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Hardness and Wear Resistance of ZrO_2 Nano Particle Reinforced Al Nanocomposites Produced by Powder Metallurgy

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Abstract

Nano particle reinforced Aluminium Nanocomposites are produced using Zirconium dioxide (n-ZrO_2). Nano particles produced by solution combustion method. Urea is used as fuel and then reinforcing into Aluminium matrix in different percentages by weight using the powder metallurgy technique. The specimens prepared are tested for their hardness and microstructure. The above mentioned properties were chosen due to the reinforcement added (n-ZrO_2) which is known to have good toughness and hardness among other structural properties at room temperature. The composites are tested for wear resistance by using pin on disc wear testing machine. Study of Wear mechanisms, Microstructure were performed on sintered specimens. Microstructure revealed near uniform distribution of n-ZrO_2 particles with slight agglomeration. The microstructure also revealed good interfacial bond between matrix and n-ZrO_2 particles. Incorporation of n-ZrO_2 particles in aluminium matrix can lead to the production of aluminium composites with improved hardness and wear resistance. These composites can find applications in automotive components like pistons, cylinder liners and connecting rods.

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Keywords:ZrO_2 Nano Particle; Aluminium Nano composite; powder metallurgy; Sliding Wear

1. Introduction

In last two decades, metal matrix Nano-composites has witnessed tremendous growth. Particulate reinforced composites have been extensively employed in the automotive industry for their capability to withstand high temperature and pressure conditions.

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Several manufacturing approaches have been used to fabricate them. Non-homogeneous particle dispersion and poor interface bonding are the main drawbacks of conventional manufacturing techniques. Unique nano order material cannot only be utilized on its own in precision industrial fields but can also provide high performance functionality in conjunction with conventional materials. For this reason, many researchers are investigating the fabrication of nano particle reinforced metal, ceramic, and polymer matrix composite materials [Hansang Kwonet al]. The primary process for producing AMCs on an industrial scale can be either solid state processing or liquid state processing. The liquid state processing especially, stir casting is a promising route for the synthesis of AMCs because of their simplicity and scalability. However, stir casting has inherent problems such as good wetting between the particulate reinforcement and the liquid aluminium alloy melt. Moreover the problem with finer reinforcement particles especially nano particles would be agglomeration. Development of a new method based on stir casting to fabricate nano-Al_2O_3 particulate reinforced aluminum composites in order to avoid agglomeration and segregation of particles found a great success in synthesizing nanoaluminium composites [S. M. Suresh et al].The effect of intermetallic inclusions, Al_2O_3, TiC, SiC, and refractory nanoscale particles served as reinforcing agents for AL25 alloy involving ex situ and in situ reinforcement of matrixes were studied effectively [T. A. Chernyshovaet al]. Investigation of fabrication of Al-Ilmenite ($FeTiO_3$)nano composites by stir casting technique and synthesis of nano particle Ilmenite by high energy ball mill via top down method proved homogeneous microstructure and increased mechanical properties [L. Rasidhar et al].

Metal matrix nanocomposites (MMNCs) with the addition of nano-sized ceramic particles can be of significance for automobile, aerospace and numerous other applications. The physical and mechanical characteristics of the light refractory carbides such as SiC, TiC and B_4C make them suitable for being used as reinforcement in aluminium base metal matrix composites. An attempt has been made to investigation the inexpensive fabrication of bulk lightweight MMNCs with reproducible microstructures and superior properties by use of ultrasonic nonlinear effects, namely transient cavitation and acoustic streaming to achieve uniform dispersion of nano-sized B_4C particles in molten aluminum alloy and found that mechanical properties of the cast MMNCs have been improved significantly even with a low weight fraction of nano-sized B_4C [Govind Nandipatiet al].

