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Contents

Volume 12
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Research Articles

Preface

Ilze Aulika, Katarzyna Bejtka, Irena Drevenšek-Olenik..... 1

Large-scale Synthesis of WS₂ Multiwall Nanotubes and their Dispersion, an Update

Alla Zak, Liat Sallacan Ecker, Roni Efrati, Larissa Drangai, Niles Fleischer and Reshef Tenne..... 1

Distributed Array of Polymeric Piezo-nanowires through Hard-Templating Method into Porous Alumina

Valentina Cauda, Davide Dapra, Ilze Aulika, Angelica Chiodoni, Danilo Demarchi, Pierluigi Civera, Marco Pizzi..... 11

Effect of Mo₆S_xI_{10-x} Nanotubes Addition on Electrooptical Properties of Polymer-dispersed Liquid Crystals

Jerneja Milavec, Aleš Mrzel, Irena Drevenšek-Olenik, Mykhailo Pevnyi, Victor Reshetnyak..... 18

Charge Transfer Properties of Surface-treated WS₂ Nanotubes and Fullerene-like Nanoparticles

Tiziana Di Luccio, Carmela Borriello, Sumeet Kumar, Giuseppe Nenna..... 26

Nanomaterials Characterization Using Nuclear Methods at IFIN-HH

Ion Burducea, Liviu Stefan Craciun, Cristina Ionescu, Mihai Straticiu, Alin Titus Serban, Petru Mihai Racolta..... 33

Transient Grating Experiments on Inorganic–elastomer Nanocomposites

Andrea Taschin, Paolo Bartolini, Antoni Sánchez-Ferrer, Raffaele Mezzenga, Aleš Mrzel and Renato Torre..... 46

The Mechanical and Tribological Properties of Epoxy Nanocomposites with WS₂ Nanotubes

Elad Zohar, Sharon Baruch, Mark Shneider, Hanna Dodiuk, Samuel Kenig, H. Daniel Wagner, Alla Zak, Alex Moshkovith, Lev Rapoport, Reshef Tenne..... 53

Ultrasonic Characterization of Dynamic Elastic Properties of Polymer Composites with Inorganic Nanotubes

V. Samulionis, J. Banys, A. Sanches-Ferrer and R. Mezzenga..... 66

Dynamic- and Thermo- mechanical Analysis of Inorganic Nanotubes/elastomer Composites

Armin Fuith, Marius Reinecker, Antoni Sánchez-Ferrer, Raffaele Mezzenga, Aleš Mrzel, Maris Knite, Ilze Aulika, Marija Duncic and Wilfried Schranz..... 71

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The Mechanical and Tribological Properties of Epoxy Nanocomposites with WS₂ Nanotubes

^{1,2} Elad ZOHAR, ¹ Sharon BARUCH, ¹ Mark SHNEIDER, ¹ Hanna DODIUK,
¹ Samuel KENIG, ² H. Daniel WAGNER, ³ Alla ZAK, ⁴ Alex MOSHKOVITH,
⁴ Lev RAPOPORT, ² Reshef TENNE

¹ Shenkar College of Engineering & Design, Ramat Gan 52526, Israel

² Department of Materials and Interfaces, Weizmann Institute, Rehovot, 76100, Israel
Tel.: +972-8-9342394, fax: +972-8-9344138

³ “NanoMaterials Ltd.”, 18 Einstein St., Weizmann Science Park,
P.O. Box 4088, Nes Ziona 74140, Israel
Tel.: 972-8-9302671, fax: 972-99302675

⁴ Department of Science, Holon Institute of Technology,
P.O. Box 305, 52 Golomb St., Holon 58102, Israel
E-mail: Reshef.Tenne@weizmann.ac.il, alla@apnano.com

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Abstract: It is now possible to synthesize inorganic nanotubes (INT) of WS₂ in a pure phase and substantial amounts. Due to their crystalline perfection, they are characterized by excellent mechanical behavior. This study is dedicated to the investigation of the effect of INT-WS₂ on the mechanical, thermal, adhesion and tribological properties of epoxy based nanocomposites. Various concentrations up to 1.0 wt% of the INT were added to the epoxy matrix.

First a method was developed to mix the nanotubes in the epoxy resin matrix. A combination of both magnetic stirring and ultrasonic mixing was used. The adhesion, fracture toughness and strain energy release rate were studied. The INT-WS₂ were found to significantly improve all these properties. The wear of the nanotubes-reinforced epoxy was eight-times lower than that of pure epoxy. These results suggest numerous applications. *Copyright © 2011 IFSA.*

Keyword: Inorganic nanotubes, WS₂ nanotubes, Epoxy nanocomposites, Mechanical properties of nanocomposites, Wear of nanocomposites.

