Effect of alumina inclusions on microstructure and mechanical properties of 62 WC, 32 (Ti-W) C and 6 Co compacts

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Abstract: This investigation is aimed at understanding the role of adding ceramic inclusion, alumina (Al_2O_3) on the densification, microstructure and mechanical properties of cemented carbide cutting inserts. The samples were prepared of an initial mixture of 62 WC, 32 (Ti-W) C and 6 Co wt% particulates; different additions of Al_2O_3 of 1, 2, 3 and 4 wt% were introduced to that mixture. The testing specimens were compacted via powder metallurgy at 200 MPa and sintered at 1,410°C under vacuum. The green and sintered densities have decreased; the figures of macrohardness, Vickers hardness and transverse rupture strength have also deteriorated with the increase of alumina content. The macrohardness has almost declined of 30% at 3% alumina. This is ascribed to the interfacial grain-boundary decohesion between Al_2O_3 and carbide particles as can be observed from scanning electron microscopy images which depicts the monolithic clusters in the final consolidation of the compacts.

Keywords: powder metallurgy; cemented carbides; alumina; inclusion; microstructure and materials properties.

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1 Introduction

Hard metals (cemented carbides) are range of very hard, refractory and wear-resistant alloys. The most common type of cemented carbides is cutting tool inserts which make by powder metallurgy techniques. The properties of cemented carbides are influenced profoundly by their microstructure. This in turn depends mainly on basic chemical composition of carbide and matrix phases, size, shape and distribution of carbide particles, relative proportions of carbide and matrix phases. Additionally, degree of intersolubility of carbides, excess of deficiency of carbon variations in composition and structure caused by diffusion or segregation are also affected the final microstructure and characteristics of the cemented carbides (Brookes, 1987; Stevenson, 1988).

Density (or specific gravity), hardness, transverse rupture strength (TRS) and residual porosity are common properties of the cemented carbides recommend to be measured in order to determine the desirable characteristics of the cemented carbide products (Brookes, 1987). The density can be identified as the weight per unit volume of the cemented carbide in grams per cubic centimetre (g/cm^3) units. The density is decreased with the increase of the lighter binder metal content particularly in grades including only tungsten carbide and a binder metal. The hardness of the cemented carbides is a measure of their resistance to penetration by a diamond indenter under a specific load. Different scales are normally used to measure the hardness of the carbides, such as Rockwell A (HRA) scale and the Vickers (HV) scale. The hardness is a function of composition and grain size. The higher binder metal contents and the coarser tungsten carbide particle sizes result in lower hardness values. In contrast, lower binder content and fine grain sizes induce higher hardness values. Furthermore, TRS of the cemented carbide is primarily a measure of tensile strength in a three point bending test performed on standard rectangular bars. It measures in Newtons per square millimetre (N/mm^2) units. Finally, the residual porosity is determined by visually examining the polished surface of a sintered sample at 100 x or 200 x magnification. Different types of porosity are commonly used; the 'A-type' is suitable for the pores diameter less than 10 μm. The 'B-type' porosity is used for the pores diameter larger than 10 μm, and the 'C-type' porosity is applicable for carbon inclusions. All porosity types are determined by comparing the size and frequencies of each pore type in the sample with those in standard photographs. In general, at high levels of porosity, the wear resistance of the product may be adversely affected; the edge strength and toughness are also decreased with increasing the residual porosity (Brookes, 1987; Haller, 1988).

Based on the machining operations and working conditions, tough and shock-resistant tools grades are needed for slow speeds and interrupted cutting. Harder tools grades for high-speed finishing and heat-resisting alloyed grades for machining superalloys and crater-resistant compositions. Hence, many tungsten carbide-cobalt compositions are modified by small but important additions from 0.5 to perhaps 3% of tantalum, niobium, chromium, vanadium, titanium, hafnium, or other carbides. The basic purpose of these additions is generally inhibition of grain growth, so that a consistently fine structure is maintained. While TaC and NbC as well as TiC play an important role (Brookes, 1987; Stevenson, 1988; Haller, 1988; Fang et al., 2009). They are often included in grade compositions for a number of reasons, including increased high-temperature deformation resistance; increased chemical stability and grain growth control. However, their scarce abundance and overwhelming preparation costs had been encouraging the researchers to find out alternative materials being capable of achieving the required properties of carbides powders. Moreover, cutting tools are reportedly two billion dollar industry worldwide and form the backbone of manufacturing operations for metals, polymers and advanced materials such as intermetallics and composites of all types (Upadhyaya, 2001).