The investigation of addition of Al_2O_3 and SiC particulates into the Aluminium matrix showed increased yield strength, ultimate tensile strength & the hardness, & decreased elongation (ductility) of the composites in comparison with those of the matrix. Increasing wt% of Al_2O_3 and SiC increased their strengthening effect and SiC is the most effective strengthening particulate, for higher strength, hardness, & grain size reduction. At the same time it decreases ductility & toughness [John Dixon et al]. Reinforcing aluminium matrix with nano-sized particles is one of the key factors in producing high-performance composites, which yields improved mechanical properties. A uniform distribution of the Al_2O_3 reinforcement phase in the Al matrix can be obtained by high-energy ball milling of Al–Al_2O_3 blends. Nearly 92% increase in the hardness and 57% increase in the tensile strength were obtained in the nano-composites as compared to the commercially pure aluminium. Ultrasonic assisted casting and powder metallurgy methods are becoming more common for the production of Al-Al_2O_3 composites [Dinesh Kumar Koliet al].

The investigation of developing aluminum alloy-nano particles composites (NMMCs) in moulds containing copper chill by reinforcing nano-Zro_2 particulates in aluminum alloy (LM 13) by vortex method was carried out in order to find out their strength, hardness and fracture toughness. Results of the investigation reveal that presence of nano-ZrO_2 particles as dispersed (up to 9Wt. %) and VHC of the chill used has improved significantly the strength, hardness and fracture toughness with slight reduction in ductility. The strength of the composite developed is highly dependent near to chill end and also on the reinforcement content present in the composite. Increase in the chilling rate and increase in the reinforcement content of the material both results in an increase in UTS and fracture toughness of the developed composite. Fractography of the specimens showed that the fracture behavior of matrix alloy has changed from ductile intergranular mode to cleavage mode of fracture. Microstructural analysis of the developed nano-composite reveals the uniform distribution of the reinforcement in the matrix alloy with significant grain refinement [G.Balakumaret al].

In this study, Al-ZrO_2 nanocomposites are fabricated bypowder metallurgy process. The microstructure, hardness and wear resistance of Al-ZrO_2 nanocomposites are characterized.

2. Materials

2.1. Matrix Material

Commercially available alluminium powder of 99% purity is used as matrix material

2.2. Reinforcement material

Nano-ZrO_2 is used as reinforcement material. ZrO_2 at room temperature is considered to be an important structural ceramic because of its excellent mechanical properties, such as fracture toughness, high strength, and hardness. It is also an important material because of its use in different fields of chemistry such as ceramics and as catalyst. Ceramics made with ZrO_2 are known to show a good improvement in strength and toughness.

2.3. Synthesis of n-Zirconia and Characterisation

Combustion synthesis method is used to synthesise n-ZrO_2. 5gms of Zirconyl nitrate along with the calculated amount of the fuel urea is mixed thoroughly with 25ml of distilled water in a Petri dish (having dimensions 100mm x 50mm). This was achieved by using a magnetic stirrer for about 15 min at the end of which a homogenous solution is obtained. The muffle furnace is pre-heated to the temperature of about 500˚C for urea. The crystalline dish containing the aqueous solution is then placed in the muffle furnace for boiling. The completion of boiling yields a viscous liquid which then spontaneously catches fire due to the high exothermic process and converts into a flaky type powder. Fig (1) (a) shows firing of aqueous solution. The dish is left in the furnace until all the fumes (Carbon dioxide, nitrogen and steam) emanating from it has been subsided. The dish containing the flaky powder is then taken out and allowed to cool. Once the dish has cooled down completely, the contents of the dish were transferred to a grinder and the flakes are ground into a fine powder. Fig (1) (b) shows synthesised ZrO_2 powder. This fine powder is weighed using an electronic balance and then transferred into plastic covers. The process described yields just about 1.5 grams each time for the said quantities of the mixture. Thus, it is repeated a number of times to get an adequate amount of n-ZrO_2 required for further tests and studies.

