To my beloved family and friends

Thanks for the support and encouragement

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ABSTRACT

Aluminium oxide is a chemical compound, also known as alumina (Al_2O_3), is being widely used as a material in a cutting tool due to its superior mechanical properties. Nevertheless, alumina is not a straightforward solution for practical application due to its brittle nature. One of the solutions was to integrate other components into the base material of alumina. For an example, adding zirconia (ZrO_2) in alumina (Al_2O_3) matrix produced a ceramic that improved the toughness of the material, though the toughness can be further improved. As a solution, the present work aims to enhance the ZTA ceramic composite's fracture toughness by introducing a combination of microwave heat treatment and multi-phasic additives. Part 1 and Part 2 of this study utilised a conventional sintering process operating at a temperature of 1600°C for a total of 1 hour dwelling time. In the case of Part 3, 2.45 GHz microwave is used for sintering process at a temperature range of 1200°C - 1400°C in a 10-minutes dwelling time. The first phase of this work reported an enhanced ZTA properties with an addition of 3.0 wt.% TiO_2. Moreover, the hardness is improved from 1516.13 HV/14.87 GPa (0 wt.% TiO_2) to 1615.8 HV/15.85 GPa (3.0 wt.% TiO_2), while the fracture toughness is improved from 5.93 $MPa.m^{1/2}$ (0 wt.% TiO_2) to 6.56 $MPa.m^{1/2}$ (3.0 wt.% TiO_2). Additionally, the enhanced mechanical properties can also be attributed to the presence of TiO_2 as a vital sintering aid, which impeded Al_2O_3 grain growth, and consequently led to the formation of a denser and finer microstructure. In the second part, Cr_2O_3 is introduced as a new additive material that can be used with ZTA-3.0 wt.% TiO_2. The outcome revealed that the properties associated with ZTA–3.0 wt.% TiO_2 ceramic composite improved after the addition of 0.6 wt.% Cr_2O_3. Subsequently, the fracture toughness (7.15 $MPa.m^{1/2}$) improved due to the formation of an isovalent solid solution between Al_2O_3 and Cr_2O_3. On the other hand, the enhanced hardness (1681 HV/16.5 GPa) is associated with the grain growth inhibition of Al_2O_3. Lastly, the microwave sintering process is used to produce ZTA-3.0 wt.% TiO_2-0.6 wt.% Cr_2O_3 to enhance the microstructure and its properties. The outcome of the process exhibited that increased hardness (1803.4 HV/17.7 GPa) and excellent fracture toughness (9.61 $MPa.m^{1/2}$) are obtained when the sample was sintered at a temperature of 1350°C within a 10-minutes dwelling time. The finding can be attributed to the process of volumetric heating, which led to shorter sintering time and lower sintering temperature. Thus, it produced tool material that has better densification, finer grain size, and excellent mechanical properties.

