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WEAR AND HARDNESS INVESTIGATION OF CRYOGENIC TREATED EPOXY REINFORCED WITH HYBRID NANO COMPOSITE

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Epoxy is often used as important material for cryogenic engineering, and can be designed to meet these high standards for cryogenic applications. CNT plays a vital role in reinforcement in polymers. In this experimental study explores the wear and hardness characteristics of epoxy reinforced with nanocomposite Multiwall Carbon nanotubes (MWCNT) and Aluminium Oxide (Al_2O_3)after the cryogenic treatment process. The specimens are prepared by varying the weight percentage of 1, 1.5 and 2% with the help sonication process. The wear properties were examined by pin on disc apparatus in dry sliding condition under different loads of 10, 20 and 30N respectively. The field emission scanning electron microscopy (FESEM) is used to understand the surface morphology of cryogenically treated and untreated samples. Hardness properties were examined by Barcol testing machine as per ASTM Standard. The results revealed that 1.5 % addition of MWCNTs and Al_2O_3 gives better wear properties. Cryogenically treated sample of 1.5 % were shows better improvement of wear resistance and hardness up to 62 % and 45 % comparing untreated samples.

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Keywords: Cryogenic, Multiwall carbon nanotubes, Al_2O_3, Epoxy

1. Introduction

The main focus for developing the polymer nano materials is to strengthen the mechanical and tribological properties of the materials used for engineering applications like piston rings, aero space, medicines, sensors semiconductors, thin films, magnetic, electro chemical and nano-gribbers. This improvement of mechanical properties like wear, hardness, and good chemical resistance will enhance the performance of automotive and aerospace applications [1] A carbon nanotubes (CNTs) are the strongest and stiffest materials that discovered in terms of tensile strength and Young’s modulus. [2] Recently alumina-CNTs have been used as a hybrid component in polymer matrix to develop the performance of multifunctional advanced composite materials. [3] This strength results from the covalent bonds formed between the individual carbon atoms. The multiwall carbon nanotubes are the most energising materials for the development of mechanical properties in various engineering applications. [4] The fibre composites are used to improve the stiffness, strength, and durability and also the Weight reduction is a very useful characteristic of long fiber composites. It is required to break through the composites to develop the mechanical strength for the various applications. The various studies revealed that tensile strength can be improved by addition of carbon nanotubes in engineering materials. Higher tensile strength can be achieved between the individual carbon atoms and sp^2 bonds for sp3 bonds. It has been found that a carbon nanotube (sp^2 bonds) is stronger than the diamond (sp^3 bond). Individual nanotubes can bond together under high pressure. The nature of sp^2 bonds in carbon nanotubes

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possibly will enhance to produce long nanotube wires. It is also found that carbon nanotubes are not only strong; also they have a good elasticity property [5-6]. This composite can give high performance in mechanical properties, thermal and electrical properties [7-15]. By using cryogenic treatment, the mechanical properties and tribological properties can improve significantly [16-20]. The literature statement clearly reveals that carbon tubes have a greater tendency to improve the mechanical properties in various engineering applications. Even though it has a greater improvement, still it has a limited scope of study in present days. It has been studied from the literature survey that carbon nanotubes will have a better performance by the enhancing cryogenic treatment.

2. Experimental methodology

2.1. Experimental materials

The commercially accessible epoxy resin diglycidyl ether of bisphenol is employed with multiwall carbon nanotubes, which is produced by exploitation chemical vapour deposition (CVD) method and functionalized with carboxylic acid to enhance the wall surface. The MWCNT'S had an outer diameter of 60-80 nm, length 5μm, number of walls7-8, and specific surface area of 330 m^2g^{-1} materials supplied from IENT Inc, India. Alumina nano powder is used in this study which has a true density of 2.9 g/cm^3 , mean aggregate size 5μm ,average pore diameter of 110Å, specific surface area lesser than 550 $m^2g^{-1}$and bulk density of 0.20 g/cm^3commercially available in SAI scientific corporation, India.

