Effect of Speed on Tribological Properties of Graphene Toughned Alumina (Al-G) Composite

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

Abstract*: *This research work discusses the influence of different wt% of Graphene addition to Alumina matrix and its impact on mechanical properties such as hardness and wear properties of prepared composites (Al-G). The Al-G composites were prepared using powder metallurgy technique with the help of microwave sintering. The hardness of prepared specimens was tested using the Vickers hardness test for various combinations of Graphene in alumina matrix. The wear behaviour of material is explored using Pin on disk apparatus at various speeds and sliding distances for different wt% of graphene added alumina samples. The weight percentages of graphene vary from 0.1 to 0.4wt% with the interval of 0.1%. It was observed that the presence of graphene in alumina and processed with microwave sintering has a significant effect on wear properties of the Al-G composite. The present work focussed on the improvement in wear resistance through microwave sintering of alumina with the addition of various wt% of graphene. The main aim is to explore best optimum AL-G composition for increased wear resistance.

Index Terms*: *Alumina, Graphene, Wear properties, Microwave sintering, nano powders.

I. INTRODUCTION

The day to day demand for new ceramic materials for varied engineering applications was increasing substantially due to increased demand in the improvement of different properties like density, strength, stiffness etc. In such a situation, ceramics like aluminium and its composite are drawing the attention of researchers due to their wide variety of applications in different areas. The addition of materials like SiC, carbon nano tubes etc as a second stage was viewed as a standout amongst the most encouraging methods for enhancing the mechanical properties of alumina-based ceramics. The size of the particle reinforcement has also a significant effect on mechanical behaviour of the composite material [1]. So preparation of the ceramic structures at nanometre became a leading research frontier .Addition of graphene oxide to alumina matrix has significant influence on mechanical properties and cutting performance of obtained composites [2]. PiotrKlimczyk et al [3] investigated the effect of graphene nano platelets (GNPs) addition to alumina matrix on the microstructure, selected physical – mechanical properties and wear behaviour of Al_2O_3– graphene composites. Studies were carried on the nickle-coated graphene (GnNi) mixed with alumina matrix and its effect on cutting performance of obtaining composites [4]. But it was identified that reinforcement of graphene in alumina matrix is more effective interms of mechanical properties [5.6]. Many studies were carried out on graphene based alumina composites and worked towards increasing the mechanical properties of derived composite. And it was observed that, in the preparation of these ceramic matrices, sintering also plays a crucial role in influencing the mechanical and tribological properties of obtaining composite. Several investigators adopted different sintering methods like spark plasma sintering, hot pressing and HF-IF sintering to prepare composite of alumina-graphene[1],[7],[8]. Several authors studied the effect of various sintering parameters al soon mechanical and tribological properties of obtained composite [9].Improved mechanical properties, including flexural strength, hardness and fracture toughness was observed by X.L. Shia et al [10]when used hot pressing to prepare Al_2O_3/SiC composites. Hot pressing sintering process has much effect on densification and microstructure and material properties of Al_2O_3-3YSZ-Ni composite [11]. Considerable work has been done on spark plasma sintering process also. X Lui et al prepared a material with graphene nano sheets (GNSs)/Al_2O_3 composites using spark plasma sintering and observed the improvement in mechanical performance of the concerned materials [12]. But in the later stages the differences between the traditional sintering with microwave process were highlighted in the ceramic community [13-14].On the other side, the intensity of microwave processing of nano phase materials was studied. The number of consequences, such as the development of a heat source within the specimen, preferential heating of porous regions, and in some cases plasma formation was explored [15].Microwave joining yields joint strength in excess of the base material strength [16].The benefits of microwave processing in the view of cost and the energy involved in microwave processing are compared to the conventional methods [17]. From the above review, it is observed that most of the authors prepared Al_2O_3 and graphene based ceramic composites using Hot Pressing and Spark Plasma Sintering. But both of these sintering methods were not economical as long sintering time is needed for hot pressing and high energy is needed for spark plasma sintering. In the present work we are proposing microwave sintering to fabricate ceramic composites, in order to increase the efficiency and at the same time to reduce the cost of processing. In case of Microwave sintering, temperature distribution is more homogeneous within the materials and the heating is also more rapid. Even though some authors

Revised Manuscript Received on July 05, 2019.