ABSTRAK

Aluminium oksida adalah sebatian kimia, yang juga dikenali sebagai alumina (Al_2O_3), digunakan secara meluas sebagai bahan dalam alat memotong kerana keunggulan sifat mekanikalnya. Walaubagaimanapun, alumina tidak dapat digunakan secara terus untuk penggunaan praktikal kerana sifatnya yang rapuh. Salah satu penyelesaian adalah untuk mengintegrasikan komponen lain ke dalam bahan asas alumina. Sebagai contoh, menambah zirconia (ZrO_2) ke dalam alumina (Al_2O_3) matriks akan menghasilkan seramik yang meningkatkan keliatan bahan dan keliatannya masih boleh dipertingkatkan lagi. Sebagai penyelesaian, kerja ini bertujuan untuk meningkatkan keliatan patah komposit seramik ZTA dengan penggunaan rawatan haba gelombang mikro dan pelbagai fasa tambahan. Bahagian 1 dan bahagian 2 kajian ini menggunakan proses pensinteran konvensional yang beroperasi pada suhu 1600°C dalam masa 1 jam. Bahagian 3 pula menggunakan 2.45 GHz gelombang mikro untuk proses pensinteran pada suhu antara 1200°C - 1400°C selama 10 minit. Bahagian pertama kajian ini menunjukkan peningkatan ciri-ciri ZTA dengan tambahan 3.0% TiO_2. Malah, kekerasan bertambah baik daripada 1516.13 HV/14.87 GPa (0% TiO_2) kepada 1615.8 HV/15.85 GPa (3.0% TiO_2), manakala keliatan patah meningkat daripada 5.93 $MPa.m^{1/2}$ (0% TiO_2) kepada 6.56 $MPa.m^{1/2}$ (3.0% TiO_2). Selain itu, peningkatan sifat-sifat mekanikal boleh juga dikaitkan dengan kehadiran TiO_2 yang membantu proses pensinteran dengan menghalang pembesaran struktur Al_2O3, dan seterusnya membawa kepada pembentukan mikrostruktur yang lebih padat dan halus. Dalam bahagian kedua, Cr_2O_3 diperkenalkan sebagai bahan tambahan baru ke dalam ZTA-3.0% TiO_2. Hasilnya menunjukkan bahawa sifat-sifat yang berkaitan dengan ZTA-3.0% TiO_2 bertambah baik selepas penambahan 0.6% Cr_2O_3. Keliatan patah (7.15 $MPa.m^{1/2}$) bertambah baik disebabkan oleh pembentukan larutan pepejal isovalent di antara Al_2O_3 dan Cr_2O_3. Peningkatan kekerasan (1681 HV/16.5 GPa) pula dikaitkan dengan perencatan pertumbuhan struktur Al_2O_3. Akhir sekali, proses pensinteran gelombang mikro digunakan untuk menghasilkan ZTA-3.0% TiO_2-0.6% Cr_2O_3 bagi meningkatkan mikrostruktur dan sifat-sifatnya. Proses ini menghasilkan peningkatan kekerasan (1803.4 HV/17.7 GPa) dan keliatan patah yang sangat baik (9.61 $MPa.m^{1/2}$) apabila sampel disinter pada suhu 1350°C dalam masa 10 minit. Dapatan kajian boleh dikaitkan dengan proses pemanasan isipadu, yang membawa kepada tempoh pensinteran yang lebih pendek dan suhu pembakaran yang lebih rendah. Oleh itu, ia menghasilkan bahan yang mempunyai pemadatan yang lebih baik, saiz butiran yang lebih halus, dan sifat-sifat mekanikal yang baik.

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LIST OF ABBREVIATIONS

Al_2O_3	-	Aluminium oxide
Al_2TiO_5	-	Tialite
ASTM	-	American Standard for Testing Materials
B_2O_3	-	Boric Oxide
$CaAl_{12}O_{19}$	-	Hibonite
$CaCO_3$	-	Calcium Carbonate
CeO_2	-	Ceria
Cr	-	Chromium
CaO	-	Calcium oxide
Cr_2O_3	-	Chromia
FESEM	-	Field Emission Scanning Electron Microscope
Fe_2O_3	-	Ferric oxide
HV	-	Vickers Hardness
ICDD	-	International Centre for Diffraction Data
MgO	-	Magnesium oxide
rpm	-	Revolution per minute
SiC	-	Silicon Carbide
SEM	-	Scanning electron microscope
$SrAl_{12}O_{19}$	-	Strontium Hexaluminate
SrO	-	Strontia
TiO_2	-	Titania
TZP	-	Tetragonal Zirconia Polycrystal
XRD	-	X-ray Diffraction
YSZ	-	Yttria-stabilized Zirconia
ZrO_2	-	Zirconia
ZTA	-	Zirconia-toughened Alumina