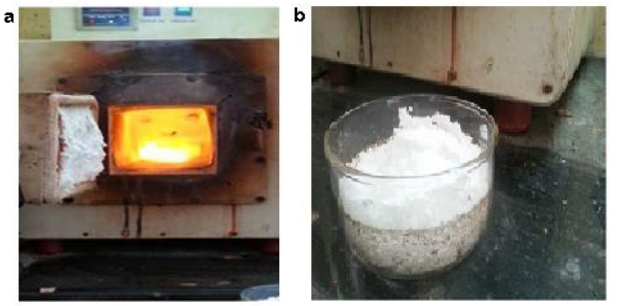

Fig (1) (a) Firing of aqueous solution; (b) n-ZrO_2 Powder

The synthesized n-ZrO_2 Powderis analyzed for phase identification using XRD. Material was finely ground, homogenized and average bulk composition was determined. The procedure for doing the XRD analysis involves specimen preparation, mounting of the specimen slide and activation of the apparatus. The intensity of the diffraction signal is plotted against the diffraction angle 2θ degrees. Data obtained from the XRD Apparatus is then plotted using the Origin software (Origin Pro 8) to generate graph. The graphs obtained were then compared with

the standard XRD patterns taken from ICD (International Centre for Diffraction data).

The obtained XRD pattern of n-ZrO_2 closely conform to the standard XRD pattern where the significant peaks of the standard XRD pattern as seen from the fig (2) occurs at around the following diffraction angles 2θ=30°, 2θ=35° , 2θ=50° and 2θ=60°. Comparing the XRD pattern of fig (2) a; with standard pattern shown in fig (2) b; very few tiny peaks were noticed at some diffraction angles. These tiny peaks which occur at certain angles correspond to the impurities present in the sample powder and hence represent very less impurities. Phases present in the sample can be determined by peak positions and approximate relative intensities. Nano-ZrO_2 prepared using urea at 500 °C exhibited only a single cubic and mono clinic phase. The size of the synthesized n-ZrO_2 was in the nano scale, negligible impurities were found in the sample and phases present were identified clearly. The SEM image of Zirconia is shown in fig (3). The n-ZrO_2 powder obtained is highly porous in nature. The particles have irregular shape, high degree of purity and the particles are flaky in nature.

