Hardness and Fracture Toughness of Ceramic Composite Using Experimental and Analytical Methods

K. I. Vishnu Vandana, K.N.S. Suman

Abstract: ***The present work investigated and discussed the impact of addition of graphene to Al_2O_3 ceramic matrix (alumina) and its effect on different mechanical properties of resulting alumina-graphene (Al-G) composite tool material. Alumina – Graphene (Al-G) ceramic composite tools were prepared through powder metallurgy technique by maintaining different weight proportions of graphene. The wt% is varied from 0.15 to 0.65 with an interval of 0.1%. Hardness and Fracture toughness properties were tested and these properties were observed to be increased at lower content of graphene up to 0.45wt% and later on a decrement trend was observed with increased content of graphene. The composite with 0.45wt% of graphene yielded the highest hardness (HV) and fracture toughness (K_{IC}) parameter values at an indentation load of 294N. The composite specimens were prepared through Microwave sintering of powder metallurgy technique to ensure uniform grain structure to the resulting composite.***

Keywords: ***Alumina –Graphene, Ceramic Composite, Fracture toughness, Hardness, microwave sintering***

I. INTRODUCTION

With the increasing demands of the manufacturing world, need of high efficiency and high accuracy in machining is also becoming vital in the manufacturing practice [1]. Ceramic cutting tools based on alumina materials own high advantages because of having high hardness and corrosion resistance and can be applied in case of high-speed machining. In the interim, their cutting performance is better when compared with traditional tools performance in hard turning. So, several researchers considered ceramic cutting tool materials as a competitive material for tools [2]. But, their brittleness is limiting their application as a tool. So far, few methods were proposed and suggested to increase the strength and toughness of the alumina-based ceramic tool materials. Several authors suggested to transform the ZrO2 phase in to ceramic matrix. The expanded particles of ZrO2 generated both shear stresses and dilatational stresses. And it was finally concluded that these both stresses prohibited the crack proliferation [3]. An alternative way to reduce brittleness is by reinforcing nano particles, like SiC, and TiN [4,5] in to alumina ceramic composites. Nano particles refine the grain by pining in to the grain boundaries and improve the strength. Now a days graphene is attracting the materials world for its remarkable mechanical properties [6]. When compared with other fillers, graphene is having larger area specific surface and good dispersion capability in to the matrix. These qualities are making graphene as an ideal material to replace most of the fillers in the alumina matrix [7]. Many authors suggested graphene as filler in different ceramic materials and found enhanced mechanical properties like fracture toughness in ceramics [8,9,10]. Liu et al. added graphene platelets (GPLs) to alumina ceramic and reported increased flexural strength and fracture toughness [11]. And several studies identified the importance of sintering in prompting the mechanical characteristics of obtained composites. Many authors adopted spark plasma sintering (SPS), HF-IF sintering and hot pressing (HP) to prepare alumina-graphene composites [12],[13],[14]. Chen prepared a composite of alumina/graphene with the incorporation of 0.2 wt% GNS through HP process and reported increased fracture toughness [15]. Walker employed SPS technique to prepare Si3N4/graphene ceramic composite and reported increased fracture toughness [16].Unusual grain growth and large sintering time are the main drawbacks of HP sintering. And high energy required by SPS also made the processes difficult to practice with economic feasibility [17]. Microwave sintering has been practiced by some authors [18,19] in fabricating composite. They concluded that it is reducing the cost and at the same time increasing the efficiency. In Microwave sintering heat is mainly generated due to reaction between total specimen and electromagnetic waves generated during sintering [20, 21]. As a result, fast heating rate and homogeneous distribution of temperature was observed in the material. Many studies have demonstrated and developed different methods of using of graphene as filler to improve the different mechanical properties of ceramic material. But, till now only few works addressed the impact of graphene and microwave sintering process on the mechanical characteristics and performance of alumina (Al2O3) ceramic composites as a tool. In the present study, alumina –graphene (Al-G) composite tool material was fabricated by using microwave sintering. Graphene was added in to the alumina matrix basing on the different weight percentages ranging from 0.15wt% to 0.65wt%. Hardness and (K_{IC}) fracture toughness values of different alumina –graphene (Al-G) composites at a load of 294N were obtained from experimental results.

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These values are analysed and discussed to acknowledge the enhancement of various mechanical properties of ceramic composite with graphene as filler. Substantial enhancement in the hardness, toughness of prepared alumina – graphene ceramic composite tool may expose to a novel field of cutting applications.