LIST OF SYMBOLS

d_i - Initial Diameter

d_f - Final Diameter

T_M - Material Temperature

T_C - Critical Temperature

ρ_b - Bulk Density

ρ_{water} - Distilled water’s density at the given temperature

W_a - Weight in air

W_b - Weight in water

N_L - Number of grains intercept/µm

F - Load

K_{IC} - Fracture Toughness

E - Young Modulus

P - Test Load in Vickers Hardness

E_r - The reinforced material’s material property

E_m - The matrix’s material property

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CHAPTER 1

INTRODUCTION

1.1 Background

Ceramic, with its outstanding mechanical properties, such as low density, strength, hardness and its inertness at high temperature, is widely known as a suitable candidate for structural materials with wide ranging applications (Ye *et al*., 2008). Regardless of their advantages, ceramic materials exhibit very low toughness which eventually limits their overall applications (Azhar *et al*., 2012; Azhar *et al*., 2009; Rejab *et al*., 2013; Sktani *et al*., 2014; Smuk *et al*., 2003). The challenge of increasing the toughness of ceramic based materials has been a key motivation in the field of ceramic research (Azhar *et al*., 2012; Basu *et al*., 2004; Magnani & Brillante, 2005). In this pursuit of improving toughness, Al_2O_3 based materials are often used as the benchmark due to its abundance, relative cheapness and excellent mechanical properties (Rittidech *et al*., 2013; Wang *et al*., 2012; Zu *et al*., 2014). The introduction of the yttria stabilized zirconia (YSZ) toughening agent further increased the toughness of the zirconia toughened alumina (ZTA) ceramic composite (Ortmann *et al*., 2012).

As of now, much attention has been given to zirconia-toughened alumina (ZTA) and its possible structural applications for use as a cutting insert material. Single phase of alumina is hard enough to be utilised in cutting tool application. However, it has very low toughness and this can result into breakage and chipping during machining (Aslantas *et al*., 2012; Azhar *et al*., 2009; Zhao *et al*., 2010).

To overcome this drawback, one of the proposed approaches is to reinforce the base material with other components. Incorporating these additives led to better tailored microstructure, a decrease in the sintering temperature, and enhanced product properties. For instance, adding zirconia (ZrO_2) in the alumina (Al_2O_3) matrix helped obtain an advanced ceramic that had better toughness (Ortmann *et al*., 2012). This is because the zirconia dispersed in the alumina matrix increased the material's toughness because of stress induced transformation toughening.

It was revealed that adding titania (TiO_2) allowed it to act as sintering aid (Taruta *et al*., 1997) and helped improve the fracture toughness via secondary phase dispersion (Lee et al., 2003). It was also revealed that TiO_2 promotes the grain growth and sintering of Al_2O_3 (Bagley *et al*., 1970; Maitra *et al*., 2007; Wang and Huang, 2008). This can be attributed to the enhanced diffusivity's effect, where Ti^{4+} substituting for Al^{3+} produced the growing concentration of the Al^{3+} vacancies. As the additive quantity approaches the solubility limit, i.e. 0.15–0.35 mol%, a further increase in the grain growth and densification rate was observed. However, beyond its solubility limit, the contrasting trend of lower grain growth and densification can be obtained. This effect is a consequence of the pinning effect observed at the second phase's grain boundaries, Al_2TiO_5 (Wang and Huang, 2008). TiO_2 encourages the grain growth of ZTA and Al_2O_3. Sufficient grain growth is important to remove residual pores in the material and leads to a dense structure. Thus, TiO_2 is a vital sintering additive that produces a completely homogeneous and dense structure.

Moreover, a lot of attention is being given to chromium dioxide or chromia (Cr_2O_3) in the α-Cr_2O_3 form (Lin *et al*., 2012; Zargar *et al*., 2012). Cr_2O_3 has a density of 5.2 g/cm^3 and a melting point of 2270°. It is also highly resistant to wear and chemicals. However, one primary disadvantage for chromium(Cr) is that it can be very volatile under high temperatures (Sammelselg *et al*., 2010). Arahori and Whitney (1988) reported on the valuable effect of Cr_2O_3 additive. They discovered that the hardness of Al_2O_3 can be enhanced via the isovalent solid solution formation and grain growth inhibition (Azhar *et al*., 2012). Fujita *et al*. (2007) stated that Al_2O_3-Cr_2O_3 ceramic, which is considered a solid composite of Cr_2O_3 and Al_2O_3, remains chemically stable even when it is under high temperatures. Both Al_2O_3 and Cr_2O_3 naturally occur as corundum, their crystalline form. Azhar *et al*. (2012) stated that after the addition of Cr_2O_3 to Al_2O_3, an isovalent solid solution is created. Arahori and Whitney (1988) stated that Cr_2O_3 is integrated to Al_2O_3 ceramics so that the growth of grains can be inhibited and a solid composite solution that has better mechanical properties can be produced. These mechanical properties include increased hardness and better resistance to thermal shock (Azhar *et al*., 2012; Seo *et al*., 2006).