Ceramics, on the other hand compete directly with hardmetals, mainly in the cutting-tool category and ceramics moving up the hardness scale, ceramics provide increasing competition for cemented carbides, both in performance and in cost-effectiveness, though not yet in reliability. Hardmetals themselves consist of ceramics-nonmetallic refractory compounds, usually carbides or carbonitrides with a metallic binder of much lower melting point. In such systems, densification generally takes place by liquid-phase sintering. Furthermore, alumina cutting tips have extreme hardness more than HV 2000 or HRA 94 and give excellent service in their limited (Brookes, 1987). For these virtues of alumina; the idea of the present paper was shaped to investigate the crucial effects of adding such materials on the principal features of carbide cutting tools, such as bulk density, hardness, fracture and their structure. Meanwhile, there is by far no particular work demonstrated the advantages or even the disadvantages of introducing a ceramic powder to cemented carbide components. This could be because of the substantial difference amongst major properties for carbides and ceramics particulates. In contrast, many researchers have studied the influence of adding conventional hard cemented carbides on the final compacts properties (Bhaumik et al., 1992; Saito et al., 2006; Ostberg et al., 2006; Barbatti et al., 2009; Petersson, 2004), also, a few have studied the effects of Alumina addition as solid inclusions on the final properties of metals, superalloys and ceramics (Lange and Hirlinger, 1987; Ekstrom, 1993; Lafer et al., 1993). In addition, several researchers (Wang et al., 2001; Acchar et al., 2005; Acchar and Cairo, 2006; Acchar et al., 2006) have investigated the effect of dispersing cemented carbide to alumina ceramic base. But, no work has specifically addressed the influence of alumina inclusions on the final composition of the cemented carbides. Therefore, the main concept of this paper is introducing the ceramic inclusions, alumina to the cemented carbide composition of tungsten carbide, titanium carbide and cobalt in order to observe the susceptibility and monitoring the detriments and the resultant properties of that combination thereof. For that purpose, several common physical and mechanical properties of the new produced compound have been measured as well as depicting their final microstructure by using scanning electron microscopy (SEM).

Figure 1 The nominal thermal cycle for the sintering process

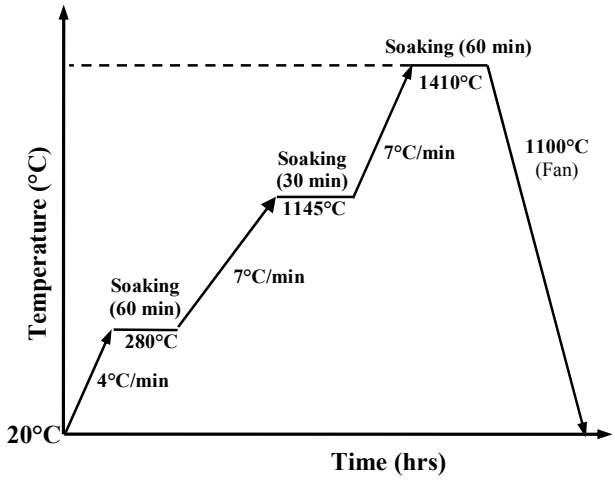


Table 1 Chemical compositions, apparent density and grain size of the carbide compacts including different alumina inclusions

Powders mixtures	*Al_2O_3 (Wt. %)*	*WC (Wt. %)*	*(Ti-W) C (Wt. %)*	*Co (Wt. %)*	*Apparent density (g/cm^3)[a]*	*Average grain size (μm)[b]*
A	0	62	32	6	2.9	5.78
B	1	61.38	31.68	5.94	2.72	5.75
C	2	60.76	31.36	5.88	2.56	5.77
D	3	60.14	31.04	5.82	2.44	5.70
E	4	59.52	30.72	5.76	2.34	5.80

Notes: a The standard deviation for the results is ± 0.05.
b The standard deviation for the results is ± 0.03.