II. ALUMINA CERAMIC COMPOSITE MATERIAL PREPARATION

Alumina and graphene powders obtained from AD-NANO TECHNOLOGIES (Bangalore) were used as raw materials to prepare the composite ceramic material in the present investigation. The particle nominal size of Al_2O_3(Al) is 300 nm and graphene is about 3-8 nm. Graphene was loaded basing on wt% in to alumina at an interval of 0.1 ranging from 0.15 to 0.65 wt%. The powders are mixed and well processed in the presence of ethanol using a ball mill for four hours to ensure uniform mixture. A hydraulic press is used to press and prepare Al-G composite samples in the required shape. The pressed green samples were placed in SiC (powder) filled crucible to prevent oxidation of samples. Now these crucibles along with samples are sintered in microwave furnace. Sintering of all samples was done at 1500⁰c holding for 30 min, as highest density was observed at this temperature. Sintered samples density was measured with the help of Archimede's principle. The compositions and densities of ceramic tool materials were displayed in the following Table 1.

Table 1 : Density Vs Theoretical Density of Prepared samples

Composition of Graphene in Alumina wt%	Sample	Sintering Conditions	Density (g/cc)	Theoretical Density (%)
0.15	Al-G (0.15 wt%)	1500°C/30Min	3.85	96.95
0.25	Al-G (0.25 wt%)	1500°C/30Min	3.89	98.2
0.35	Al-G (0.35 wt%)	1500°C/30Min	3.96	99.9
0.45	Al-G (0.45 wt%)	1500°C/30Min	3.89	98
0.55	Al-G (0.55 wt%)	1500°C/30Min	3.85	97.12
0.65	Al-G (0.65 wt%)	1500°C/30Min	3.81	96.11

III. III. TESTING OF HARDNESS AND FRACTURE TOUGHNESS (KIC)

While choosing a material for cutting tools, selection is to be based on two crucial parameters i.e hardness and fracture toughness. So, in this work, after preparing the specimen with powder metallurgy and microwave sintering technique, the hardness values and fracture toughness values are computed. The hardness test has been conducted at IIT Basara, Telangana, India using Vickers Hardness Tester. To maintain the accuracy in the result, at each combination of constituents of ceramic composite, 4 specimens were prepared. These specimens were well polished on single disc polishing machine by using diamond paste of different micron size such as 5, 3, 1, 0.5microns etc.. to achieve the mirror finish appearance on the specimens. To perform this operation, Pyramid shaped diamond tip was selected and indentations were taken at a load of 294N (30Kg) with a dwell period of 10seconds. Indentations were taken at 10 different locations of same sample and values are averaged to get the complete information of hardens of prepared specimens.

Along with the hardness of the ceramic composites, fracture toughness is also evaluated to test the specimens by using analytical approach. Fracture toughness values were computed using fallowing empirical relationship Eq.1. proposed by Evans y Charles [22].

$$K_{IC} = 0.16 \times (c/a)^{-1.5} \times \sqrt{HV} \qquad \text{Eq.1}$$

Where HV is Vickers Hardness (HV), 'c' is length of cracks (averaged) obtained from tips of vicker marks and finally 'a' is the length of diagonals (mean length) that we obtained from Vickers hardness test.

IV. EFFECT OF GRAPHENE ON MORPHOLOGY OF COMPOSITE

Fig.1 and Fig 2. are Showing the grain image of ceramic composite at 0.35 and 0.55 wt% of graphene.

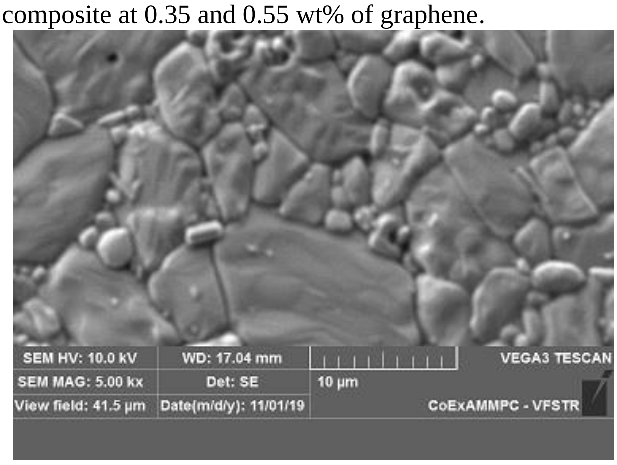

Fig.1: SEM Image of Al-G (0.35 wt% of graphene)

SEM image in Fig.1 is clearly revealing the uniform distribution of graphene in ceramic matrix.