1. Introduction

Due to their superior properties composite materials are used extensively in the automotive, aerospace, sport and defense industries as well as in home appliances, consumer products and numerous other applications. The enhanced mechanical behavior is achieved by choosing the right components for composite and the ratio between them. Epoxy resin systems are increasingly used as matrices in composite materials. They serve as casting resins, adhesives, and as high performance coatings for tribological applications, such as sliding bearings, anti-drug paintings and calendar roller covers. The profile of the mechanical properties of epoxy matrices can be influenced by modifying the molecular architecture and structure. For example, the density of cross-linking could be increasing to generate higher stiffness and strength, at the cost of undesirable brittleness, which is attributed to the constrained plastic deformation.

Toughening agents like rubber particles [1-3], glass beads [4-6], microvoids [7], thermoplastic [8, 9] and hyperbranched polymers [10, 11], and more, often show adverse effects on composites. Thus, such additives often induce a decrease in elastic modulus, strength, strain, and thermal properties [12]. The reinforcement of epoxy resins by nanoparticles such as carbon nanotubes [13, 14], nano-platelets and nanospheres [15-19], as well as block-copolymer nanoparticles [20-24] could enable toughening of epoxy matrices without compromising other mechanical properties. This is achieved to some extent by avoiding perturbations caused by micro level fillers, such as local stress concentrations and microcracking [4, 18]. A known drawback of nanoparticles is their tendency to agglomerate. Moreover, their high cost, complex processing, technical difficulties in up-scaling, and sometimes poor reproducibility, impede the large scale commercialization of high-performance nanofillers. The discovery of inorganic fullerene-like (IF) nanoparticles and nanotubes (INT) of WS₂ [25] opened a new field in solid state inorganic chemistry. In the ensuing years their growth mechanism from oxide nanoparticles was elucidated, leading to IF and INT pure phase production [26]. Inorganic fullerene-like nanoparticles were found to exhibit superior mechanical properties, especially regarding tribology and high wear resistance under high loads [27]. Several reports alluded to the enhanced mechanical behavior of IF-WS₂ and INT-WS₂ based nanocomposites with both thermoplastics and thermosetting polymers [28, 29]. Scale up of the synthesis of WS₂ nanotubes has been recently described [30]. In particular, the latest synthetic route [30b] which provides 50-100 g INT-WS₂ per batch in high purity and yields (95 %) and facile dispersion of the nanotubes opens numerous opportunities. This development allows studying their effect on reinforcing polymer nanocomposites, and epoxy matrices in particular, which is described in the present work.

2. Experimental Methods

2.1. Materials

In the current investigation, epoxy resin was mixed with WS₂ nanotubes. For most of the results reported here, the resin is based on di-glycidyl ether of bis-phenol-A (DGEBA) (D.E.R. 331 product of Dow Chemical, Midland, MI, USA). The tribological studies were carried out with another epoxy resin, i.e. Epon 828. The curing agent which was used here was polyamido-amine (Versamide 140 product of Miller Stephanson, Sylmar, CA, USA) with theoretical equivalent weight of 97. The mixing ratio was 70:30 w/w. Multiwall WS₂ nanotubes (INT-WS₂) with diameter of 30-150 nm and length of 1-20 μm (Fig. 1) were produced by "NanoMaterials"[30]. Note however, that the nanotubes used in this study are based on the synthesis described in Ref. 30a, which required a heavy de-agglomeration procedure, which could hamper their mechanical properties. The nanocomposite containing nanotubes from the more recent route described in Ref. 30b were tested in tribological measurements only.

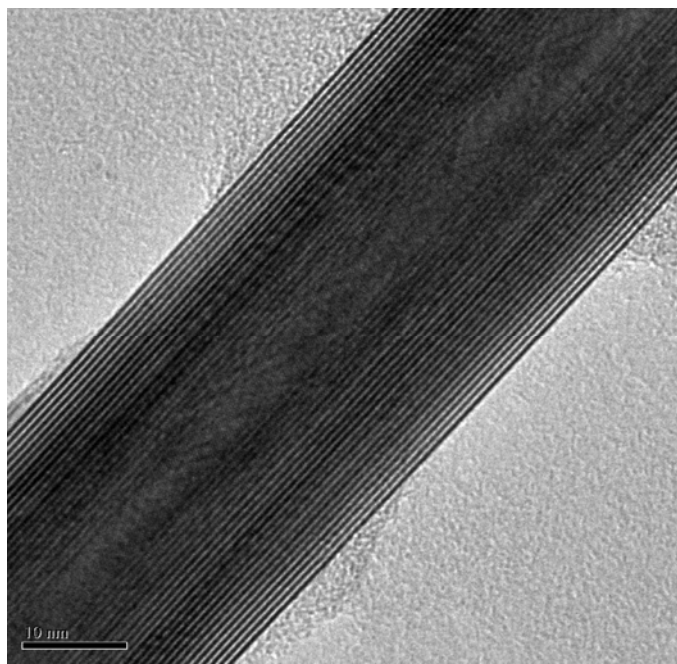


Fig. 1. TEM images of INT-WS₂.