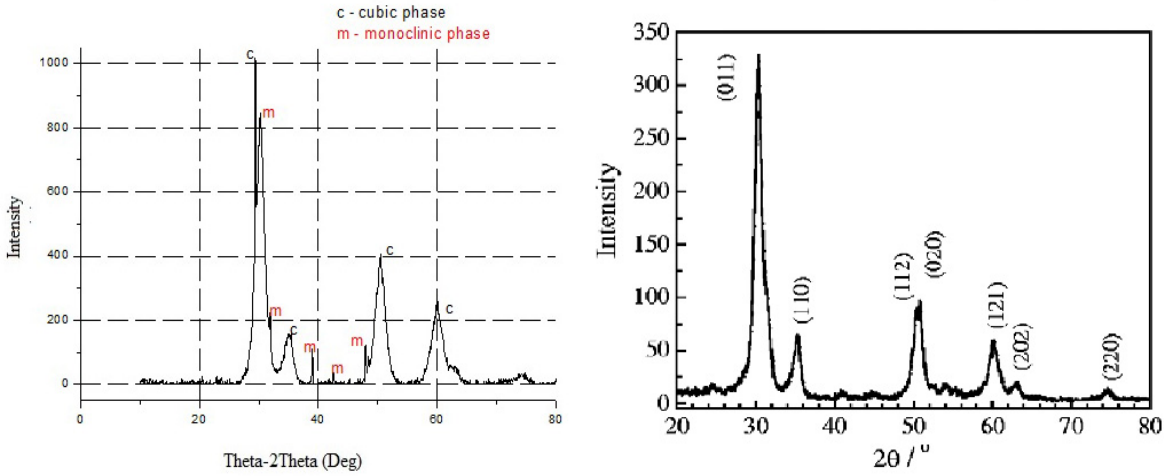

Fig (2) (a) n-ZrO_2synthesised at 500 °C; (b) Standard ZrO_2 XRD pattern

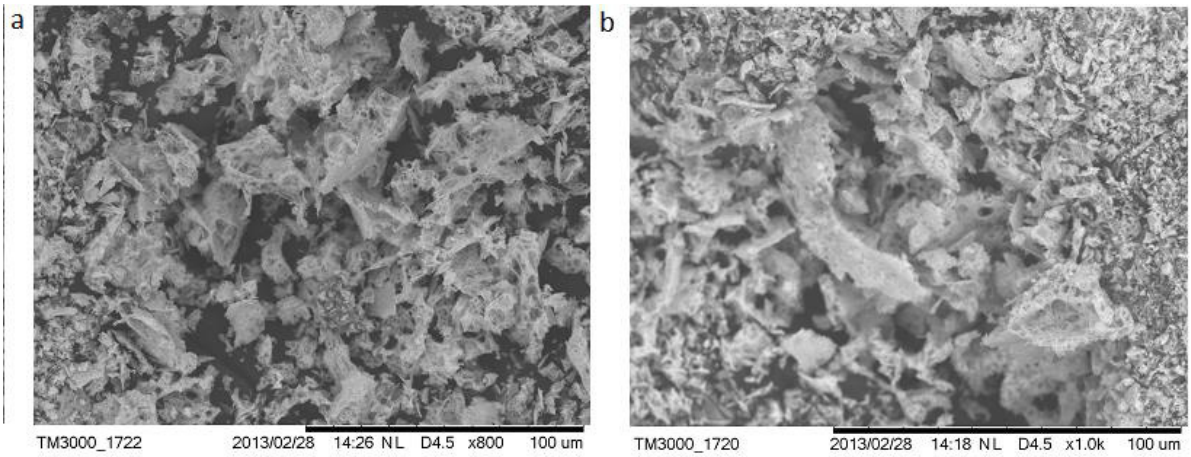

Fig (3) (a) n-ZrO_2at 800X; (b) n- ZrO_2at 1KX

2.3.1. Computation of particle size of n-ZrO2

The particle size is calculated using the Scherer's equation, $L = \frac{0.9\lambda}{\beta cos\theta}$

Where, L= particle size in nm. λ = wavelength of x-ray used in nm. β = width of peak at half peak length of highest peak (FWHM) in radians. θ = half of the angle at which highest peak occurs in degrees. The measurements obtained from the software are shown in fig (4)

Equation	y=y0 + (A/(w*sqrt(PI/2)))*exp(-2*((x-xc)/w)^2)		
Adj. R-Squar	0.98295		
		Value	Standard Erro
tube	y0	82.19664	9.7309
tube	xc	30.26723	0.00155
tube	w	0.27619	0.00318
tube	A	1781.3928	18.68292
tube	sigma	0.1381	
tube	FWHM	0.32519	
tube	Height	5146.1989	

Fig(4) measurements obtained from the software

β = 0.32519°, 2 θ =30.26723° , θ = 15.1336°, λ = 0.154nm

From Scherer equation, crystal size is given by

L = (0.9 * 0.154* 10^-9) / (0.32519 * (π / 180) * cosine (15.1336))

L= 25.28nm

2.4. Fabrication of Al-ZrO_2 Nanocomposite.

The fabrication of Al-ZrO_2 Nanocomposites is by powder metallurgy technique which involves blending-mixing of powders, compaction to produce green composites and sintering.

2.4.1. Blending and mixing of Al and Zirconia (ZrO_2) powders

Blending and mixing is carried out to achieve uniformity of the product manufactured. Lubricants are also mixed with powders to minimize the wear of dies and reduce friction between the surfaces of dies and the particles of powder during compaction. Mixing time depends upon the results desired. For making the test specimen compacts, Aluminium metal matrix was added with the reinforcement at different weight percentages. The specimen made composed of n-ZrO2 in 1 %, 2 %, 3 %and 4 % by weight.

2.4.2. Compaction and sintering

The mixture of Aluminium and n- ZrO_2 is introduced into the die cavity. The plunger in the centre is aligned by trial and error with the axis of the die and the load (75KN) is applied through UTM. The load is applied only when the plunger and the die are effectively engaged. This was taken as a reference load for making the entire test specimen. Once the compaction is complete, the compact is forced out of the die from the end opposite to the end on which the load was applied by application of force by the UTM. The compact is then carefully removed and wrapped in soft sponge sheets and placed in plastic storage units/ containers. Fig(5) exhibit steps involved in preparation of the aluminium matrix-ZrO_2 reinforced composites through powder metallurgy technique.