Consequently, the ZTA's mechanical properties depend critically on their microstructures. These mechanical properties can be controlled via densification processes and powder preparation (Wang and Stevens, 1989). On top of developing cost effective processing techniques, material processing improvements are extremely important in manufacturing a better product. The industry's objectives have always been the development of new technologies that can address the growing demands of faster, better, and cheaper products. One of the most vital processes of ceramics is the green compact sintering into final products. Among all the different sintering methods, microwave sintering technology is the one that has attracted the most attention in terms of materials processing, especially for those of oxide ceramics. This is due to the fact that this technology has significant advantages over traditional heating methods. Rapid heating can be accomplished via the microwave sintering technique, since it heats the material by energy conversion instead of energy transfer (Agrawal, 1998). This has significant contributions to volumetric heating (Birnboim and Gershon, 1998), which in turn decreases the densification temperature and shortens the processing time. It also hastens the densification process and produces a material product that has a finer and a more uniform microstructure (Brosnan *et al.*, 2003; Menezes and Kiminami, 2008). Hence, the mechanical properties are enhanced and the performance of the processed materials is significantly improved (Benavente *et al.*, 2014). Moreover, from an economic standpoint, microwave sintering method reduces the production cost since it decreased the sintering time, and thus saves large amounts of energy (Oghbaei and Mirzaee, 2010). Thus, it is expected for microwave heating process to be a promising alternative for conventional heating methods. It has the potential to produce end products that have distinguishable microstructural features and improved mechanical properties.

1.2 Problem Statement

ZTA is considered as one of the most popular ceramic composites that are used for cutting tools. This can be attributed to their excellent mechanical properties like hardness, high wear resistance, corrosion resistance, and high temperature stability. According to Varma *et al.* (2016), ZTA with 1585 HV/15.54 GPa (hardness) and 5.8 MPa.m$^{1/2}$ (fracture toughness) had shorter lifetime when it was subjected to high cutting speed while cutting carbon steel. Thus, consistent efforts should be expanded to achieve improvements in toughness and strength, which then lead to better tool life. The ceramic composites' mechanical properties can be improved via detailed microstructural design. The microstructure of materials can be customized by modifying the proportion, composition, and processing parameters.

In particular, based on the previous works, three gaps have been identified. For example, Bian *et al.* (2012) came up with an Al_2O_3-TiO_2 system that did not require the addition of ZrO_2 powder. However, the composite material produced still had poor toughness because the ZrO_2 element was not present. This ZrO_2 element, via its transformation toughening mechanism, is supposed to lead to higher toughness. Moreover, previous research has discovered that fracture toughness can be enhanced by the creation of elongated grains like tialite (Al_2TiO_5). However, increasing the amount of the elongated grains affected Al_2O_3 densification adversely (Sathiyakumar and Gnanam, 2002), which in turn affected the hardness value of the material. This is due to the fact that the arrangement of the elongated grains hampered the microstructure joining, resulting in reduced material hardness and low density.

Additionally, the work done by Azhar *et al*. (2012) proposed ZTA-Cr_2O_3 ceramic composite. Compared to the ZTA ceramic composite, the fracture toughness and hardness properties were improved. This can be attributed to the production of an isovalent solid solution after Cr_2O_3 was added to Al_2O_3. Arahori and Whitney (1988) stated that the purpose of adding Cr_2O_3 to Al_2O_3 ceramics is for inhibiting grain growth and producing a solid composite solution that possess better mechanical properties, including better resistance to thermal shock and increased hardness (Azhar *et al*., 2012; Seo *et al*., 2006). Moreover, adding Cr_2O_3 led to the production of plate-like grains in the microstructure, which improved the structure's fracture toughness (Riu *et al*., 2000). This phenomenon also leads to a crooked crack path, yet the fracture toughness value obtained by Azhar *et al*. (2012) still needs to be improved to beat the performance of commercial cutting tool. Thus, combining the unique properties of $TiO_{2,}$ ZTA, and Cr_2O_3 can help develop a single composite that has better properties than ZTA commercial cutting tool.