2.2. Dispersion and Mixing Process

In contrast with the more recent procedure [30b], the early synthesis of bulk nanotubes described in Ref. [30a], produced highly agglomerated INT-WS₂. Therefore, a two step mixing process was adopted for the preparation of the epoxy nanocomposite. The agglomerates were first mechanically mixed in the (liquid) epoxy resin, before the curing agent was added. For that purpose a magnetic stirrer was applied for 2 hours. In the second step, dispersive mixing was applied by a 130 W ultrasonic probe mixer (Sonics, Vibra Cell, VC-130) to break the agglomerates of the nanoparticles to a sub-micron level and disperse them in the epoxy resin. The ultrasonic dispersion must be applied carefully, since it may damage the epoxy resin by promoting chain scission and radical formation [31]. Furthermore, it was shown that carbon nanotubes can be easily broken into shorter fragments by excessive ultrasonic treatment [32, 33]. To avoid damaging the nanotubes and disperse the epoxy/nanoparticle mixture effectively and uniformly, the ultrasonic mixer probe was used in 5 different locations of the vessel for duration of 3 minutes in each location. The sonication process produced high mixing shear rates which break the agglomerates but also leads to rising temperatures and, therefore, to a decrease in the viscosity of the fluid. The vessel was cooled in ice water to reduce heat and retain the viscosity of the mixture. The epoxy/nanotube mixture and the curing agent were separately degassed in a vacuum desiccator for 30 min. After degassing, the curing agent was introduced into the epoxy/nanotube mixture and manually mixed for 2 min, and the mixture was degassed again for 15 min.

2.3. Shear and Peel Testing

Shear and peel tests were carried out to test the adhesion properties of selected formulations. The adherent substrates used for the specimens were Aluminum 6061-T651 (shear test) and Aluminum 1100-O alloy (peel test), respectively (Chongqing Meifan Metal Material Co. Ltd, Shiqiaopu District, Chongqing, China). The dimensions of the specimens were 25 × 305 × 1 mm for the peel test and 25.4 × 88.9 × 1.6 mm³ for the shear test. The yield strength and Brinell hardness of aluminum (Al)

6061 are 267 MPa and 95 AA, respectively, which makes it suitable for the shear test. Al 1100-O is softer and more flexible, with yield strength of 34 MPa and Brinell hardness of 23 AA, making it appropriate for peel tests.

Both adherents were mechanically polished to increase their surface roughness and improve the adhesion of the epoxy to the metal. After polishing, the specimens were chemically treated with N-aminoethyl-3-aminopropyltrimethoxysilane (Dynasylan® DAMO, Degussa, Parsippany, NJ, USA) by rinsing in a solution of isopropanol/water/silane in the weight ratio of 98:1:1 for 15 min. The treated specimens were then inserted into a heated oven (100 °C) for 1 hr to accelerate the evaporation of the solvents. The epoxy/nanotube mixture was then spread on the aluminum surface and 0.1 mm layers were produced under pressure. The specimens were cured at 80 °C for 16 hr. Single lap shear (SLS) joints were prepared with Al 6061 and tested according to ASTM D-1002 at a loading speed of 1.3 mm/min and adherent overlap of 25.4 mm. T-peel joints were prepared with Al 1100-O and tested according to ASTM D-1876 at a loading speed of 254 mm/min. The specimens were tested in a universal testing machine (Instron 4201, Grove City, PA, USA).

2.4. Fracture Toughness Testing

The nanocomposite was characterized by standard test methods for plane-strain fracture toughness. The critical-stress-intensity factor, K_{IC} and the energy per unit area of crack surface or critical strain energy release rate, G_{IC} , at fracture initiation (ASTM D-5045), were determined using a miniature materials tester (MiniMat2000, Rheometric Scientific Inc., NJ, USA). The testing geometry of the specimens was the compact tension (CT), previously described in [34]. Typically, a given amount of epoxy with nanoparticles and curing agent (after mixing) was poured into a silicone (RTV) mold, then degassed for 15 min and cured into an oven for 16 hr at 80 °C. After curing, the specimens were carefully shaped to standard dimensions using a carving machine. Vacuum degassing caused air/gas bubbles to surface and these were removed by carving/polishing.