2.2. Sample preparation

Sonication method is adopted to disseminate in sodium dodecyl sulphate enclosing acetone solution with MWCNT and Aluminium Oxide (Al_2O_3). The Al_2O_3 – MWCNTs is mixed in a weight ratio of 4: 1 and Araldite HY951 was supplemented in the volume ratio of 10:1 as a curing agent. The sonication method is processed for 1 hour at 500W and frequency of 15-145 KHz. The mixed suspension was transported into moulded metal die which was manufactured by using laser cutting machine in the ASTM G99 Standard. The mould kept in an oven at 110^{o}C for 11 hours for drying and also the same process followed for cryogenic treated samples [17] & [22]. The fabricated samples are shown in Fig. 1.

a) b) c)

Fig. 1. Wear Tested Samples of (a) 1 % weight ratio of MWCNT and Al_2O_3, (b) 1.5 % weight ratio of MWCNT and Al_2O_3(c) 2 % weight ratio of MWCNT and Al_2O_3.

2.2. FESEM

Field emission scanning electron microscopy (FESEM) provides topographical and elemental information at various magnifications, with virtually unlimited depth of field. The FESEM image suggests cryogenic treatment of multi walled carbon nanotubes has more intensities than the untreated multi walled carbon nanotubes. The Fig. 2 shows untreated pure epoxy.

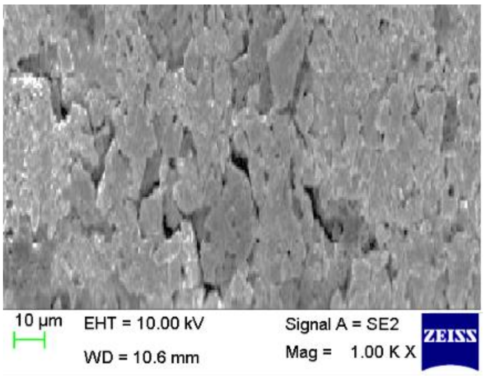


Fig.2. SEM Image of untreated pure epoxy.

This experiment carried out with Zeiss sigma SEM with Scotty Field Emission (FE) source and GEMINI electron optical column, which has working distance of 6.8m, current of 80µA along with aperture size of 30µm and the image obtained on an on-screen polaroid display at various magnifications with an in-lens detector at 10kV. The fracture surface in morphology of cryogenically treated of pure epoxy and combined samples of MWCNT and Al_2O_3 is observed using field emission scanning electron microscope. The Fig. 4 shows that there is a large smooth surface was achieved when compare to the Fig. 3 cryogenically treated pure epoxy. The Fig. 4a and b depicts that the rate of dispersion of carbon nanotubes and alumina nanocomposites is optimal. The Fig. 4b exhibits that the MWCNTs interlocked with alumina particle are dispersed in host matrix evenly.

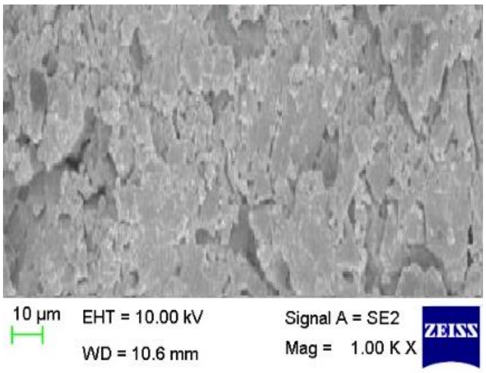
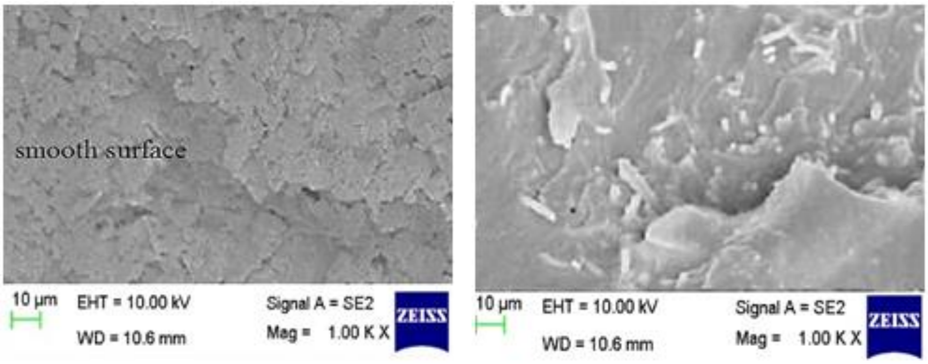