K. I. Vishnu Vandana[1], Department of Mechanical Engineering P.V.P. Siddhartha Institute of Technology, Kanuru, Vijayawada, AP.

Dr. KNS Suman[2], Department of Mechanical Engineering, Andhra University, Vishakhapatnam, Ap.

reported their work on microwave sintering, still there is a scope to reveal the behaviour of microwave sintering process on mechanical characterization of ceramic tools. The present work addresses the effect of microwave sintering on the mechanical and wear properties of prepared Al-G composites.

II. EXPERIMENTAL METHODOLOGY

The industrially accessible α-Al_2O_3 powder (99.95%, D50 300 nm) and Graphene nano powder were utilized as antecedents for this investigation. The load level of graphene in alumina was kept at a range of 0.1-0.4% in the present examination. The powdered materials are blended and processed with ethanol for four hours in room temperature on a planetary ball mill processor. The load proportion of balls to powder was kept as 30 : 1, and the tungsten carbide vial rotation rate was kept at 300 rpm. Using SEM, Microstructuralobservations were carried out on milled powders. Using a Hydraulic Press, the dried and sieved powdered composite (Al-G) was pressed uniaxially. It was carried at a load of 6 Tonne in a tungsten die which is 12mm in diameter. Sintering was carried on pressed samples using Microwave Furnace (Fig 1) in controlled environment at three distinct temperatures 1300°C, 1400°C and 1500°C for 30 minutes each.

Fig 1: Microwave furnace

Fig 2: Pin shaped samples

a. *Density and Hardness of Al-G composite:*

After completion of specimen preparation, the next phase of the work is focused on quantification of density and hardness using Archimedes principal and the Vickers hardness test at various compositions of Al-G which are provided in Table.1. From the table it is clearly evident that , the 0.1, 0.2 , 0.3 , 0.4 wt % of Graphene toughened alumina samples sintered in microwave at 1500°C for 30 minutes , yielded high relative densities when compared the densities at other sintering temperatures. So remaining all samples at different wt% of graphene are sintered at 1500°C for 30 minutes to get the advantage of high density. Hardness values of all sintered samples were measured using Vickers hardness test at 10kgf load for 10 seconds by employing diamond shaped indenter and these indentations were observed under SEM. The hardness values are as follows in Table 2.After wear test, scanning electron microscopy (SEM) was used to examine the worn surfaces of samples. The wear rate of each sample was calculated by using the fallowing formulae.

$$\Delta w = w_i - w_f$$

$$Wear\ rate = (\Delta W)/\rho * S$$

w_i = Initial weight of sample in grams

w_f = Final weight of sample in grams

Δw = Weight loss in grams

ρ =Density of material in g/cc

L= Applied load in N

S= Sliding distance in meters

Table.1. Density Vs Theoretical Density from samples

Composition of Graphene in Alumina wt%	Sample	Sintering Conditions	Density (g/cc)	Theoretical Density (%)
0.1	Al-G (0.1wt%)	1300°C/ 30Min	3.25	82
		1400°C/ 30Min	3.6	91
		1500°C/ 30Min	3.82	96.2
0.2	Al-G (0.2wt%)	1300°C/ 30Min	3.25	82
		1400°C/ 30Min	3.7	93
		1500°C /30Min	3.88	98
0.3	Al-G (0.3wt%)	1300°C/ 30Min	3.4	86
		1400°C/ 30Min	3.84	96.7
		1500°C/ 30Min	3.96	99.9
0.4	Al-G (0.4wt%)	1300°C/ 30Min	3.28	82
		1400°C/ 30Min	3.72	94
		1500°C/ 30Min	3.89	98

Table 2: Hardness Values of Prepared composite samples

Material	Hardness Value (HV)
Al-G (0.1wt%G)	1680
Al-G (0.2wt%G)	1710
Al-G (0.3wt%G)	1750
Al-G (0.4wt%G)	1760

b. Wear testing of Samples:

In the present experiment, Pin on disk machine is used to carry out the two body abrasion tests. The tests were carried at a speed of 0.5m/Sec - 1.5m/Sec and sliding distance of 50m -150m on silicon carbide emery paper with a grit size of 150 µm. Before experimentation, the surface morphology of emery paper was examined by SEM and was represented by Fig 3. This emery paper was served as an abrasive medium. It was cut to size and bonded to a wheel of 160 mm diameter. The specimens were weighed before and after experimentation to measure the weight loss during wear test.