2 Experimental procedures

2.1 Starting materials and samples preparation

Weighed concentrations of WC, 32 (Ti-W) C and 6 Co powders having average grain sizes of 2.5, 2.5 and 1.35 μm, respectively; provided by HCST Berlin GmbH, Goslar, Germany were prepared as a master mixture. This mixture was blended with acetone in 4.5 dm^3 rotary ball milling. The acetone was added with ratio (1:4) to the whole powders weight and operated for 12 hours thereby the slurry was dried in vacuum furnace (Paulo ABBC, INC, USA) at 90°C to remove the acetone. The resultant powders were a Fischer sub-sieve size (FSSS) of 5.78 μm. The paraffin was added of 2 wt. % as die wall lubricant, along with different values of 1, 2, 3, and 4 wt. % of Al_2O_3 particulates having 1.23 μm grain size and apparent density of 0.79 g/cm^3 supplied by ALM-41-sumito Company, UK. The final mixture as tabulated in Table 1 was mixed intimately by a small scale batch mixer before compacted via double die compaction press type at 200 MPa

and sintered under vacuum at 1,410°C of 5°C for heating and cooling rates following the thermal cycle shown in Figure 1.

2.2 Microstructure and testing procedure

In order to understand explicitly the effects of adding Al_2O_3 particles on the final consolidation of the cemented carbide compacts as well as to observe their generated interaction due to alumina addition. The microstructure of the final carbide's compacts incorporated with different Al_2O_3 inclusions has been depicted using scanning electron microscope type (5900LV, JEOL, Japan). The SEM samples of 5 mm × 5 mm × 5 mm were prepared using wire-cut in electro discharge machining (EDM) technique from TRS's specimens.

Green and sintered densities were measured applying Archimedes method; the shrinkage was assessed by measuring the dimensions of the compacts before and after the sintering process. The macrohardness Rockwell-A scale (HRA) and Vickers hardness (Hv) were assessed by diamond indenter, 60 kg load and microhardness tester HV-1000, China, applying 0.98 N load, respectively. TRS of the resultant samples were calculated depending three-point bending test according to ASTM standard B528 (Philadephia, 1989). Five tons load at 0.5 mm/min increasing rate was applied on TRS specimens (5.2 mm × 5.5 mm × 51 mm) via Shimadzu Servopulser Machine Model-FHF20, Japan.