Fig. 3. SEM image of cryogenically treated pure epoxy.



(a) *(b)*

Fig. 4. a) Untreated 1.5 weight % MWCNT and Al_2O_3, b) Cryogenic treated 1.5 Weight % MWCNT and Al_2O_3.

3. Wear test

The wear test was conducted on pin on disc type apparatus was used to observe the wear and friction behavior of pure epoxy and multiwall carbon nanotubes/alumina / epoxy nanocomposites. The sample specimens were prepared as per the ASTM standard . The tests were conducted within the temperature for all the samples while not ever-changing the operational conditions. The sliding load of 10N, 20N and 30N was applied to judge the wear and friction behavior of pure epoxy and projected nanocomposites. The machine was connected with the hardware unit to initialize and set the parameter of disc speed, time and load. The wear testing machine are shown in Fig. 5.

Fig. 5. Wear Testing Machine.

4. Result and discussion

The Fig. 6 potrays the comparison of wear losses for non-cryogenic treated and cryogenic treated samples with the application of 1 kg load. The samples were prepared with pure epoxy, 1, 1.5 and 2 % of MWCNT and Al_2O_3. The untreated nanocomposites results for 1, 1.5 and 2.0 wt. % of MWCNTs were compared with cryogenically treated nanocomposites about 1, 1.5 and 2.0 wt. % of MWCNTs and Al_2O_3. From the comparison analysis, it has been found that 1.5 wt. % performs the better wear resistance than 1 and 2.0 wt.%. Also it has been observed that the wear resistance increased further when there is an absence of MWCNTs and Al_2O_3 i.e pure epoxy. The comparison between the treated and non-treated pure epoxy combination samples revealed that cryogenically treated pure epoxy gives the better wear resistance.

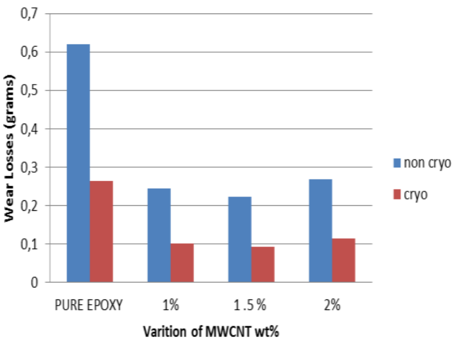


Fig.6.Losses of cryogenic and non cryogenic treated samples at 10N.

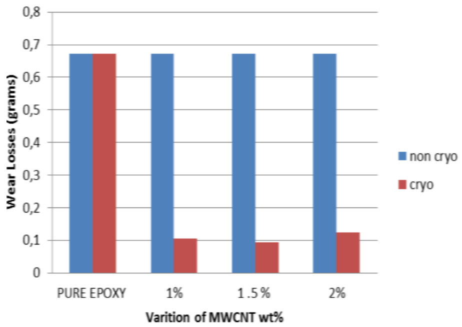


Fig. 7. Wear losses of cryogenic and non cryogenic treated samples at 20N.