2.5. Thermal Analysis

Dynamic mechanical thermal analysis (DMTA) was carried out on a DMA-7e (Perkin Elmer) apparatus according to ASTM-D 5023 in three point bending mode. The analysis was carried out at a frequency of 1.0 Hz and within a temperature range of 0 to 120 °C, at a heating rate of 2 °C/min. Sample bars of dimensions 1 × 5 × 20 mm were used for these tests.

2.6. Scanning Electron Microscopy (SEM) Observations

The fractured surfaces were examined by SEM (Zeiss LEO Supra 55VP and Zeiss ULTRA 55, Oberkochen, Germany). A 9 nm thick chromium coating was applied on the surface to prevent electrical charging of the insulating specimens. The accelerating voltage of the electron beam was between 5-15 kV.

2.7. Tribological Measurements

Dry (oil-free) tribological measurements were carried out using a pin on disk set-up. The velocity used was 0.35 m/sec; the load was 20 N (0.25 MPa) in this series.

3. Results and Discussion

3.1. Dispersion

The dispersion state of the nanoparticles in the matrix plays a significant role in determining the nanocomposites properties. Fig. 2 presents a histogram of the WS₂ nanotubes agglomerate sizes (cross section area) at different sonication times. The histogram of the nanotubes agglomerate sizes at different sonication times demonstrates that short sonication times (2 min) leaves a large distribution of (nanotubes) agglomerates, while longer sonication times lead to breakdown of the agglomerates and fracture of the nanotubes. Therefore the sonication was applied in 5 intervals of 3 minutes each, at 5 different positions in the vessel.

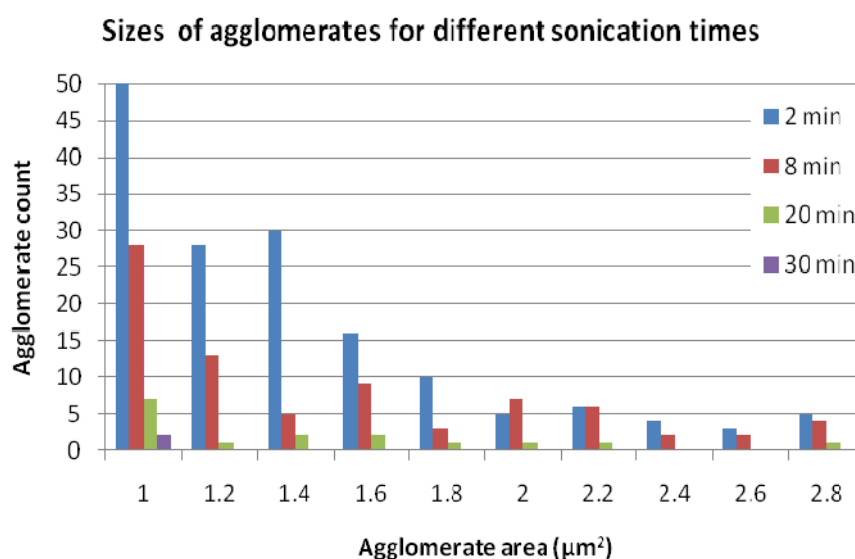


Fig. 2. Histogram of WS₂ nanotubes agglomerate sizes (cross section area in μm²) for different sonication times (2, 8, 20, 30 min). The sample area is 1043×834 μm².

Note that if relatively large agglomerates remain in the matrix, larger areas of stress concentration usually form in the sample, thus promoting premature failure. This effect becomes more predominant as the nanoparticles content increases, since the likelihood of agglomeration in this case increases. Ideally, when perfect dispersion is achieved, each nanoparticle is discrete and fully interacts with the epoxy matrix (full exfoliation) [13, 14, 18].

3.2. Mechanical Tests – Peel, Shear and Fracture Toughness

In the present study the peel force, the shear stress and also the resistance to crack propagation (fracture toughness) were studied. Complementary series of thermo-mechanical tests (DMTA) were also performed. Cohesive failure permits the characterization of the mechanical behavior of the adhesive itself, rather than of the adhesive-adherent interaction. SEM inspection of the tested specimens surface indicates a partially cohesive failure of the peel and shear specimens.

Table 1 and Figures 3-7 summarize the mechanical test results. The effect of the INT-WS₂ on the epoxy matrix is examined with respect to the un-toughened (neat) epoxy. As seen, increasing the concentration of the nanotubes leads to an increase in the peel strength, with a maximum at 0.5 wt%

where an 85% increase relative to pure epoxy is observed. At a nanotube concentration of 1.0 wt% the increase is only about 40%, likely due to increased nanotube agglomeration. Similarly, the INT-WS₂ have a positive effect on the shear strength and shear strain, reaching a maximum also at 0.5 wt% (39% and 87% improvement, accordingly). Beyond this value the positive effect of the nanotubes levels off. In discussing the properties of a brittle composite, the shear strain is of great importance, which is mostly pertinent to the behavior of adhesives.