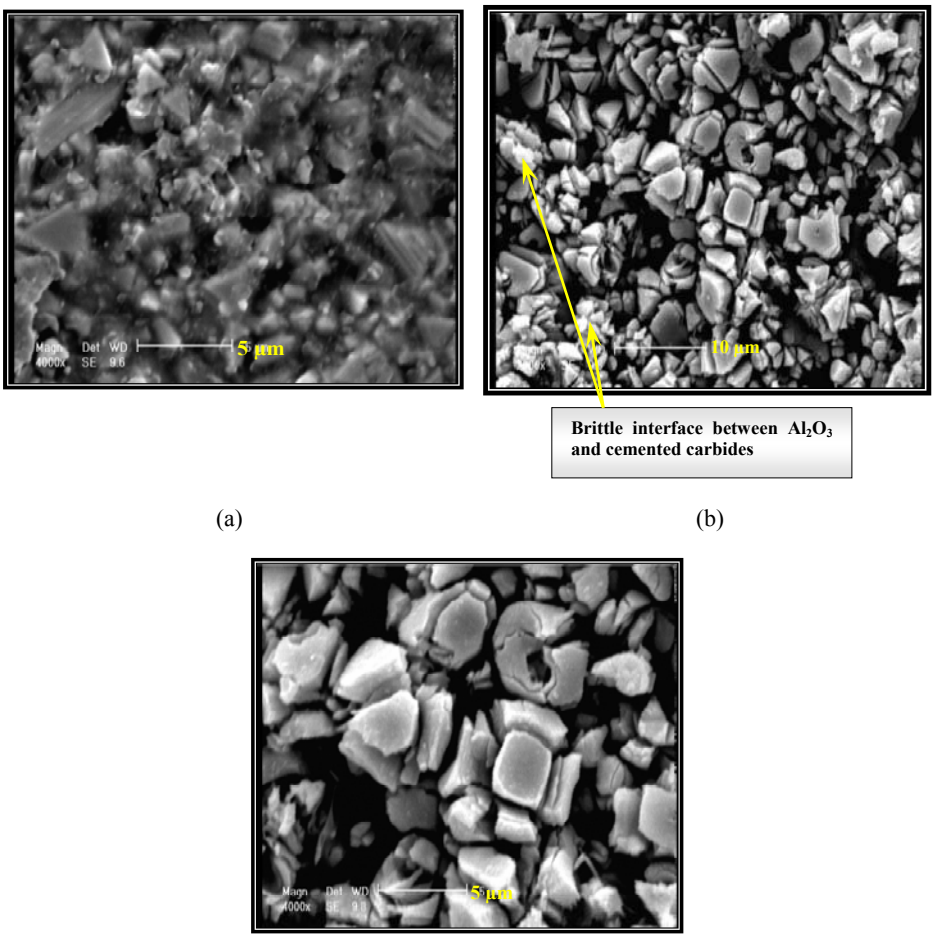
3 Results and discussion

3.1 Microstructure characterisation

The microstructure of sintered WC-32 (Ti-W) C-6 Co cemented carbides doped by different proportions 1–4 wt.% of Al_2O_3 inclusions was observed by scanning electron microscope as shown in Figure 2(a), Figure 2(b) and Figure 2(c) and Figure 3(a), Figure 3(b) and Figure 3(c), respectively. In general, it can be seen that alumina inclusions have agglomerated and made relatively substantial clusters- which subsequently created obvious separated regions or inhomogeneous even with low alumina contents. It is clear from Figure 2(a) that the core-rim structure did not form (exist) as presumably should be existed for cemented carbide's structure due to the poor interaction and the poor diffusion of atoms across interface between particles bonding the particles together as one coherent mass. This diffusion presumably should occur during the sintering operation but perhaps the sintering temperature was insufficient as ceramic powders require higher temperatures. However, there was a distinctive change in microstructure morphology in addition to heterogeneous distribution of particles, this may reflect that the structures of cemented carbide are very susceptible and sensitive for adding any reinforcements or inclusions whether these additive are isotropic or anisotropic particles. Increasing Al_2O_3 particles content to 3%, the distribution and the structure of Al_2O_3 seem to be separated by a small layer of the binder. In other words, it seems to be split up from the other carbides and induces forming an abnormal grain growth. Thus, a coarsening in the grain size

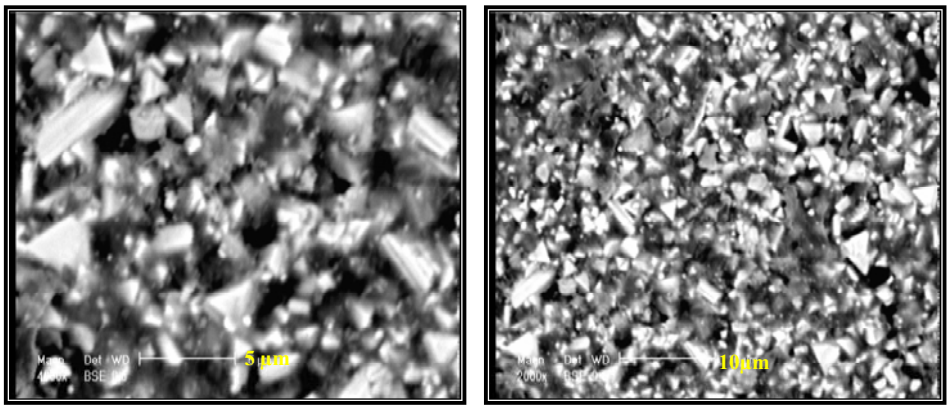
and consequently an embrittlement phase will be generated as can be noticed in Figure 2(b).

