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Evaluation of Microstructure and Mechanical Properties of Al_2O_3-TiC Composite Synthesized by Spark Plasma Sintering Process

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Abstract—*Al_2O_3-10TiC composite materials with Ni as a binder were synthesized by high energy ball milling followed by spark plasma sintering (SPS) process. The Ni binder was mixed as sintering additive with two different ratio 1 wt. % and 2 wt. % to improve bonding strenght of the composite. The microstructure of the sintered composite samples reveals the homogeneous distribution of the TiC particles in Al_2O_3 matrix. Physical and mechanical properties of the sintered composite samples prepared by varying sintering time as well as different ratio of Ni binder at constant temperature 1500^0C were investigated. The sample with 2 wt% Ni as a binder and sintered at 1500^0C for 6 min shows a density of 95.55% of their theoretical density and hardness of 16 GPa. These results demonstrate that powder metallurgy combined with SPS is a suitable method for the production of Al_2O_3-10TiC composites with Ni as a binder.*

Keywords*: Composite, spark plasma sintering, Mechanical alloying, Hardness*

1. INTRODUCTION

Alumina based ceramic composite material is widely used in the high temperature structural and wear resistant material [1]. Alumina has low fracture toughness, brittle fracture bheviour and poor sinterability,these properties could be improved by incorporating the reinforcement material in the alumina matrix[1].There are some soft metallic material like Ni, Al, and Cu have been also added to alumina to improve the ductile behaviour [2].The reports are also available which shows that alumina-based ceramic composites reinforced with hard secondary phases such as carbides, borides, and nitrides to produce novel ceramic materials with high strength and toughness have already used in cutting tools [3,4]. The secondary phase titanium carbide have better reinforcing material due to higher melting point better corrosion resistant, better oxidation resistant and also better wear resistant at high temperature [3,4].The Spark Plasma Sintering (SPS) have been widely used for processing of bulk sintered composite materials. SPS is a pressure sintering method, based on high temperature plasma (spark plasma) momentarily generated in the gaps between powder materials by electrical discharge at the beginning of on-off DC pulse energizing. In this process the milled powder is directly used in graphite die and applying pressure with high temperature spark plasma. In contrast to this in conventional sintering due to long holding time and slow heating rate chances of grain growth which is disadvantage for cutting tool materials [5,6]. Also long holding time at high temperature generated the secondary porosity which declines the mechanical properties of sintered composite material [5]. Spark plasma sintering (SPS) or pulsed electric current sintering (PECS) is a sintering technique utilizing uniaxial force and a pulsed (on-off) direct electrical current (DC) under low atmospheric pressure to perform high speed consolidation of the powder.This direct way of heating allows the application of very high heating and cooling rates, enhancing densification[8].In the SPS the rapid heating power is distributed homogeneously over all volume of powder compaction and also homogeneously distributed between gap of powder particle at microscopic level [8].SPS systems offer many advantages over conventional systems using hot press (HP) sintering, hot isostatic pressing (HIP) or atmospheric furnaces, including ease of operation and accurate control of sintering energy as well as high sintering speed, high reproducibility,safety and reliability.The heating rate during the SPS process depends on the geometry of the container/sample ensemble, its thermal and electrical properties, and on the electric power supplier [8].This sintering mechanism and mechanical properties of SPS sintered compact show different characteristics compare to the conventional pressure sintering process.Nano-structured ceramics or nano-composites can be easily prepared by SPS having more densification and fewer defects [8].The metallic binders (Ni) as sintering additive which is enhanced the densification at lower sintering temperature and it also to improve the flexural strength and fracture toughness[7].The melting point of sintering additives Ni has about 1400^0-1450^0C so at high temperature the Ni is completely melt and provided better bonding strength between reinforcement and matrix[7].

In the present work the composite was synthesized by high energy ball milling followed by spark plasma sintering (SPS) process. The effect of Ni binder addition and sintering time on the physical and mechanical property was studied. Mechanical alloying mechanism is used to promote the diffusion process by producing nanostructured while sintering is expected to enhance the diffusion by forming a new phase.