From the overall analysis, it has been strongly observed that 1.5 wt.% of MWCNTs and Al_2O_3 performs better wear resistance than pure epoxy, 1 and 2.0 wt.% of MWCNTs and Al_2O_3. Cryogenically treated samples (MWCNTs and Al_2O_3)of varying percentage about 1, 1.5 and 2 wt.% are improved 61.5, 65 and 56 % respectively compare to untreated samples. The Figure 7 portrays the comparison of wear losses for non-cryogenic treated and cryogenic treated samples with the application of 2 kg load. The samples were prepared with pure epoxy, 1, 1.5 and 2 % of MWCNT and Al_2O_3. The untreated nanocomposites results in 1, 1.5 and 2.0 wt. % of MWCNTs were compared with cryogenically treated nanocomposites about 1, 1.5 and 2.0 wt. % of MWCNTs and Al_2O_3, from the comparison analysis, it has been found that 1.5 wt. % performs the better wear resistance than 1 and 2.0 wt.%,also it has been observed that the wear resistance increased further when there is an absence of MWCNTs and Al_2O_3 i.e pure epoxy. The comparison between the treated and non-treated pure epoxy combination samples revealed that cryogenically treated pure epoxy gives the better wear resistance. From the overall analysis, it has been strongly observed that 1.5 wt.% of MWCNTs and Al_2O_3 performs better wear resistance than pure epoxy, 1 and 2.0 wt.% of MWCNTs and Al_2O_3. Cryogenically treated samples (MWCNTs and Al_2O_3) of varying percentage about 1, 1.5 and 2 wt.% were achieved 84, 86.1 and 81 % respectively. The Figure 8 portrays the comparison of wear losses for non-cryogenic treated and cryogenic treated samples with the application of 3 kg load. The samples were prepared with pure epoxy, 1, 1.5 and 2 % of MWCNT and Al_2O_3. The untreated nanocomposites results for 1, 1.5 and 2.0 wt. % of MWCNTs were compared with cryogenically treated nanocomposites about 1, 1.5 and 2.0 wt. % of MWCNTs and Al_2O_3, from the comparison analysis, it has been found that 1.5 wt. % performs the better wear resistance than 1 and 2.0 wt. %, also it has been observed that the wear resistance increased further when there is an absence of MWCNTs and Al_2O_3 i.e pure epoxy. The comparison between the treated and non-treated pure epoxy combination samples revealed that cryogenically treated pure epoxy gives the better wear resistance. From the overall analysis, it has been strongly observed that 1.5 wt. % of MWCNTs and Al_2O_3 performs better wear resistance than pure epoxy, 1 and 2.0 wt. % of MWCNTs and Al_2O_3. Cryogenically treated samples (MWCNTs and Al_2O_3) of varying percentage about 1, 1.5 and 2 wt.% are improved 62.2, 64.1 and 59.6 % respectively compare to untreated samples.

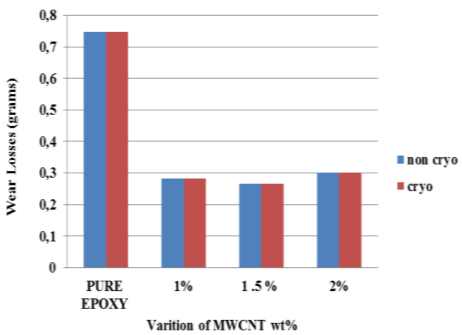


Fig. 8. Losses of cryogenic and non cryogenic treated samples at 30N.

4.1. Comparison of wear losses of 1.5 % cryogenically treated sample at 10, 20 and 30N

The Fig.9 depicts the comparison of wear losses at various loads about 1, 2 and 3 kg for cryogenic treated samples. Cryogenically treated sample (MWCNTs and Al_2O_3) of percentage about 1.5 wt.% will reduce the wear losses for various loads. It is observed from the Figure that improvent is attained in wear losses at 1 kg load following to 2 kg and 3 kg load respectively. Thus the analysis concludes that 1 kg load gives the better wear resistance results compared to 2 and 3 kg loads.