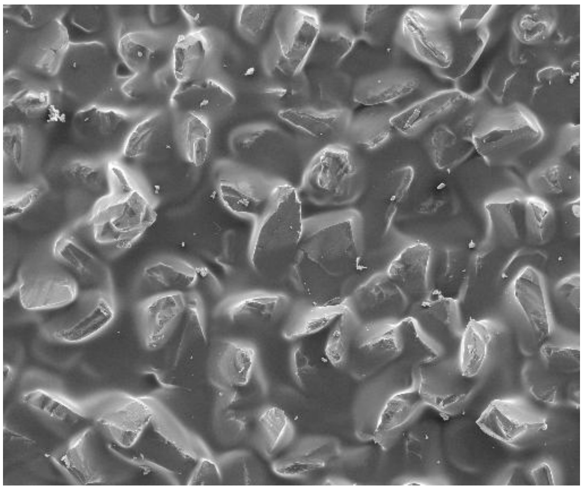


Fig 3: Surface Morphology of emery paper before wear test

c. Examination of worn surfaces of pin samples, emery papers and debris:

After abrasive wear tests, the worn out surfaces of different samples were examined to learn and predict the possible changes in the mechanism of removal material removal. The pictures were captured in SEM at 1000X amplification. Fig 4a - 4d show the abrasive worn surface topography of 0.1wt%, 0.2wt%, 0.3wt% and 0.4wt% of Al-G composites respectively tested at lower speeds i.e 0.5m/sec. Fig 4e - 4hrepresents the abrasive worn surface topography of 0.1wt%, 0.2wt%, 0.3wt% and 0.4wt% of Al-G composites respectively tested at higher speeds i.e 1.5m/Sec.

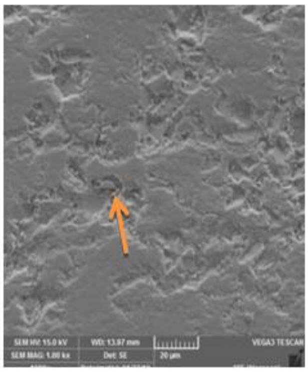


Fig 4a :0.1wt% Al-G

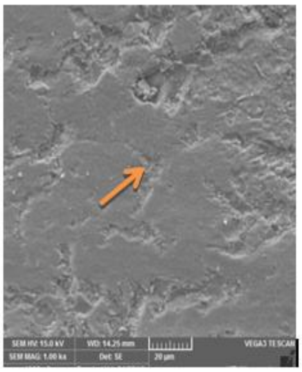


Fig 4b : 0.2wt% Al-G

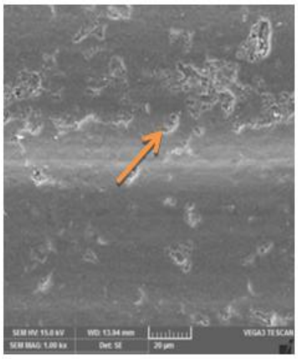


Fig 4c : 0.3wt% Al-G

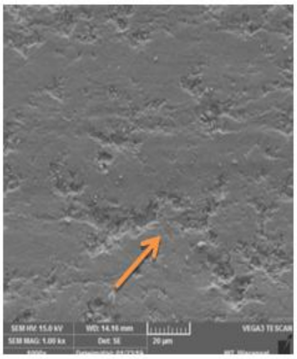
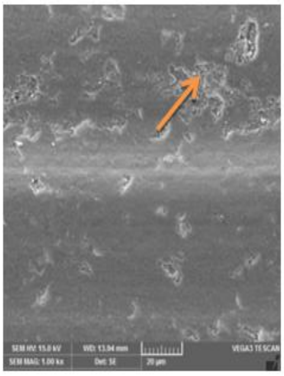