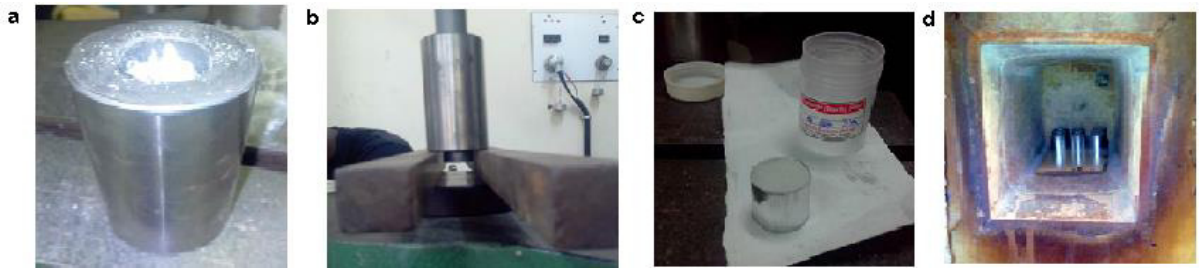

Fig (5 (a) Blended powder in a die; (b) Compaction process; (c) Green compact; (d) Sintering

2.5. Characterization

Microstructure characterization of Al-ZrO_2Nanocompositesis carried out using high-resolution scanning electron microscopy (HRSEM-HITACHI TM3000). Brinnel hardness tests were performed to evaluate the hardness of Al-ZrO_2 Nanocomposites. Pin-on-disk type wear test is performed to evaluate the wear resistance and wear micrographs are taken to analyze wear mechanism under dry sliding condition. The wear tests are carried out at a sliding speed of 600 rpm, at applied load 10N and using a rotating counter face disk made by hi-carbon steel of 64HRC. Wear micrographs are taken using optical microscope of NIKON make with 500X magnification.

3. Results and discussion

3.1. Microstructure

Fig (6) shows SEM images of sintered n-composites with 1 to 4 percent ofn-ZrO_2as reinforcement. From the SEM images, it is observed that there is slight agglomeration of n-ZrO_2particles in the metal matrix. It is very clear that porosity in the compacts has decreased after sintering. This decrease in porosity led to grain growth and good bonding between matrix and reinforced particles.

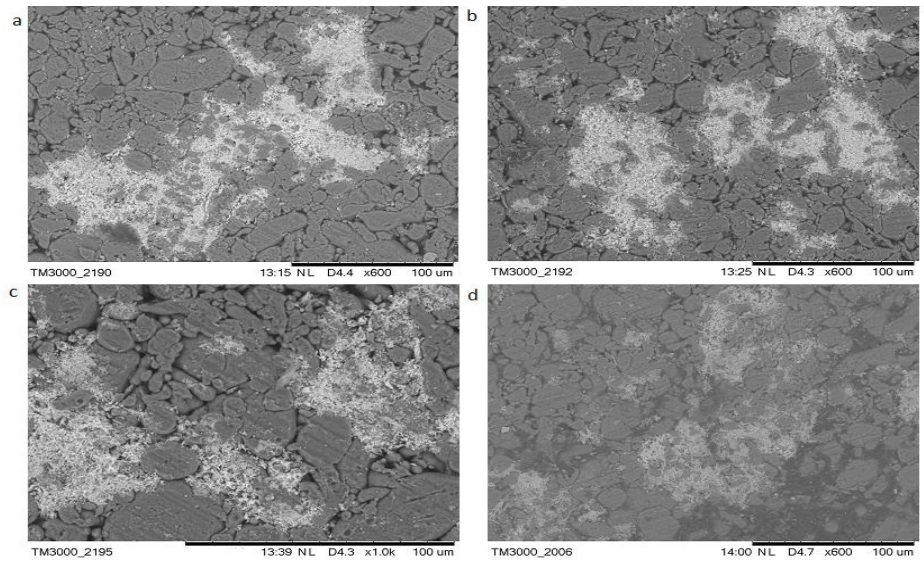

Fig (6) SEM micrographs of (a) 1% ZrO_2 (b) 2% ZrO_2 (c) 3% ZrO_2 (d) 4% ZrO_2reinforced composites

3.2. Hardness

The hardness values of the n-composite specimens are shown in Fig. 7. It is clear that the hardness of the n-composite is higher than that of the unreinforced alluminium processed under identical conditions. Further, hardness increases with increase in the n-ZrO_2 content.