Table 1. Mechanical properties of INT-WS₂ toughened epoxy.

Concentration [wt%]	Shear strain [%]	Shear strength [MPa]	Peel strength [N/mm]	K _{IC} [MPa*m ^{0.5}]
0.0	1.67 ± 0.20	5.28 ± 0.84	0.487 ± 0.025	0.84 ± 0.07
0.1	2.42 ± 0.48	6.82 ± 0.71	0.447 ± 0.073	1.02 ± 0.11
0.3	2.30 ± 0.32	6.29 ± 0.39	0.469 ± 0.047	0.93 ± 0.18
0.5	3.13 ± 0.12	7.36 ± 0.01	0.906 ± 0.131	1.25 ± 0.17
1.0	2.48 ± 0.17	7.28 ± 0.33	0.718 ± 0.103	1.05 ± 0.11

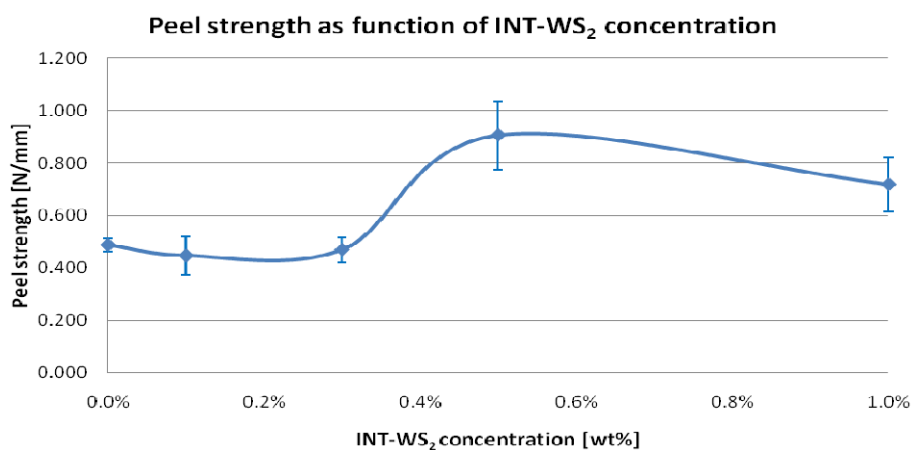


Fig. 3. Peel strength vs. INT-WS₂ conc.

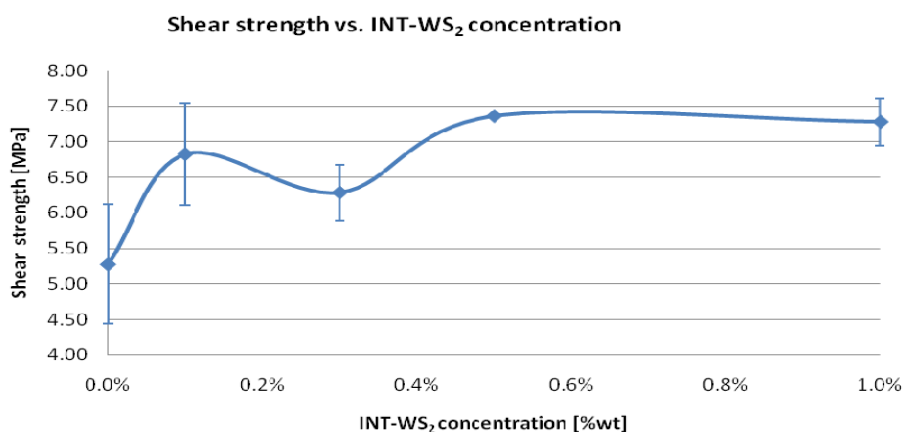


Fig. 4. Shear strength vs. INT-WS₂ conc.

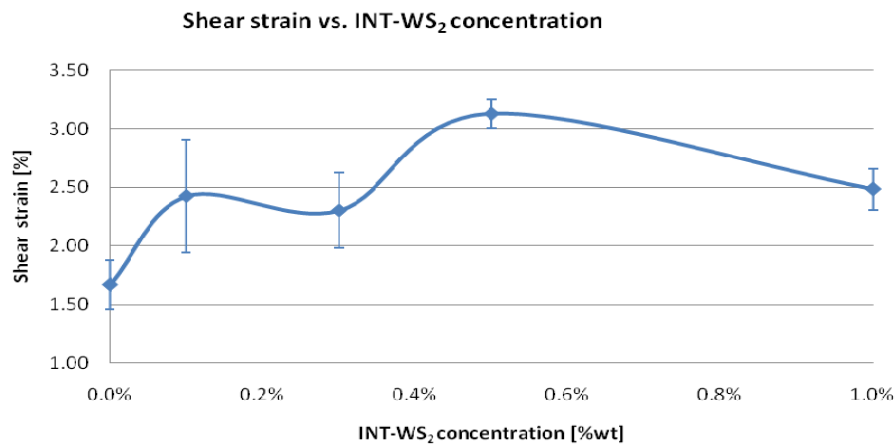


Fig. 5. Shear strain vs. INT-WS₂ concentration.