Fig 4d : 0.4wt% Al-G



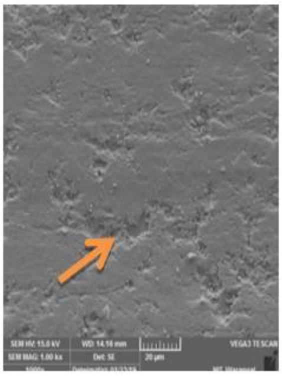
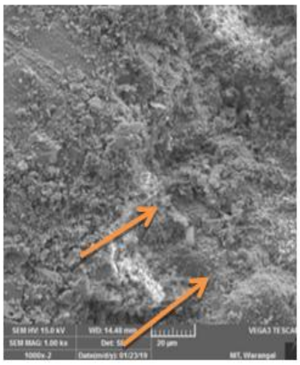


Fig 4c : 0.3wt% Al-G **Fig 4d : 0.4wt% Al-G**



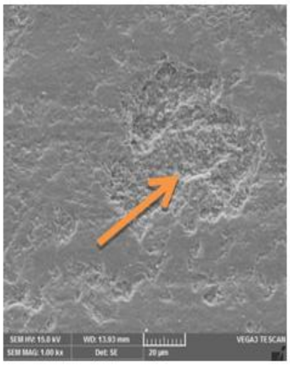
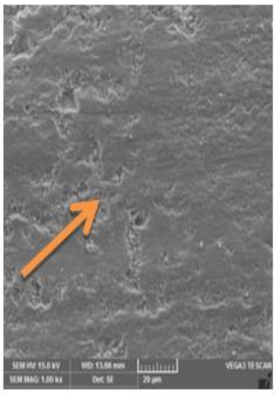


Fig 4e : 0.1wt% Al-G **Fig 4f : 0.2wt% Al-G**



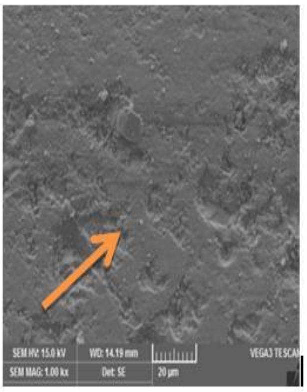
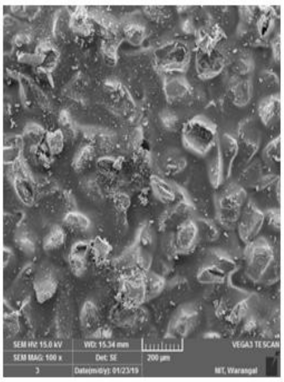


Fig 4g :0.3wt% Al-G **Fig 4h : 0.4wt% Al-G**

Fig 4: abrasive worn surface topography of samples



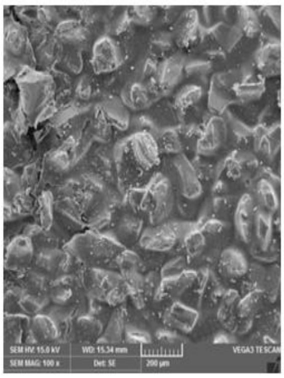
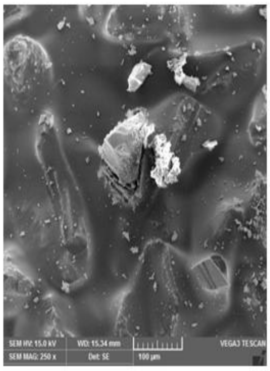