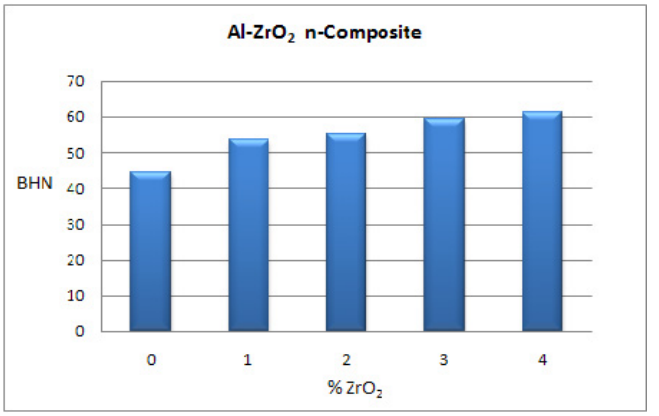

Fig (7) BHN Value of Al-ZrO_2 composites

3.3. Sliding wear

The variation of wear rates of unreinforced alluminium and n-composites are shown in fig (8). From the figure it is evident that the reinforcement n-ZrO_2particles decrease the wear rate of composites compared to unreinforced alluminium. Also the wear resistance increases (decrease in wear rate) with increase in percentage of n-ZrO_2 particles. The improvement in the wear resistance of the composites with increased contents of reinforcement can be attributed to the improvement in the hardness of the composites.

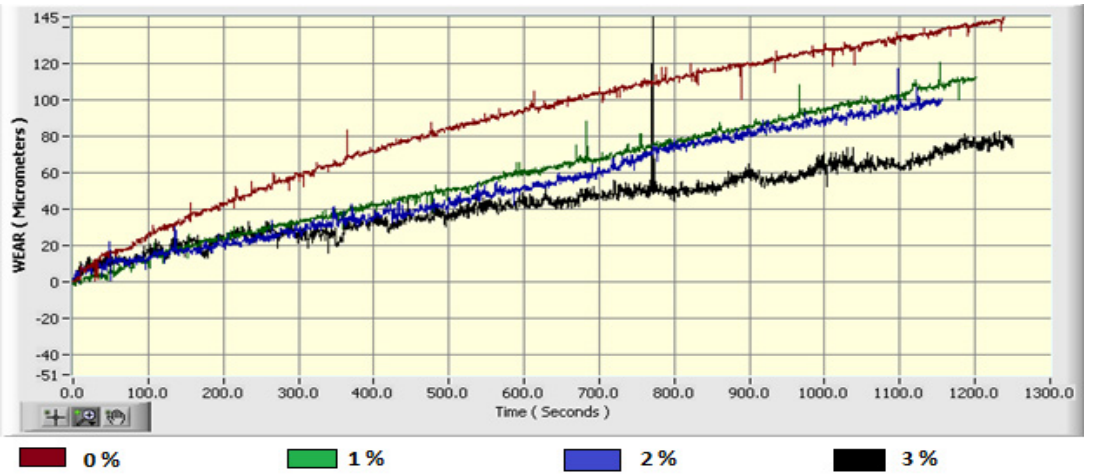

Fig (8) Wear behavior of Al-ZrO_2 reinforced Nanocomposite

3.4. Wear micrographs

Fig (9) shows the wear mechanisms dominant during sliding wear of Al-ZrO_2 reinforced nanocomposites. The microphotographs clearly show oxidation, microcracks/cutting and thermal softening are the principal mechanism of material removal during sliding wear test of n-composites. Fig (9) a reveals black spots indicating oxidation wear mechanism and fig (9) b indicates micro cutting of aluminum by hard particles which acts as third body between counter face and pin. Fig (9) d indicates thermal softening and collection of pin material around circumference of the pin because of rise in temperature during sliding wear.