Figure 2 Scanning electron micrographs of the compacted carbides including different Al_2O_3 inclusions (front-scattered electron-SE), (a) 1% Al_2O_3, (b) 3% Al_2O_3 (2000X), (c) 3% Al_2O_3 (4000X) (see online version for colours)

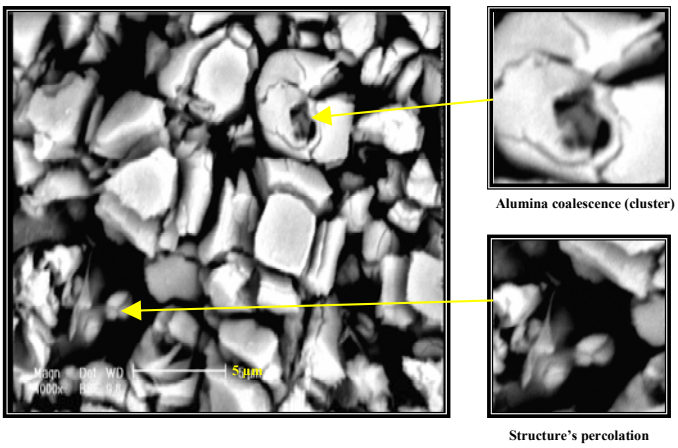


Generally speaking, the microstructure of the sintered WC-(Ti-W)C-Co cemented carbides must include several phases of angular crystals of almost pure tungsten carbide WC (α phase), rounded TiC, mixed crystals of carbides (Ti, W) C (γ phase) and dark grey for the liquid phase Co binder (β phase) (Brookes, 1987; Stevenson, 1988; Haller, 1988; Fang et al., 2009) but when adding new carbide or inclusion powders. The microstructure should adopt new phases such as eta phase (η phase): Co_3W_3C (M_6C), Co_6W_6C ($M_{12}C$) and di-tungsten carbide (W_2C) (Brookes, 1987; Stevenson, 1988; Haller, 1988). However the x-ray diffraction entails to prove these discrepancies of microstructural phases and to better understanding the final structure. But unfortunately, the availability of this service has not been available.

Figure 3 Scanning electron micrographs of the compacted carbides including different Al_2O_3 inclusions (backscattered electron -BSE), (a) 1% Al_2O_3, (b) 2% Al_2O_3, (c) 3% Al_2O_3 (see online version for colours)



(a) (b)



(c)

3.2 Densification behaviour

The green and sintered densities of WC-32(Ti-W)C-6Co cemented carbides containing different additions (1, 2, 3, and 4 wt%) of Al_2O_3 inclusions were plotted in Figure 4. Both densities declined significantly with increasing Al_2O_3 content. This is an obvious result as Al_2O_3 has very low apparent density of 0.79 g/cm^3 in comparison with 2.9 g/cm^3 for the neat cemented carbides. Furthermore, the incomplete reaction between alumina and other carbides particles as observed in SEM micrographs. This reaction believes to be caused due to

1 no affinity from WC for Al_2O_3, i.e., poor wettability between WC and Al_2O_3 since WC has only a small tolerance for changing its carbon content and a part of carbon will absorb by Al_2O_3 thereby a slight change of carbon leads to form W_2C phase. Consequently, the brittle free carbon (η-phase) will appear in the final sintered carbides which in turn induce poor densification (Brookes, 1987; Stevenson, 1988).

2 insufficient mixing time or inadequate sintering time and/or temperature to complete the full densification and combustion reaction of alumina powder

3 the lack of intermixing by the difference in the original particle size while the alumina inclusion apparently having irregular-shape in reverse to the carbides shape.