Fig 5a: 0.1wt% Al-G **Fig 5b: 0.2wt% Al-G**



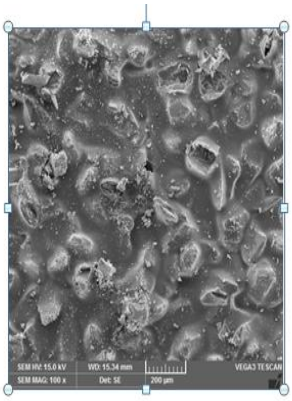


Fig 5c: 0.3wt% Al-G **Fig 5d: 0.4wt% Al-G**

Fig 5: worn emery paper during wear test

Fig 5a - 5dIndicates worn surface topography of emery paper on which different Al-G sample pins were tested at higher speeds i.e 1.5m/sec. The pointed marks (shown in red color) over the worn surface of sample represents damaged portion. The damaged surface shown in Fig 4e seems to be much higher when compared with the damage happened to other samples. The more damage can be attributed to low % of graphene and higher speeds. The self-lubricating property of graphene restricted the higher damage in 0.2, 0.3 and 0.4 wt% of graphene samples. This property was enhanced for the composite by increasing the reinforcement contribution of graphene. In the present study, the damage in the composite specimen of 0.2wt% graphene was reduced when compared with 0.1wt% of graphene. It was further reduced in 0.3wt%graphene samples. Even though damage reduced when compared with 0.1wt% of graphene, a different scenario is observed at 0.4% weight fraction of graphene. In case of 0.4 wt% the inter Spacing between the particle decreases as a result the there may not be an effective load transfer between the reinforcement and the hosting medium. This was explained with the Fig.6.

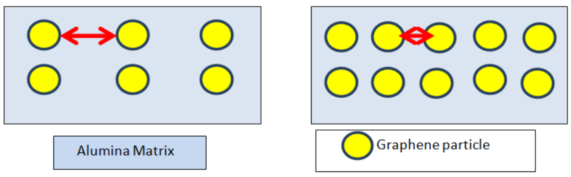
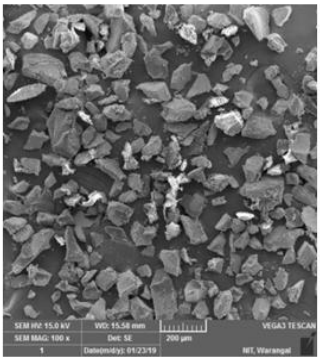


Fig. 6: Interspatial arrangement of alumina and Graphene Matrix in sample

And from SEM images it was clearly evident that 0.3wt% of graphene samples damage is less at different sliding speeds and sliding distance. In case of 0.3wt% of graphene, instead of total damage to the surface, the graphene particles are ploughed from the surface of samples. This action can be attributed to self-lubricating property of graphene. In this case moderate amount of graphene acted as a superficial layer and reduced the further damage. So, wear resistance was improved in 0.3wt% of graphene samples. The worn surfaces of emery sheets contained the following: 1. Particles debonded from the emery paper.2. Abrasive grit particles which became blunt during test 3. Broken abrasive particles of emery paper 4. The debris on the emery sheet. The de-bonded particles, blunt abrasive grit particles and fractured abrasive grit particles generated are observed to be higher at higher speeds only. It is clearly understood that the appearance of these particles mainly depends on the intensity of the speed with which a test was carried out. The particles which are detached from either pin surface or emery sheet was formed as debris. Wear debris was removed from samples after tests at higher speed of 1.5m/sec and were indicated in SEM images in Fig 7a to Fig 7d.



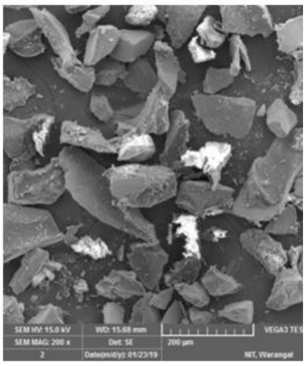
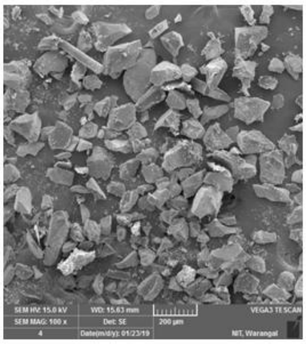


Fig 7a: 0.1wt% Al-G samples Fig 7b: 0.2wt% Al-G samples



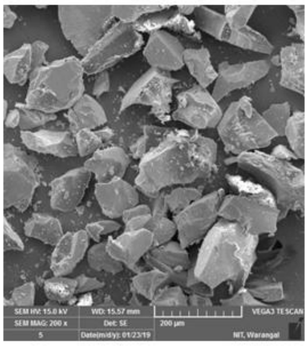


Fig 7c: 0.3wt% Al-G samples Fig 7d: 0.4wt% Al-G samples

Fig 7: Debris Images of samples

III. RESULTS AND DISCUSSIONS:

The rate of wear obtained from the weight loss values of samples after test were depicted in the form of bar graphs. It was observed that from fig 8 at the sliding distance 50m, as speed increased from 0.5m/sec to 1.5m/sec, the wear rate of composite also increased for all samples. But this is increasing wear rate was in decreasing fashion in 0.3wt% Al-G samples, even at higher test speeds.