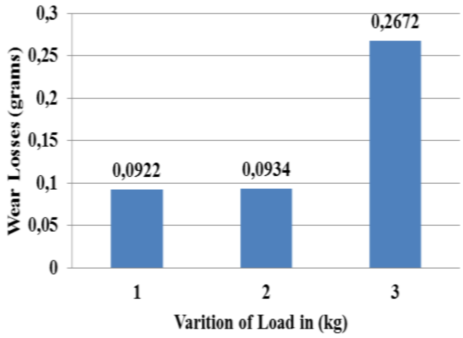


Fig. 9. Wear losses of cryogenically treated 1.5wt% sample at various loads.

4.2. Barcol Hardness Test

The hardness values are recorded for the fabricated samples using barcol Hardness testing machine for the ASTM D2583 samples. The barcol hardness test characterizes the indentation hardness of materials through the depth of penetration of an indenter, loaded on a material sample and compared to the penetration in a reference material. The method is most often used for composite materials such as reinforced thermosetting resins or to determine how much a resin or plastic has cured. The test complements the measurement of glass transition temperature, as an indirect measure of the degree of cure of a composite. It is inexpensive and quick and provides information on the cure throughout a part. The indentor contains a hardened steel frustum having an angle of 260 with a flat tip of 0.157 mm (0.0062 in) in diameter. It shall fit into a hollow spindle and be held down by a spring –loaded plunger. The depth of the penetration is converted into absolute barcol numbers shown in Table1.For untreated nanocomposites the tests result was taken for 1, 1.5, 2.0 wt.% of MWCNTs-Al_2O_3, where 1.5wt.% MWCNTs- Al_2O_3 samples shows the better hardness than other samples. The tabulated results of cryogenically treated samples indicate better hardness number of 26.6 better than other samples. This shows cryogenic treatment of nanocomposites has overall better performance than other remaining samples. The hardness was better for 1.5wt% MWCNTs of cryo treated samples than other samples.

Table 1. Barcol Hardness Test Results.

Samples	Wt %	Barcol Hardness					Avg
		1	2	3	4	5	
After cryogenic treatment	1	21	20	23	20	22	21.2
	1.5	24	26	27	25	26	26.6
	2	21	17	20	19	18	19
Before cryogenic treatment	1	17	17	19	19	18	18
	1.5	16	17	16	17	16	16.4
	2	10	11	12	11	12	11.2

5. Conclusions

In this exploration work wear and hardness, disintegration and mechanical conduct of epoxy/MWCNTs and cryogenically treated epoxy/MWCNTs with different weight rates were estimated. The ragged and disintegrated surface morphologies were examined for the component of material evacuation process. The numerical recreation is additionally done for the expectation of wear with the test comes about. As per the outcomes the accompanying conclusions were drawn.

Tribological conduct is enormously enhanced in epoxy/MWCNTs nanocomposites with the impact of homogeneous scattering of MWCNTs in epoxy network and interracial quality of nanocomposites.From the result, it has been strongly observed that 1.5 wt. % of MWCNTs and Al_2O_3 performs better wear resistance than pure epoxy, 1 and 2.0 wt. % of MWCNTs and Al_2O_3.

Cryogenically treated samples (MWCNTs and Al_2O_3) of varying percentage about 1, 1.5 and 2 wt.% are improved 61.5, 65 and 56 % respectively compare to untreated samples fo1kg load. Cryogenically treated samples (MWCNTs and Al_2O_3) of varying percentage about 1, 1.5 and 2 wt.% are improved 84, 86.1 and 81 % respectively compare to untreated samples for 2 kg load.
Cryogenically treated samples (MWCNTs and Al_2O_3) of varying percentage about 1, 1.5 and 2 wt.% are improved 62.2, 64.1 and 59.6 % respectively compare to untreated samples for 3 kg load.

The bar graphs reveal that 1 kg load gives the better wear resistance results compared to 2 and 3 kg loads.The cryogenically treated Nano composites shows 26.6 better results and 1.5 weight percentages of MWCNTs is better than other samples.

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