Figure 4 Effect of alumina content on green and sintered densities of the compacted cemented carbide (see online version for colours)

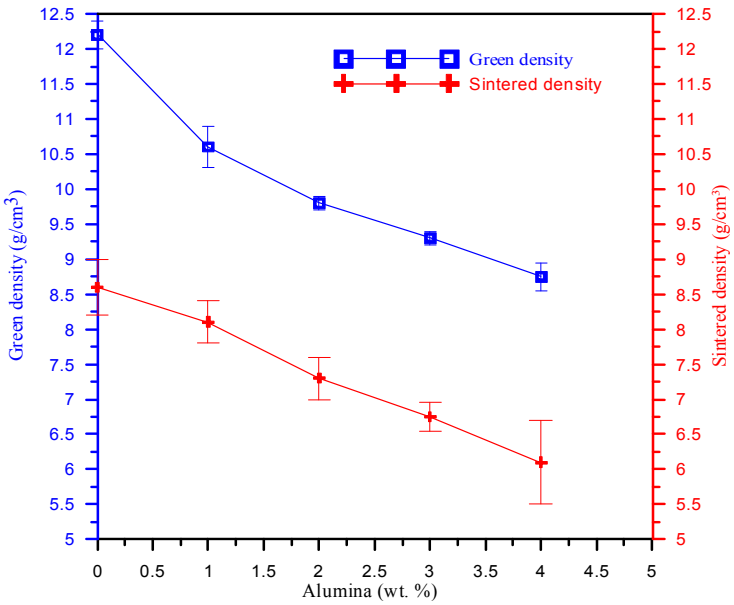


Figure 5 Effect of alumina content on shrinkage ratio of the compacted cemented carbide

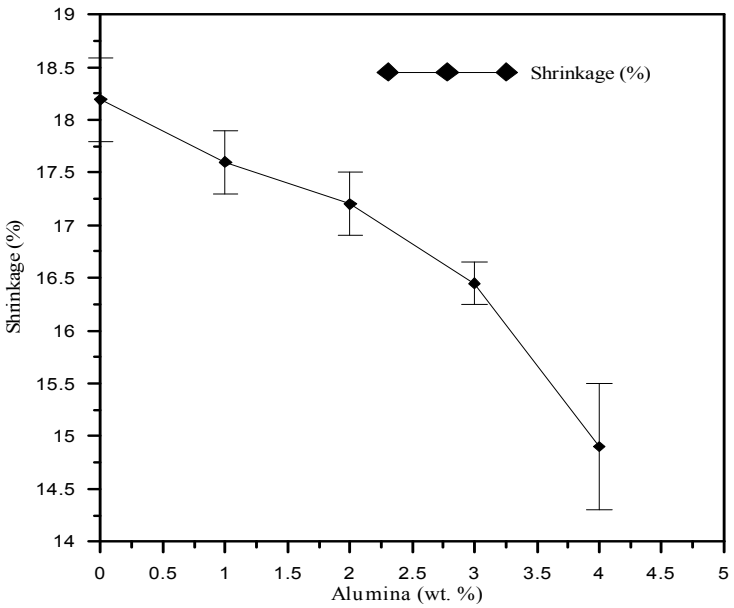


Figure 5 demonstrates the effect of alumina additions on the shrinkage of the sintered cemented carbides. As the Al_2O_3 percentage increases, the percentage of the shrinkage decreases; it was 18.19% for the neat cemented carbides but this percentage reduced to 16.45% and 14.9% when adding 3 and 4 weight percentage of alumina, respectively. Once again, this behaviour is attributed to the extreme difference between densities of Al_2O_3 and carbide compacts along with the incomplete interactions between the carbides particles and the alumina powder. Additionally, it is ascribed to the poor driving force which resulted in poor densification in all stages of the liquid phase sintering and subsequently in high shrinkage percentages. Interestingly, the good driving force is required to minimise the total surface tension and to overcome the agglomeration of particles. Hence, in order to obtain a maximum attainable densification and subsequent plausible shrinkage; several parameters need to be mentioned here those highly influence the rearrangement of particles, are: The amount of a liquid phase present, the particle size, the contact angle or surface area of particles and the solubility of the solid in the liquid (Petersson, 2004).