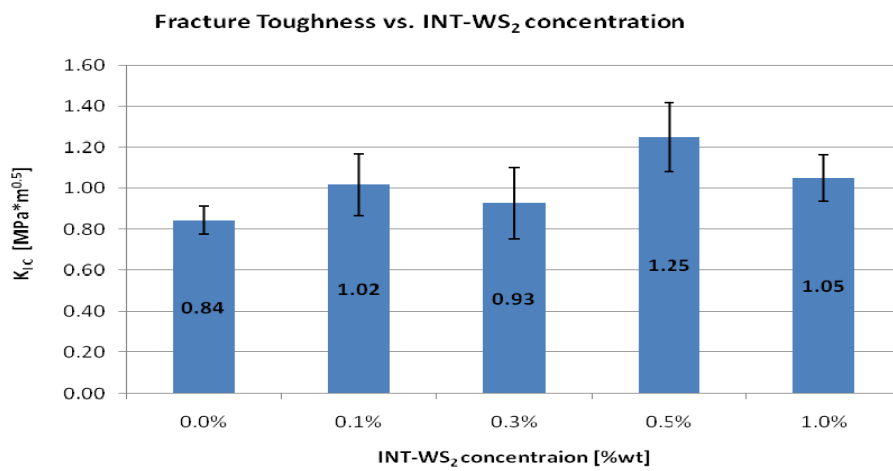


Fig. 6. Fracture toughness vs. INT-WS₂ concentration.

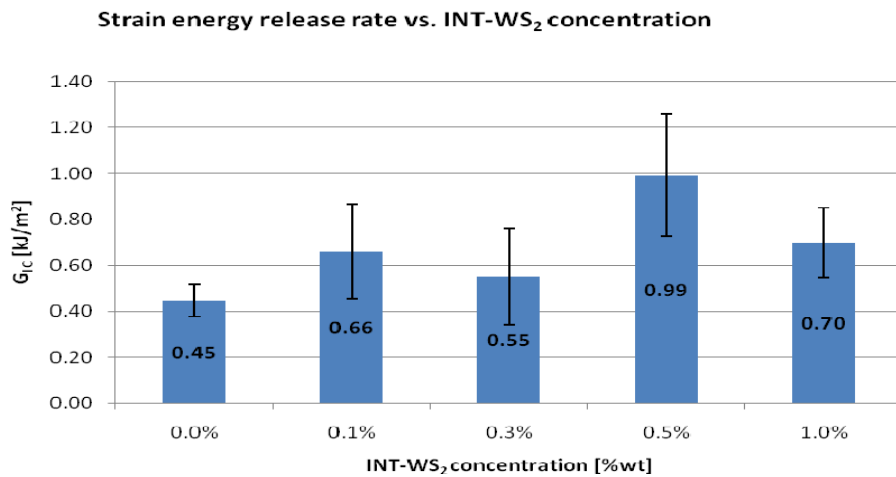


Fig. 7. Energy release rate vs. INT-WS₂ concentration.

The critical stress intensity factor (K_{IC}), or fracture toughness, also reaches a maximum value at nanotube content of 0.5 wt% (Fig. 6). The detailed calculation may be found in [34]. Fracture toughness is a property which describes the ability of a material containing an initial crack to resist

fracture (crack propagation), and is one of the most important properties of any material for virtually all design applications. The fracture toughness is a measure of the brittleness of the composite. It is denoted by K_{Ic} and has the units of $\text{Pa}\cdot\text{m}^{0.5}$. To calculate the K_{Ic} we are using the energy and crack dimensions.

The calculated strain energy release rate (G_{Ic}) during fracture is shown in Fig. 7. Again, it is evident that at 0.5 wt% more energy was released compared to all other concentrations. Strain energy release rate (or energy release rate) is the energy dissipated during fracture per unit of newly created fracture surface area. The tougher the material is the more energy is required for the crack to propagate through it during fracture. This implies that the increase in strain is the result of energy absorption that yields from strong nanotube-matrix interaction. As demonstrated by SEM analysis, the main source of increased toughness is the pullout/bridging mechanism [35]- see for example Fig. 8. These findings are similar to those of studies of the toughness of other epoxy-based nanocomposites [13, 29], including carbon nanotubes-based ones [14, 34].