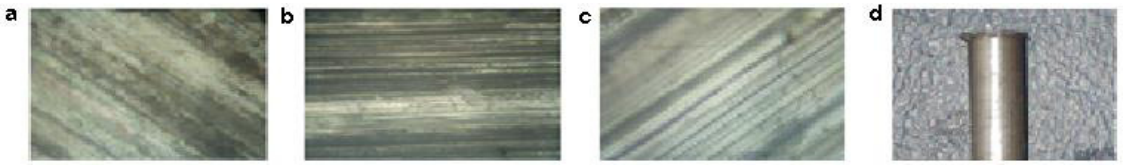

Fig (9) Optical micrographs indicating (a) Oxidation (b) Micro cutting (c) Wear groves (d) thermal softening

4. Conclusions

Hardness of composites increased with increase in n-ZrO_2 content. Wear rates of Nano composites are lower when compared with unreinforced alluminium indicating increase in wear resistance with increase in ZrO_2 content. Oxidation, micro cutting and thermal softening are predominant wear mechanisms during sliding wear conditions. The microstructure of Nano composites revealed near uniform distribution of n-ZrO_2particles and slight agglomeration.

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References

Balakumar .G, “Development and Property Evaluation of Copper- Chilled Aluminum Alloy Reinforced with Nano-ZrO2 Metal Matrix Composites (NMMCS)”, International Journal of Networks and Applications.ISSN 0976-5859Volume 4, Number 1 (2013), pp.1-11.

Chernyshova .T. A, L. I. Kobeleva, P. A. Bykov, L. K. Bolotova, I. E. Kalashnikov, A. T. Volochko, and A. Yu. Izobello,2013 “Nanostructuring of Dispersion_ReinforcedAluminum_Matrix Composite Materials”, ISSN 2075_1133, Inorganic Materials: Applied Research, Vol. 4, No. 3, pp. 247–255.

Dinesh Kumar Koli, GeetaAgnihotri, Rajesh Purohit, “Properties and Characterization of Al-Al_2O_3 Composites Processed by Casting and Powder Metallurgy Routes (Review)”, International Journal of Latest Trends in Engineering and Technology, Vol. 2 Issue 4 July 2013.

GovindNandipati.Dr, Dr.Ravindrakommineni, Dr.Nageswara Rao Damera, Dr.RamanaiahNallu, 2013 “Fabrication and Study of the Mechanical Properties of AA2024 Alloy Reinforced with B_4C Nano-particles using Ultrasonic cavitation method”, IOSR Journal of Mechanical and Civil Engineering, Volume 7, Issue 4, PP 01-07.

Hansang Kwon., Gil-Geun Lee.,Marc Leparoux., Akira Kawasaki., 2013 “Functionally Graded Dual-Nanoparticulate-Reinforced AluminumMatrixCompositeMaterials” , Journal of Physics: Conference Series 419(2013)012004.

John Dixon, SajaGhannam, 2013, “Strengthening of Aluminum Matrix Nano Composite using Al_2O_3.SiC”, European Journal of Applied Engineering.vol10, 2668-3792.

Rasidhar .L, Dr. A. Rama Krishna and Dr. Ch. Srinivas Rao.2013“Fabrication and Investigation on Properties of Ilmenite (FeTiO3) based Al-Nanocomposite by Stir Casting Process”, International Journal of Bio-Science and Bio-Technology Vol. 5, No. 4.

Suresh .S. M, Debadutta Mishra, A. Srinivasan, R. M. Arunachalamand R. Sasikumar, 2011 “Production and characterization of micro and nano Al_2O_3 particle-reinforced LM25 aluminium alloy composites”, ARPN Journal of Engineering and Applied Sciences, VOL. 6, NO. 6.