2. EXPERIMENTAL

The starting material were commercially available Al_2O_3 (99.99% purity from Alfa-Asear) and TiC (99.99% purity from Alfa-Asear) powder with average particle size 24 μm and 7 μm respectively. The Ni powder with an average grain size of 4 μm (99.99%, from Alfa-Asear) was used as the sintering additive. Initally Al_2O_3–TiC composite powder was milled in high energy planetary ball milling (PM-400) in dry condition for 30 h. The milling was performed with ball to powder ratio is 10:1 and speed of 150 r.p.m. After this Ni powder (1wt. % and 2 wt. %) was added into Al_2O_3–TiC composite powder and powder mixture was further milled for 5 h. Phase analysis of the milled powder was done using powder diffraction technique with the help of X'Pert PRO PANalytical's materials research Diffractometer (λ = 1.54184 Å) and particle size was analyzed by particle size analyzer (Mastersizer) with water as dispersant which is based on laser diffraction technique. Field emission scanning electron microscopy (FESEM) was used to study the morphology of the milled powder .The composite milled powder with Ni as a sintering additive was sinterd in SPS (Dr. SINTER SPS-625). The powder was directly feed in 10 mm graphite die and sintering was performed for two different sintering time 3 min and 6 min for 1wt.% and 2 wt.% respectively at a constant temperature 1500^0C and pressure 60 MPa with constant heating rate 50^0C/min. After sintering the samples was ejected from the die and further investigated for mechanical and physical properties. The densities of the sintered composite sample were measured by Archimedes principle. The hardness of the samples were measured with the help of vicker hardness tester LECO (AMH43) with diamond indenter in the form of a pyramid having square base (angle of 1360) by applying load 100 gf and dwell time 13 sec. Nano-indentation tests were performed in a UMIS system (Fisher-Cripps, Australia) with a diamond Berkovich indenter at 50 mN load using the Oliver-Pharr method. The load-depth data were analyzed to derive the Young's modulus and hardness values.

3. RESULTS AND DISCUSSIONS

3.1 Powder Characterization

The X-ray diffraction (XRD) pattern (λ=1.54184 Å) of the Al_2O_3-TiC composite with Ni binder 1wt.% and 2 wt.% and without Ni binder were shown in (Fig.1). Presence of only two phases in the XRD pattern indicates that no reaction has been taken place during dry milling of the Al_2O_3-TiC powder for 30 h.The peaks of the Ni also could not observed, it may be due to the lower % of Ni, which is below the detection limit of the XRD system. The particle morphology was observed in field emission scanning electron microscopy (FESEM) and presented in (Fig.2a).The micrograph reveals rounded and regular shapes of the particles. The size distribution of the milled powder particles was measured by Malvern laser particle analyzer and shown in (Fig.2b). Malvern laser particle analyzer consists of a sample holding bath from which particles are pumped and pressed continuously through an annular space. A beam of laser light continuously detects the particle in annular space and fed the sample to the software which reports the particle size. The results show that the cumulative particle size of the particulate matter, where 40-50 vol.% of particle lies is near 5-7 μm.The Specific area was reported as 2.47m^2/g.