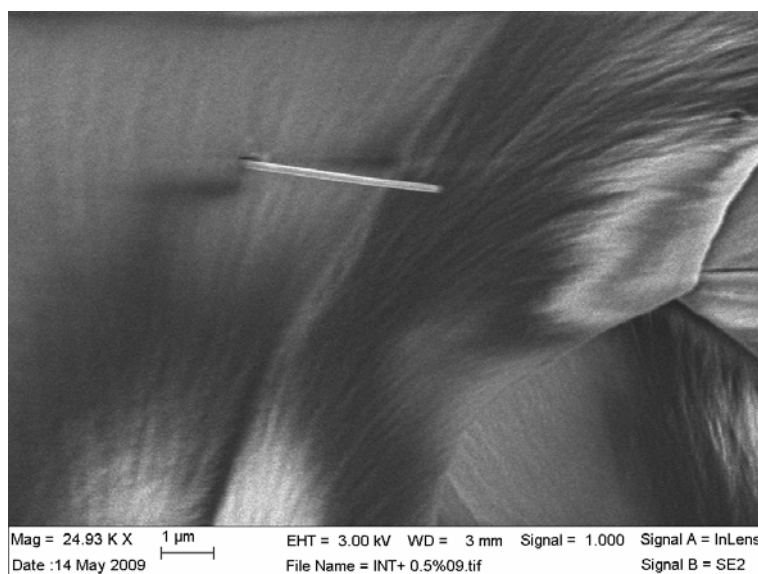


Fig. 8. Evidences of pullout mechanism - pulled out INT-WS₂.

3.3. SEM Observations

The mechanical analysis was supplemented by extensive SEM analysis, shown in 29b in greater detail. The fractured nanocomposite specimens exhibit significant differences in surface roughness compared to neat epoxy. The latter show vast plains with flat and smooth appearance, typical of brittle materials, as seen in other work [13, 14, 18]. The few sites where local yielding is observed to occur are far from each other resulting from curing inconsistencies/defects. On the other hand, the 0.5 wt% INT-WS₂ toughened epoxy specimens exhibit a shear band failure mechanism [2, 3, 36], a result of much more extensive, denser local yielding. This is also the reason for the increased surface roughness, which was previously correlated with an increase in fracture toughness [37, 38]. Nanoparticles produce localized matrix yielding and act as inhibitors of crack propagation via pinning, bowing, and deflection, whereas nanotubes trigger bridging and pullout mechanisms as well (see Fig. 8) [13, 14, 18, 19, 35]. Furthermore, the pulled out nanotubes appear to be coated with epoxy: the apparent tube diameter is about 200 nm, whereas the diameter of pristine INT-WS₂ is 150 nm at most. This suggests a strong adhesion between the nanotubes and epoxy, and provides a satisfactory explanation of the mechanical properties improvement.

3.4. Dynamic Mechanical Thermal Analysis

DMTA tests show that the INT-WS₂ have a positive influence on the matrix thermal properties at all nanotube concentrations, again with a maximum effect at 0.5 wt%, where the glass transition temperature (T_g) increased by up to 6.5 °C (Fig. 9). These findings are in accordance with studies conducted on epoxy matrices with various types of nanofillers, which have shown that T_g of epoxy is increased by about 10% [18, 29, 39]. We presume that these changes in T_g (shown by shift in the $\tan\delta$ maximum [29b]) are generated by the restriction of the polymer segment mobility, due to the presence of the nanoparticles which act as anchors. At greater concentrations this effect is counteracted by agglomeration, which reduces the cross-linking density and stiffness. It is apparent that in contrast with the other mechanical results, the flexural storage modulus holds the highest value for the 1.0 wt% INT-WS₂ (Fig.10). This observation is only true at temperatures lower than 85°C, above which the 1.0 wt% INT-WS₂ reinforced nanocomposite became less of its storage modulus in comparison to the 0.5 wt% INT-WS₂ reinforced one.

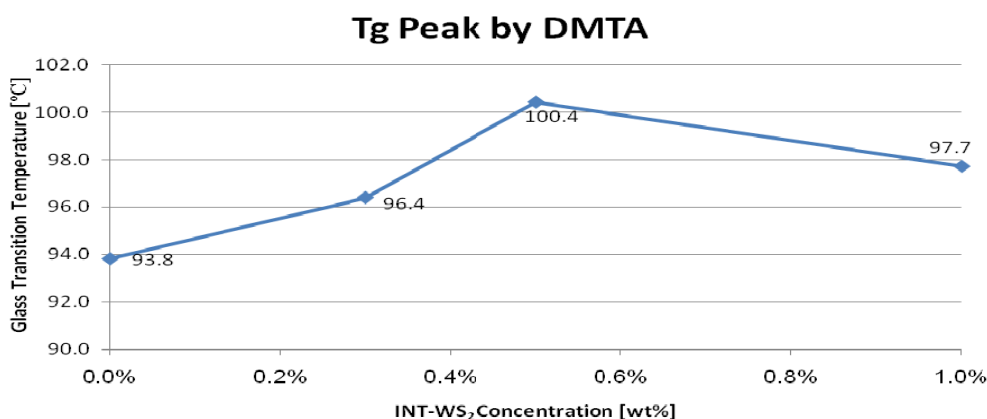


Fig. 9. Glass temperature (T_g) vs. INT-WS₂ concentration.