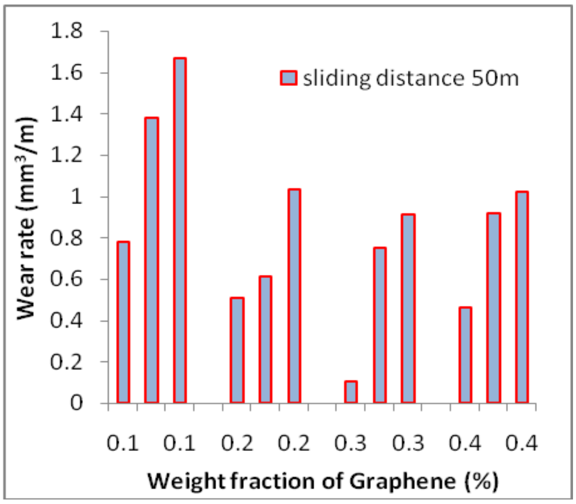


Fig 8.a

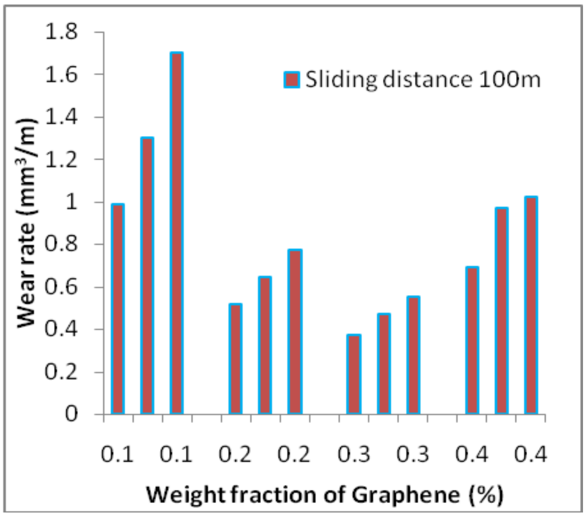


Fig.8.b

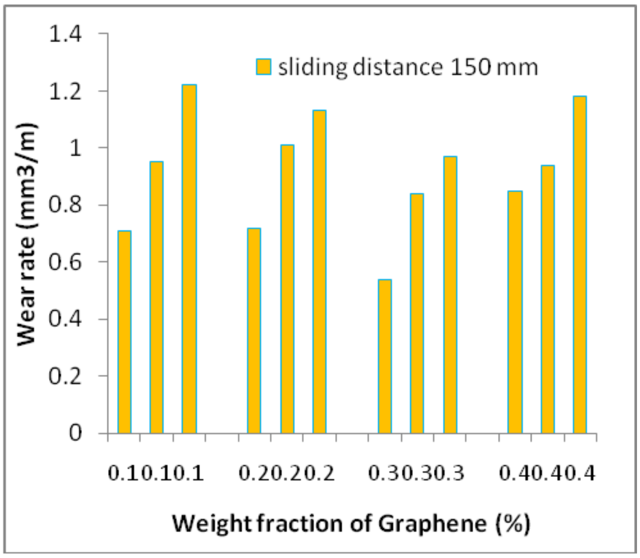


Fig.8.c

Fig 8 : Wear rate Vs Speed of samples at differesliding distance : a – 50m; b -100m; c -150m on 150 μ grit size emery paper.

At the starting of the wear process, the material on the pin sample surface was ploughed plastically by the hard asperities on the opposite surface. With increased speed, the level of penetration of the hard asperities in to pin surface also increased. Due to this ploughing and penetration action material flow, formation of grooves and fragmentation takes place. These fragments of pin material would be accumulated between peaks and valleys of the hard particle opposite surface. With the increase of sliding distance of 50m to 150 m, the time duration of experimentation also increases. The increase in time duration also increases the abrasive action. This increased abrasive action can also be attributed to increased deformation of abrasive grit particles on the opposite surface. The brittleness of asperities could be the main reason for deformation.