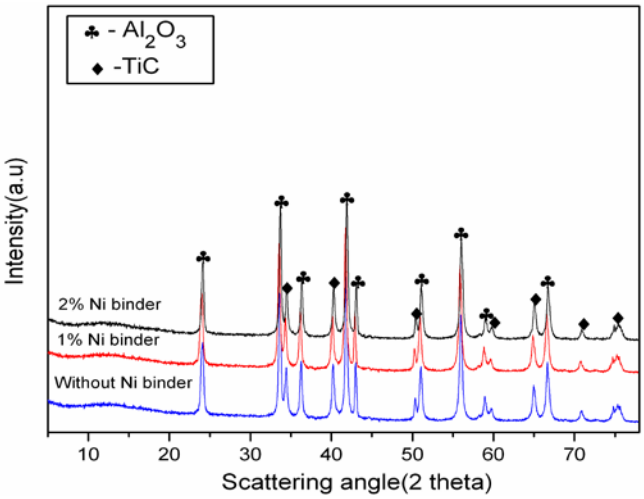
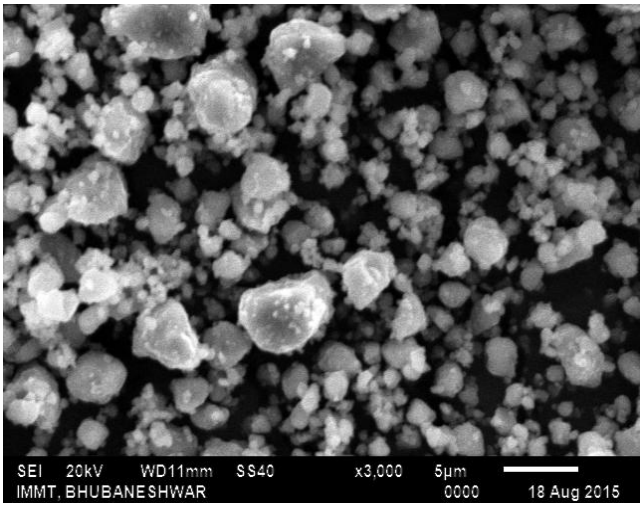
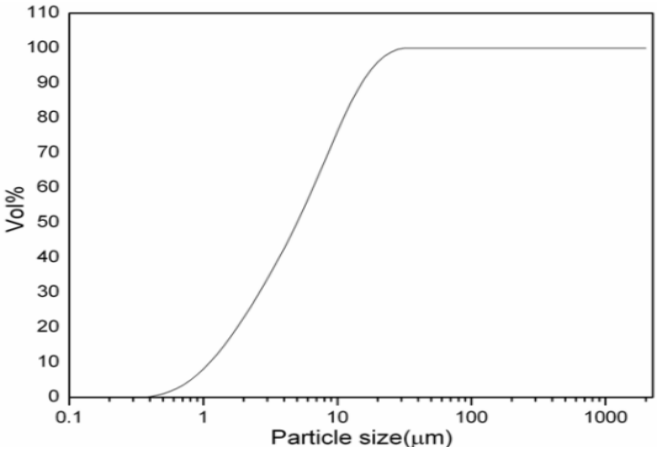


Fig. 1: XRD pattern (Cu radiation, λ=1.54184 Å) of the mechanically milled Al2O3-TiC composite powder



(a)



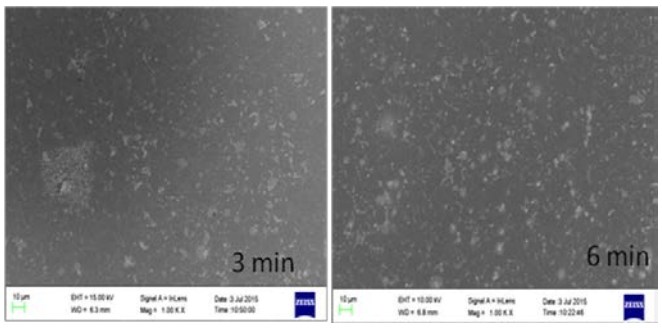
(b)

Fig. 2 (a) SEM migrograph of the mechnically milled powder, showing morphology and distribution (b)particle size distribution in different volume fraction

4. MECHANICAL AND PHYSICAL PROPERTIES

4.1. Microstructure and Densification

The microstructure of sintered composite sample of the samples sintered for 3 min and 6 min (1 wt% Ni and 2wt% Ni respectively) are shown in (Fig. 3a&b). This microstructure of this sintered composite reveals that there is a homogeneous distribution of TiC (white phase) in alumina matrix (gray phase). The TiC phase hindered the crack propagation and also increases the fracture toughness and provided the strengthening the matrix. The densities of the sintered composite material were investigated by Archimedes immersion technique using deionized water as a medium. The maximum density has been achieved at 6 min. sintering, which is around 95.55% of their theoretical density and for 3 min. sintering density was found to be 95.26%. Increase of sintering time indicate that the microstructure is more dense for a longer sintering time and the average grain size increased with increase of the sintering time, that corresponded to FSEM results (Fig. 3 a&b). More sintering time provides more diffusion at the interface that results better interface bonding and increase density.