Fully dense ceramic composite were able to obtain due to reduced time of sintering and rapid heating rates of novel Microwave sintering technique. This technique limits the damage to the graphene due to prolonged exposure at high temperature which is present in other sintering methods. This even distribution of graphene is the vital factor in enhancing relative density parameter of resulting composite. Sample which consisting of 0.35wt% of graphene is exhibiting highest relative density and compaction. But from 0.45wt% this parameter is decreasing. This may be due to agglomeration of graphene particles at higher concentrations. This agglomeration is increasing further with increasing content of graphene from 0.55wt% of graphene as shown in Fig.2.

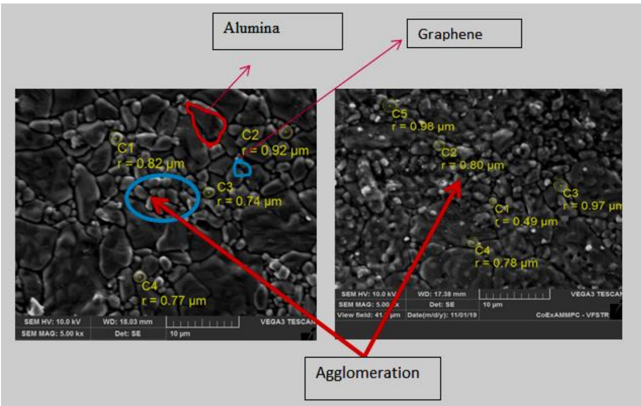

Fig.2: SEM Image of Al-G (0.55 wt% of graphene)

V. V. RESULTS: HARDNESS AND FRACTURE TOUGHNESS

The measured Fracture toughness and Vickers hardness values of composites (Taking indentation load of 294N) with different graphene wt% are given in the following Table.2.

Table.2: Hardness and Fracture Toughness of composite samples

Composition of Graphene in Alumina wt%	Sample	Hardness (GPa)	Fracture Toughness (MPa $m^{1/2}$)
0.00	Al-G (0Wt%)	15.72	2.68
0.15	Al-G (0.15wt%)	16.80	3.42
0.25	Al-G (0.25wt%)	17.76	3.78
0.35	Al-G (0.35wt%)	19.04	4.32
0.45	Al-G (0.45wt%)	21.27	4.91
0.55	Al-G (0.55wt%)	18.41	3.82
0.65	Al-G (0.65wt%)	16.97	3.25

From the Table.2 it is clearly evident that hardness and fracture toughness values of the ceramic composite was increasing up to 0.45wt% of graphene. But further increment of graphene is showing a decrement trend in the resulting hardness and fracture toughness values of the ceramic composite and was displayed in Fig. 3. When compared with monolithic Al_20_3 samples hardness of 15.72 GPA, prepared under same conditions, a noticeable improvement of 35% is obtained in hardness of Al-G (0.45wt %). This is due to the interspace between the grains of constituents of the ceramic composite. The size of grains of alumina is more compared to the graphene. The interspace distance between the alumina, accommodate few percentage of nano graphene i.e up to 0.45 wt%. After that, these nano graphene is agglomerated which diminishes the hardness of respective composite. This agglomeration can be clearly observed in Fig.2.

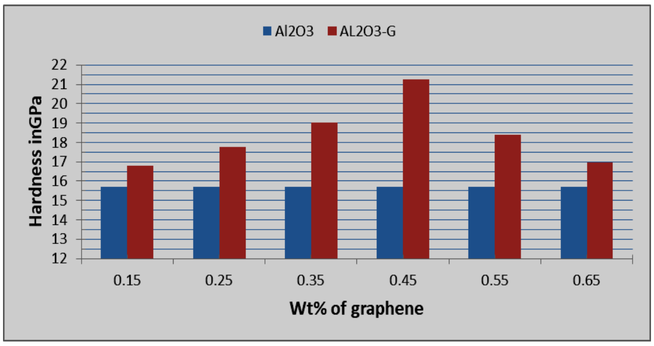

Fig. 3.Variation of Hardness with respect to Wt% of graphene

Fig.4. shows the variation of fracture toughness of ceramic composite calculated by using analytical formula. This analytical formula used the data of hardness and fracture lengths which are obtained from Vicker's hardness experimental results. The fracture toughness increased for composites up to 0.45wt% of graphene and later decrement trend was observed in composites from 0.55wt% of graphene.