Finally, the utilisation of the conventional sintering method involved the application of high temperature under a long sintering time. In ceramics, both sintering time and temperature have a direct relationship to grain growth. Thus, conventional sintering may lead to abnormal grain growth and in turn cause degradation in the mechanical properties (Breval *et al*., 2005). It has been suggested that microwave heating can be an alternative and enhanced sintering process because it involve a shorter sintering cycle (Oghbaei and Mirzaee, 2010). However, different materials require different sintering temperature and dwell time to achieve sufficient grain growth and densification. As of now, no study has been done comparing the use of conventional and microwave sintering on this particular ceramic composite used in this work.

1.3 Research Objectives

It has been established that the ceramic composites' properties depend on the materials' proportion, combination, and processing parameters. Thus, one should practice thorough selection of the materials to be added and the sintering parameters to be observed. Consequently, this study's specific objectives are as follows:

(i) To study the effects of TiO_2 content on the microstructural, physical, and mechanical properties of ZTA

(ii) To evaluate the influence of Cr_2O_3 content on the microstructure, as well as the mechanical and physical properties of ZTA - TiO_2

(iii) To evaluate the effect of microwave heat treatments, with varying sintering times and temperatures, on the final characteristics and microstructure of ZTA – TiO_2 – Cr_2O_3 ceramic composite

1.4 Scope of The Study

The aim of this study is to synthesise new and tougher ceramic composites by incorporating appropriate second phases in alumina (Al_2O_3), the primary ceramic matrix material. It was observed that adding zirconia (ZrO_2) to the alumina matrix produced an advanced ceramic that had better toughness. This can be attributed to the fact that the dispersion of zirconia in the alumina matrix improves its toughness via stress induced transformation toughening (Hossen *et al.*, 2014). Moreover, zirconia-toughened alumina (ZTA) system was selected as the base material for this current study.

The ceramic powders that were utilised as ZTA's dispersed second phases are titania (TiO_2) and chromia (Cr_2O_3). To study the effects of TiO_2 content in ZTA, part 1 of this work was limited to the addition of 0 wt.% to 10 wt.% TiO_2. This limitation is in accordance to the addition of 0 wt.%-8 wt.% TiO_2 in Al_2O_3-ZrO_2 composite by Zu *et al*. (2014). Part 2 of this work was subjected to the addition of 0 wt.% to 1 wt.% Cr_2O_3 in the best composition of ZTA-TiO_2 composite obtained in part 1. Finally in part 3, the proportions and combinations of ceramic powders that possessed the optimum mechanical properties in part 2 were made to go through microwave hybrid sintering under varying temperature (1200°C-1400°C) and time (5-20 minutes).

1.5 Significance of Knowledge/Contribution

Despite their poor toughness, ceramics cutting tool are gaining more uses in the metal cutting industry because of their superior hardness. Thus, many studies have been conducted with the aim of improving the ceramic material's fracture toughness since it is a main concern in the structural field. Because of this, the field of ceramic research has shifted to the development and usage of multiphase composite ceramics instead of just utilising a single phase material like Al_2O_3. As such, extensive research has been conducted to identify the optimum materials proportion and combination that could satisfy the desirable requirements: i.e. high toughness, high temperature resistance, high hardness, and inertness towards machining parts.

This current research made use of the combination of grain growth inhibition by TiO_2, crack deflection by Cr_2O_3, transformation toughening by YSZ, into the alumina-based composite. It is one of the methods that can be used to overcome the brittle ceramics' lack of toughness. This approach involves the particular selection of the proportion of the materials and the sintering parameters in order to produce ceramic-matrix composites using a classical powder metallurgy technique. The composite is then physically and mechanically characterised. The result obtained will then provide data for the fabricated composites. These data can then be compared to the already known properties of current and existing alumina-based ceramic composites.

It is expected for this study to produce an improved cutting insert material that can be characterised by excellent fracture toughness and hardness. Consequently, these properties can also result in the manufacture of cutting inserts that have improved wear resistance (Azhar *et al*., 2012). Overall, the final product's impact will be lower production costs for the metal cutting industry because the cutting insert will have excellent wear resistance and longer lifetime.

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