(a) **(b)**

Fig. 3: SEM micrograph of the sintered composite at 1500^0C(a) 3 min and (b) 6 min

4.2 Hardness measurement

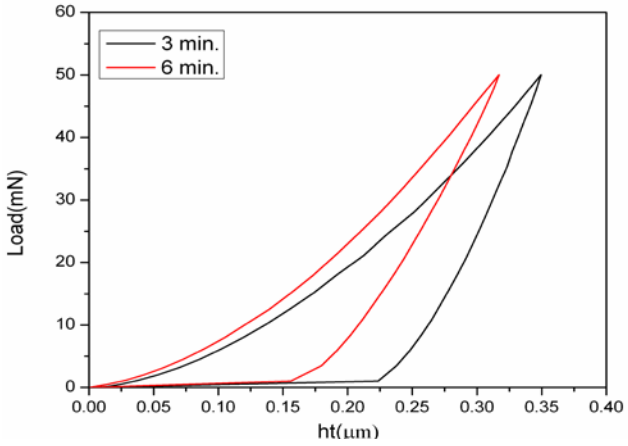
The hardness of the sintered composite material was investigated by vicker's hardness tester with load 100gf and dwell time 13 sec. The hardness has been observed to be increased linearly from 15 GPa to 16 GPa with increase of sintering time from 3 min to 6 min (see table-1). This is attributed to increase of density and decrease of porosity at higher sintering temperature because higher sintering temperature favors shrinkage of the open pores and the grain boundary diffusion [10]. This leads to strong ceramic bond between the interfaces of the particle as resulting in increased hardness of the material.This densification also leads the bonding sterengths between the matrix and reinforcement with metallic sintering additives Ni powder.

Table 1: The hardness and density relation of Sintered composite sample

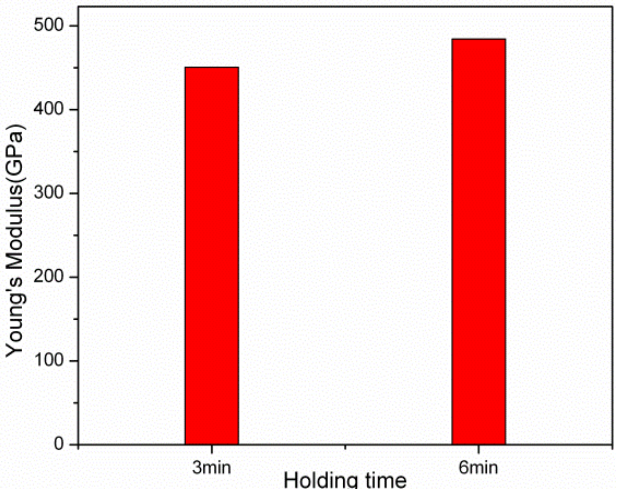
Holding stage	Hardness	Densification
3min	15GPa	95.26%
6min	16GPa	95.55%

5. NANO-INDENTATION OF COMPOSITE MATERIAL

The modulus of elasticity and stiffness were measured by nano-indentation test, which is based on Oliver-Pharr method. This test provided the data between load-depth. There are two steps in this methods first is loading and second is unloading of each sintered composite material. The loading curve indicates as expected, an increasing resistance to deformation with increasing depth in indentation and unloading curve which indicates that maximum recovery of the position of indentation depth and also indicates that permanent deformation has taken place[9].The area under curve lower between loading and unloading is shows that maximum modulus of elasticity and stiffness. Thus the maximum modulus of elasticity 485 GPa has been achieve at 6 min holding stage and lower modulus of elasticity 451 GPa has been achieve at 3 min holding stage of this sintered composite material.



(a)



(b)

Fig. 4: (a) Nanoindentation curve P(mN)-ht(µm) (b) Young's modulus of elastic of Al2O3-TiC+Ni composite material.

6. CONCLUSION

Al_2O_3-10TiC composite material with Ni as sinteing additives has been successfully preapared by high energy ball milling followed by spark plasma sintering process.The ball milling reduces particle size as well as improve interface bonding, which resulted incrase of density ahnd hardness of the composite material. Sample sintereted at 1500^0C for 6 min at 60MPa leads to significant incrase of denssity(95.55% of theoretical density) and hardness (16GPa).The Young's modulus of elasticity has been also obtained higher in 6 min sintering to the value of 451 GPa.

7. ACKNOWLEDGEMENTS

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