IV. CONCLUSIONS

- Composites were prepared using microwave sintering method.
- After examining different sample densities, composites prepared at 1500^{0}c degree centigrade through microwave sintering was identified with highest relative density. The remaining samples are sintered at that temperature only.
- The hardness tests were performed for all the samples.
- Abrasive tests were carried at speed 0.5m/sec - 1.5m/sec with varying sliding distance from 50 to 100mm on 150 µ grit size emery paper at load 10N.
- As the speed increases to 1.5 m/Sec, the improvement in wear rate at 50m sliding distance was 52.97%, 50%, 88.88%, 54.7% for 0.1wt%, 0.2wt%, 0.3wt% and 0.4wt% of Al-G samples respectively. At the 100m sliding distance it was 41.76%, 33.11%, 32.72%, 26.09% for 0.1wt%, 0.2wt%, 0.3wt% and 0.4wt% of Al-G samples respectively. And at 150m sliding distance it was 41.39%, 36.19%, 44.87%, 27.96%for 0.1wt%, 0.2wt%, 0.3wt% and 0.4wt% of Al-G samples respectively
- This shows that 0.3wt% Al-G composite samples are exhibiting enhanced wear resistance properties than other samples. The reinforcement of graphene in alumina is found to be beneficial due to self-lubricating property of graphene. This property of graphene serves as a sacrificial layer during cutting mechanism in abrasive test. And at that the same time microwave sintering also assisted as a tool in improving quality of sintered specimens.

