of the  $\eta$  ( $\text{Co}_3\text{W}_3\text{C}$ ) phase available in this microstructure (Figure 5) [23,24,27,28].

The highest hardness value around 1670 kgf/mm<sup>2</sup> and fracture toughness of 15.89 MPa/m<sup>1/2</sup> were observed in samples sintered at 1700°C/3 min. Samples sintered at 1800°C and 1900°C, with holding time of 2 min, presented higher fracture toughness that is in accordance with low hardness and grain growth of these samples. For



**Figure 7.** IFT as a function of sintering temperature for different sintering times of 2 and 3 min.

**Table 2.** Comparison of hardness and fracture toughness combinations obtained with different techniques [26].

Composition	Sintering method	Hardness (kgf/mm <sup>2</sup> )	Fracture toughness (MPa/m <sup>1/2</sup> )	Sintering temperature (°C)	Holding time (min.)
WC-10Co	HFIHS	1280	16.4	1150	1
WC-9.2Co	HFIHS	1992	11.9	1150	1
WC-10Co-0.7VC	CLPS	1610	10	1370	60
WC-10Co-0.7VC	CLPS	1610	10	1410	60
WC-10Co	CLPS	1310	13	1370	60
WC-10Co	CLPS	1410	11.8	1410	60
WC-11Co	CLPS	1782	9.4	1390-1470	-
WC-17Co	CLPS	1591	11	1390-1470	-
WC-21Co	CLPS	1483	12.3	1390-1470	-
WC-12Co	CLPS	1748	9.2	1390-1470	-
WC-20Co	CLPS	1359	13.6	1390-1470	-
WC-14Co	CLPS	1426	11.8	1390-1470	-
WC-17Co	CLPS	1335	13.8	1390-1470	-
WC-21Co	CLPS	1264	20.8	1390-1470	-
WC-13Co	CLPS	1395	12	1390-1470	-
WC-12Co-0.4VC	HIP	1340-1381	12.62-18.62	1390	60
WC-10Co-2VC	HIP	1430	14	1260	30
WC-20Co	CLPS	1582	14.3	1350	1
WC-20Co-2.5VC	CLPS	1693	14.1	1350	1
WC-20Co-5VC	CLPS	1709	15.1	1350	1
WC-20Co-7.5VC	CLPS	1870	14.4	1350	1
WC-20Co	CLPS	1566	15.5	1400	1
WC-20Co-2.5VC	CLPS	1701	13.8	1400	1
WC-20Co-5VC	CLPS	1649	14.7	1400	1
WC-20Co-7.5VC	CLPS	1687	12.4	1400	1

Note: CLPS: conventional liquid phase sintering; HIP: Hot iso-static pressing; HFIHS: high-frequency induction-heated sintering.

samples sintered at 1500°C and 1600°C, with holding time of 2 and 3 min, higher fracture toughness was expected. Nevertheless, as can be seen in [Figure 7](#), the fracture toughness is nearly low. It seems that presenting porosity and incomplete sintering reduced the fracture toughness. Intermediate phase (W<sub>2</sub>C or WO<sub>3</sub>) formation also can play a negative role in fracture toughness.

Kumar and Singh [26] collected some hardness and fracture toughness combinations, obtained by some researchers, when hardmetals were sintered by different techniques. Those results along with results achieved by Kumar and Singh [26] are listed in [Table 2](#). The results are in good agreement with HPHT sintering of this study.

All the above-achieved results show that the best HPHT sintering conditions, with a good combination of hardness and fracture toughness are 7.7 GPa/1700°C/3 min, and 7.7 GPa/1800°C/2 min.

#### 4. Conclusion

The following conclusions have been drawn from the fracture toughness study of HPHT-sintered WC/Co hardmetal:

- (1) Higher sintering temperature resulted in grain growth. The formation of the  $\eta$  phase decreases the hardness of the samples when sintering temperature is raised to 1800°C;
- (2) Fracture toughness increases with sintering temperature. Higher toughness at high sintering temperature is related to grain growth, and low toughness at lower

temperature is related to higher porosity, incomplete sintering and the formation of intermediate phases.

- (3) High hardness along with good fracture toughness is observed at 1700°C and 1800°C with 2 and 3 min of holding time, respectively. Small grain size and minimum porosity are responsible for high hardness (1673 kgf/mm<sup>2</sup>) with high toughness (16.34 MPa/m<sup>1/2</sup>).

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## Disclosure statement

No potential conflict of interest was reported by the authors.

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