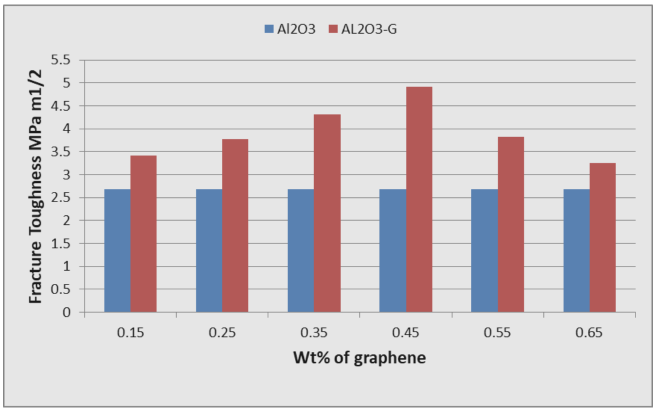

Fig. 4. Variation of Fracture Toughness with respect to Wt% of graphene

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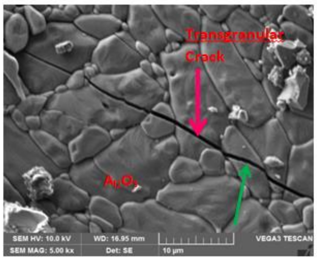

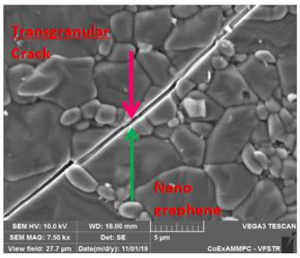

Fig.5. SEM Image of Al-G(0.15wt%)
Fig.6.SEM Image of Al-G(0.45wt%

The addition of graphene into the Al_2O_3 changes the fracture behaviour which can be understood by Fig.5 and 6. The Fig.5. Shows the SEM image of ceramic composite at lower wt% (0.15) of graphene and Fig. 6. shows the image of ceramic composite at 0.45wt% of graphene.

The addition of Nano graphene into the Al_2O_3 changes the mode of fracture from intergranular into transgranular. With the addition of more nano graphene, composite is getting more protection as the grain of graphene takes the major load. Another important point noticed from the present work is at lower weight fraction of graphene, the crack prone to extend through the grain of Al_2O_3 and will be changed towards graphene Fig.7. Fracture toughness values for unreinforced Al_20_3 and Al-G nano composites were determined. The fracture toughness values of unreinforced Al_20_3 was 2.68 $Mpa.m^{1/2}$ and it was increased by 83% for Al-G(0.45wt%) sample at a load of 294N.

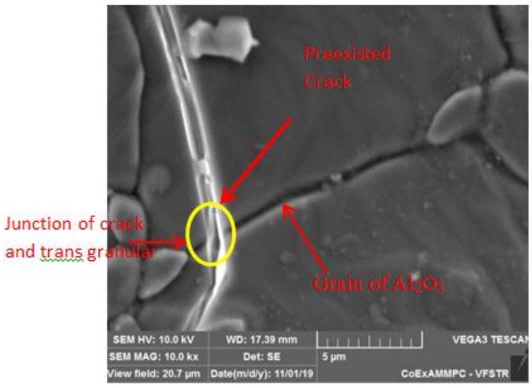

Fig.7. SEM Image of Al-G (0.15 wt% of graphene)

VI. CONCLUSIONS

Alumina and graphene composites are finding in many applications and one of the applications is cutting tools. To suggest theses material to the cutting tools, the vital factors to be evaluated are hardness and fracture toughness. Using powder metallurgy technique with micro wave sintering, the composite material has been prepared with different composition of graphene i.e 0.15 to 0.65 wt% in the alumina. The following conclusions have been drawn from present work.

- The hardness of the resulting alumina graphene composite is increases by increasing the graphe percentage up to 0.45 wt%. Later decrement in the same property is observed from 0.55wt% of graphene.
- The fracture toughness of the present composite is increasing at lower wt% of the graphene and is decreasing with the increasing graphene content beyond 0.45wt%.
- The type of failure in the constituent is changed from alumina to graphene due to the reinforcement effect. It is suggested to use more graphene content to change the fracture behaviour of ceramic composite. With the graphene reinforcement, the crack extension or propagation will be delayed through the composite showing less fracture toughness at higher weight fraction.