3.3 Mechanical properties

Figure 6 shows the effect of adding alumina inclusions on macrohardness (Rockwell-A) and Vicker's hardness of the sintered WC/32 (Ti-W)C/6 Co cemented carbides formed at 200 MPa and sintered at 1,410°C in vacuum furnace using powder metallurgy. Each of hardness number has repeated three times and the results have averaged to conduct the validity of results since there is a big unevenness and segregation. From Figure 6, we can notice all hardness figures have decreased with the increase of alumina content. This reduction can be attributed to several reasons:

1 The appearance of eta phase owing to changing carbon content when dispersing ceramic alumina particles. This brittle phase will replace with the binder during the sintering process and results in not only the embrittlement of the structure, but also reduces the effective contribution of WC to strengthen the final compound. Therefore the carbon content must be maintained within narrow limits in order to obtain the desired composite with optimum properties. However, unfortunately there is no possibility to maintain that limit with the existence of alumina in the cemented carbides.

2 The distinctive coalescence, porosity and agglomeration of alumina particulates in the structure due to the high surface energy and large specific area for alumina. The surface area of Al_2O_3 was 35,963.21 cm^2/cm^3 at (1.23μm) average grain sizes in contrast of 27,545.7 cm^2/cm^3 at (5.78μm) for the master alloy of WC/32(Ti-W)C/6Co. Additionally, the significant difference in specific gravities (densities) between alumina and tungsten carbide definitely results in poor interfacial bonding. Meanwhile, the fluctuating results of Vicker's hardness in some samples rather than others counterparts are a good example showing the segregation behaviour and its influence on the properties.

3 The lower densification of produced compounds, the short distances between components, the impurities and the segregation are highly adversely affected the properties. For instance, when adding of 1 wt.% alumina, the hardness of the compacted samples has slightly reduced due to the rule-of-mixtures but when the

alumina values was risen up to 3 wt.% or more, both macro and microhardness have noticeably deteriorated.

Figure 6 Effect of alumina content on Rockwell macrohardness and Vickers hardness of the compacted cemented carbide (see online version for colours)

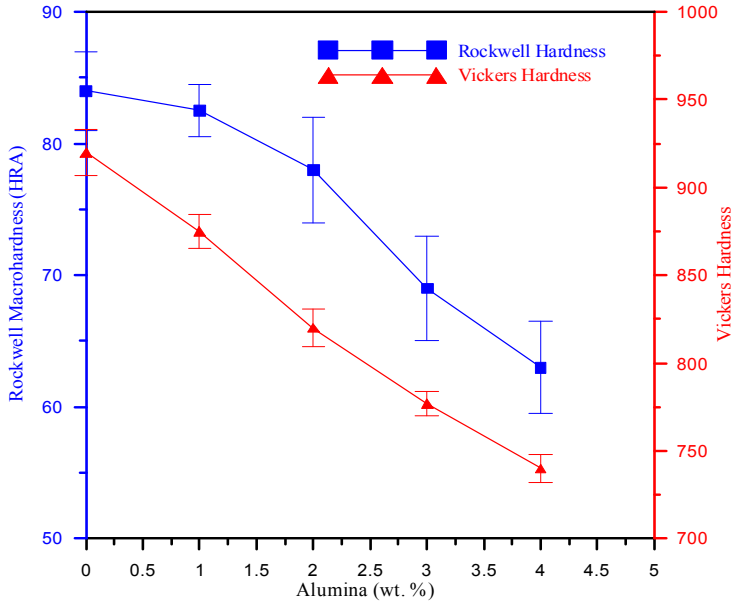
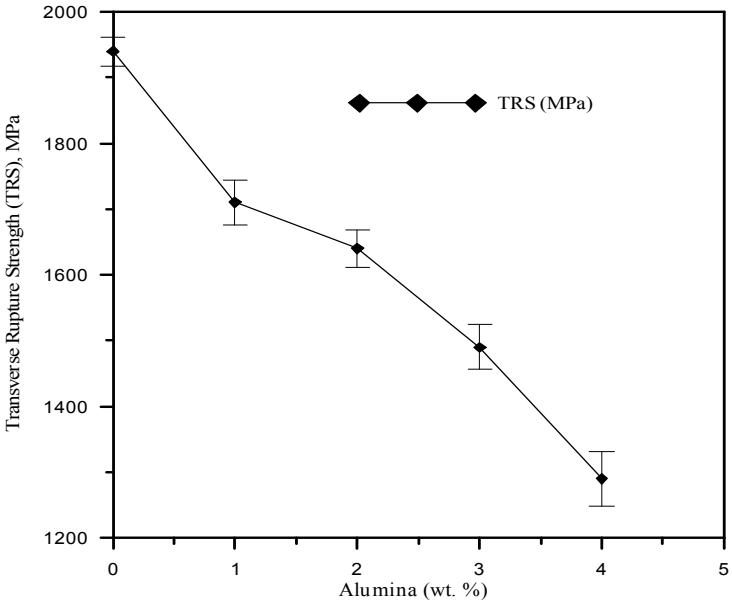