REFERENCES

1. 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
2. Jarosławwoźniak1,Kamilbroniszewski1, Marekkostecki1,Kazimierzczechowski2, Lucynajaworska2, Andrzejolszyna" cutting performance of alumina – grapheneoxide Composites"
3. PiotrKlimczyk*, Lukasz Major, LucynaJaworska, Jerzy Morgiel, SlawomirCygan "Mechanical properties and wear resistance of graphene toughened alumina" International Conference on Materials Science & Technology 2016 Structural, Constructional and Egineering Materials.
4. Tomasz cygan, Jarosławwoźniak, Marekkosteck, Kazimierzczechowski,Norbertgieru, Lucynajaworska ,Andrzejolszyna, Katarina geric "Machinability tests using ceramic tools reinforced by nickel- coated graphene" innovative manufacturing technology 2014
5. HarshitPorwalPeterTatarko Salvatore Grasso ,JibranKhaliq , Ivo Dlouhy´ Mike J. Reece " Graphene reinforced alumina nano-composites" C A R B O N 6 4 (2 0 1 3) 3 5 9 –3 6 9.
6. Katarina GERIC " ceramics tool materials with alumina matrix" may 18th 2010 , 50th anniversary of faculty of technical science NOVISAD, Machine Design , ISSN 1821-1259.
7. Ahmad " Multi layer Graphene (MLG)-Reinforced Al2O3 Nanocomposites Fabricated by High-frequency Induction-heat Sintering Technology" Manufacturing Science and Technology 3(4): 98-105, 2015.
8. 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
9. Tomasz Cygan, Jaroslaw Wozniak, MarekKostecki, AndrzejOlszyna, "Mechanical and tribiological properties of alumina and graphene composites. Proceedings of the World Congress on New Technologies (NewTech 2015) Barcelona, Spain – July 15 - 17, 2015 Paper No. 452
10. X.L. Shi, F.M. Xu, Z.J. Zhang, Y.L. Dong, Y. Tana,⁎, L. Wang, J.M. Yang " Mechanical properties of hot-pressed Al2O3/SiC composites" Materials Science and Engineering A 527 (2010) 4646–4649.
11. Ali Hosseini and Hossam A. Kishawy" Cutting Tool Materials and Tool Wear" http://www.springer.com/978-3-662-43901-2.
12. X. Liu, Y.Y. Fan, J.L. Li, L.J. Wang, W. Jiang, Preparation and mechanical properties of graphenenanosheet reinforced alumina composites, Adv. Eng. Mater.17 (2015) 28–35
13. Apurbba Kr. Sharma , R. Krishnamurthy " Microwave processing of sprayed alumina composite for enhancedperformance" Journal of the European Ceramic Society 22 (2002) 2849–2860
14. Dinesh K Agrawal" Microwave processing of ceramics" Current Opinion in Solid State & Materials Science 1998, 3:480-485.Electronic identifier: 1359-0286-003-00480. C Current Chemistry Ltd ISSN 1359-0286.
15. A. Goldstein, W.D. Kaplan, A. Singurindi": Liquid assisted sintering of SiC powders by MW (2.45 GHz) heating " Journal of the European Ceramic Society .No. pages 6, DTD=4.3.1 Version 7.5.
16. Ammar Ahmed, Elias Siores " Microwave joining of 48% of Alumina-32% Zirconia-20% Silica ceramics" Journal of material processing technology 118(2001)88-95.
17. S. Aravindan, R. Krishnamurthy " Joining of ceramic composites by microwave heating" Materials Letters 38 _1999. 245–249
18. H. Porwal, M. Kasiarova, P. Tatarko, S. Grasso, J. Dusza3 and M.J. Reece " Scratch behaviour of graphene alumina nanocomposites" ISSN: 1743-6753 (Print) 1743-6761 (Online) Journal homepage: http://www.tandfonline.com/loi/yaac20
19. H. Porwal, S. Grassoand M. J. Reece" Review of graphene–ceramic matrix composites" Article (PDF Available) *in* Advances in Applied Ceramics 112(8):443 · May 2013 *with* 5,117 ReadsDOI: 10.1179/174367613X13764308970581
20. R. Benaventea,n , M.D. Salvadora , F.L. Penaranda-Foixb , E. Pallonec , A. Borrella " Mechanical properties and microstructural evolution of alumina–zirconia nanocomposites by microwave sintering " Ceramics International 40 (2014) 11291–11297.
21. 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.
22. Z.B. Yin, J.T. Yuan, Y. Cheng, C. Wang, Z.H. Wang, X.Q. Hu, Microstructure and mechanical properties of Al2O3/Ti(C, N) ceramic tool materials by one-step and two-step microwave sintering, Mater. Sci. Eng. A 670 (2016) 159–165.
23. C. Monaco, F. Prete, C. Leonelli, L. Esposito, A. Tucci, Microstructural study of microwave sintered zirconia for dental applications, Ceram. Int. 41 (2015) 1255–1261.
24. J. Barry, G. Byrne, "Cutting tool wear in the machining of hardened steels. Part I": alumina/tic cutting tool wear, Wear 247 (2001) 139–151.
25. [25] Dinesh Agrawal , Materials World, Vol. 7 no. 11 pp. 672-73 November 1999
26. E.T. Thostenson*, T.-W. Chou "Microwave processing: fundamentals and applications" Composites: Part A 30 (1999) 1055–1071
27. LidijaĆurković, Vera Rede, KrešimirGrilec, AlenMulabdić" hardness and fracture toughness of alumina ceramics" 12.conference of materials process , Friction and wear , MATRIB 07, Vela luka , 21-23 .06.2007
28. Iftikhar Ahmad, BaharehY azdani and Yanqiu Zhu

"Recent Advances on Carbon Nanotubes and Graphene Reinforced Ceramics Nanocomposites" Nanomaterials2015, 5, 90-114; doi:10.3390/nano5010090

AUTHORS PROFILE

K. I. Vishnu Vandana is an Assistant professor at the Department of Mechanical Engineering, Prasad V Potluri Siddhartha Institute of Technology, Vijayawada, AP. She completed her B.Tech in the year 2001 and M.Tech in the year 2003 from Andhra University, Vishakhapatnam. At present she is doing her research in the department of Mechanical Engineering, Andhra university, Vishakhapatnam. She is working on the broad area of characterization and applications of ceramic nano composites. She published 12 papers in different national and international Journals and presented 3 papers in conferences. She has total 15years of teaching experience.

Dr K N S Suman is working as Assistant professor Department of Mechanical Engineering, Andhra University, Vishakhapatnam. He completed his Ph.D. from Andhra University in the year 2006 and **BEST THESIS AWARD** by Andhra University , 2006. At present he is guiding many Ph.D. students and 5 students completed Ph.D. successfully under his guidance