REFERENCES

1. H. Schulz, T. Moriwaki, High-speed machining, CIRP. Ann. Manuf. Technol. 41 (1992) 637–643.
2. W. Grzesik, Wear development on wiper Al2O3–TiC mixed ceramic tools in hard machining of high strength steel, Wear 9 (2009) 1021–1028.
3. W.H. Tuan, R.Z. Chen, T.C. Wang, C.H. Cheng, P.S. Kuo, Mechanical properties of Al2O3/ZrO2 composites, J. Eur. Ceram. Soc. 22 (2002) 2827–2833.
4. Z.S. Rak, J. Czechowski, Manufacture and properties of Al2O3-TiN particulate composites, J. Eur. Ceram. Soc. 18 (1998) 373–380.
5. B. Baron, C.S. Kumar, G.L. Gonidec, S. Hampshire, Comparison of different alumina powders for the aqueous processing and pressureless sintering of Al2O3– SiC nanocomposites, J. Eur. Ceram. Soc. 22 (2002) 1543–1552
6. Ujjal kumar Sur, Graphene: A Rising Star on the Horizon of Materials Science, International Journal of Electrochemistry,Volume 2012, Article ID 237689, 12 pages, http://dx.doi.org/10.1155/2012/237689
7. Graima Mittal ,Vivek Dhand, Kyong Yop Rhee, Soo-Jin Park, A Review on Carbon Nanotubes and Graphene as Fillers in Reinforced Polymer Nano composites, Journal of Industrial and Engineering Chemistry 21 · March 2014, DOI: 10.1016/j.jiec.2014.03.022.
8. J. Dusza, J. Morgiel, A. Duszová, L. Kvetková, M. Nosko, P. Kun, C. Balázsi, Microstructure and fracture toughness of Si3N4+graphene platelet composites, J. Eur. Ceram. Soc. 32 (2012) 3389–3397.
9. H. Porwal, P. Tatarko, S. Grasso, J. Khaliq, I. Dlouhý, M.J. Reece, Graphene reinforced alumina nano-composites, Carbon 64 (2013) 359–369.
10. Tomasz Cygan, Jaroslaw Wozniak, Marek Kostecki, Andrzej Olszyna, Mechanical and Tribological Properties of Alumina – Graphene Composites, Proceedings of the World Congress on New Technologies (NewTech 2015) Barcelona, Spain – July 15 - 17, 2015 Paper No. 452
11. J. Liu, H. Yan, K. Jiang, Mechanical properties of graphene platelet-reinforced alumina ceramic composites, Ceram. Int. 39 (2013) 6215–6221.
12. Z.B. Yin, C.Z. Huang, B. Zou, H.L. Liu, H.T. Zhu, J. Wang, Study of the mechanical properties, strengthening and toughening mechanisms of Al2O3/TiC micro-nanocomposite ceramic tool materials, Mater. Sci. Eng. A. 577 (2013) 9–15
13. Ahmad " Multilayer Graphene (MLG)-Reinforced Al2O3 Nanocomposites Fabricated by High-frequency Induction-heat Sintering Technology" Manufacturing Science and Technology 3(4): 98-105, 2015.
14. Iftikhar Ahmad ⁎, Mohammad Islam , HanySayedAbdoa, TayyabSubhani , Khalil Abdelrazek Khalil , Abdulhakim A. Almajid , BaharehYazdani , YanqiuZhud " Toughening mechanisms and mechanical properties of graphenenanosheet-reinforced alumina" Materials and Design 88 (2015) 1234–1243.
15. Y.F. Chen, J.Q. Bi, C.L. Yin, G.L. You, Microstructure and fracture toughness of graphene nanosheets/alumina composites, Ceram. Int. 40 (2014) 13883–13889.
16. L.S. Walker, V.R. Marotto, M.A. Rafiee, N. Koratkar, E.L. Corral, Toughening in graphene ceramic composites, ACS. Nano 5 (2011) 3182–3190.
17. I. Ahmad, B. Yazdani, Y.Q. Zhu, Recent advances on carbon nanotubes and graphene reinforced ceramics nanocomposites, Nanomaterials 5 (2015) 90–114.
18. R. Benavente, M.D. Salvador, F.L. Penaranda-Foix, E. Pallone, A. Borrell, Mechanical properties and microstructural evolution of alumina–zirconia nanocomposites by microwave sintering, Ceram. Int. 40 (2014) 11291–11297.

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19. Y. Cheng, S.S. Sun, H.P. Hu, Preparation of Al2O3/TiC micro-composite ceramic tool materials by microwave sintering and their microstructure and properties, Ceram. Int. 40 (2014) 16761–16766.
20. R. Roy, D. Agrawal, J.C. Amp, S. Gedevanishvili, Full sintering of powdered-metal bodies in a microwave field, Nature 399 (1999) (304-304).
21. L. Acevedo, S. Usón, J. Uche, Exergy transfer analysis of microwave heating systems, Energy 68 (2014) 349–363.
22. Evans A. G. and Charles E. A. (1976). Fracture Toughness Determination by Indentation, J. Am. Ceram. Soc., Vol. 59, (1976), pp. 371-372.

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