Figure 7 demonstrates the variation of TRS of 62WC/32(Ti-W)C/6Co cemented carbides with different Al_2O_3 content. It is noticed that the TRS values have decreased with increasing of alumina content. The TRS is dependent on the WC grains size and the volume fraction of WC/(Ti-W)C core-rim phase (Brookes, 1987). Hence, the addition of alumina inclusions to the cemented carbides resulted in embrittling the body, influencing the sintering process of aggregate compacts and retarding the diffusion at certain temperatures, weakening of the interparticle bonding, and results in heterogeneous structure and a drastic grain-boundary decohesion. Besides, the reduction in the TRS values may come as a consequence of the poor solubility (if any) of alumina in WC from one hand; and forming hard phase in the solid solution due to the solubility of WC in the TiC forming (W-Ti) C phase on the other hand. This diverse gradient in structure could play as stress concentration thereby accelerates the failure. Thence, the desirable properties and the hardening role of using alumina in cemented carbide will display and the attempt of using such materials with carbide being inviability. However, there are some suggestions might be viable for the future work, for example (Petersson, 2004) revealed that high carbon content increases the densification rate in cemented carbide in compared to stoichiometrical mixtures. Also, there is another suggestion is based on (Acchar et al., 2001) which involved a combined addition of Y_2O_3 with Al_2O_3 in small amounts to the carbides and raising the sintering temperatures higher than 1,760 °C. In which, the result showed a formation of a liquid phase which in turn resulted in good densification and uniform distribution of secondary intergranular phase within the grains.

Figure 7 Effect of Alumina content on TRS of the compacted cemented carbide



4 Conclusions

The effect of introducing Al_2O_3 inclusions on the densification, microstructure and mechanical properties of 62WC/32(Ti-W)C/6Co cemented carbides can be summarised as below:

1 The microstructure of the cemented carbide is very susceptible for dispersing any material whether is isotropic or anisotropic as the cemented carbide properties are obviously highly influenced by their microstructure and composition. Hence, dispersing of small amount of alumina inclusion particles is highly affected the properties and microstructure of the carbides.

2 Dispersing Al_2O_3 in the cemented carbides is uncontrolled process to maintain the final configuration solid solution. Since, the alumina has a strong tendency and large surface area to form coalescence, cluster and intermittent bonding in the final sintered composites.

3 The addition of Al_2O_3 to the cemented carbides resulted in the reduction of the green and sintered densities, the shrinkage and hardness and TRS.

4 Al_2O_3 is decarburising materials resulted in creating eta phase, embrittling the structure and leaves a bright skin or surface layer on the produced samples, this layer containing a diffused aluminium which should be removed by grinding the component before service.

5 The addition of 3 wt. % henceforth of alumina to the carbides in which the tungsten carbide seems has not melted congruently but has decomposed to a fragile mixture of W_2C, or WAl_2O_3C formed on cooling. The poor densification of Al_2O_3/cemented carbides powders could result due to the insufficient sintering temperature which also resulted in more dissipated pore network that prevents early sealing of the pores.

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