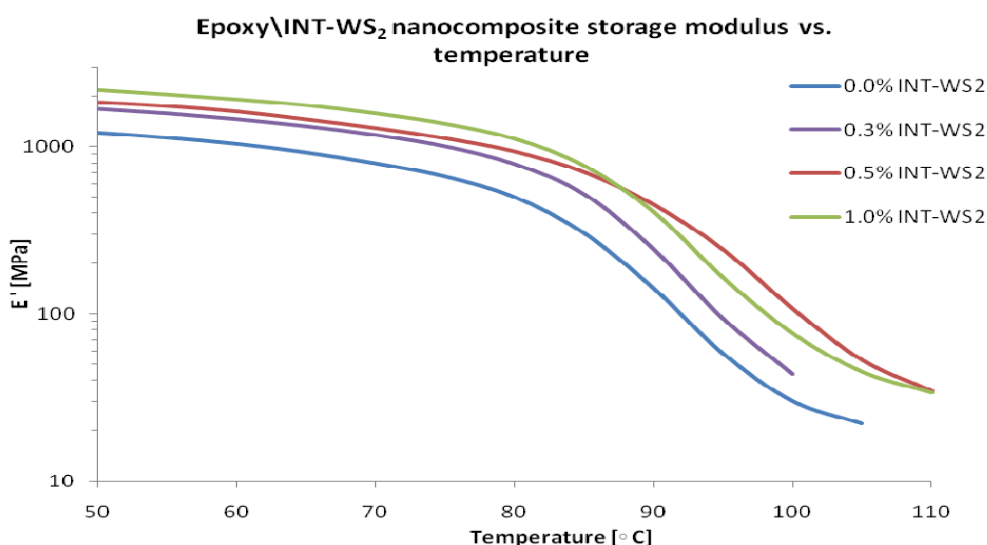
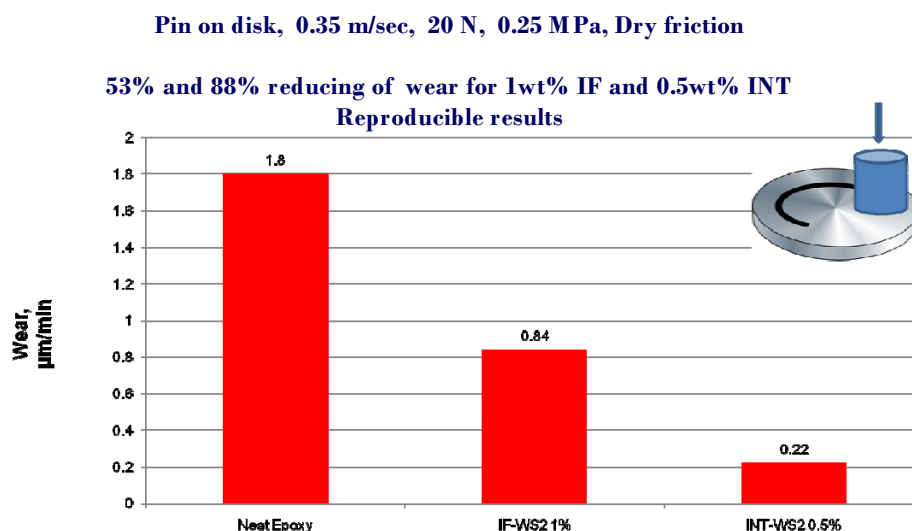


Fig. 10. DMTA curve showing storage modulus vs. temperature for various INT-WS₂ concentrations.

3.5. Tribological Measurements

Pin on disk tribological measurements of the epoxy nanocomposites with 1.0 wt% of IF-WS₂ and 0.5 wt% of INT-WS₂ indicated substantial reduction in wear as a result of adding the nanoparticles and nanotubes. Epon 828 and Versamide 140 were used to prepare these composites. Fig. 11 shows reproducible results of this study, where adding of 0.5 wt% of nanotubes led to a remarkable reduction in the wear of the epoxy nanocomposite. The wear of the nanotubes-reinforced epoxy was more than eight-times lower than that of pure epoxy. Adding 1 wt% of nanospheres decrease the wear by more than two-times, showing inferior results to the 0.5 wt% of nanotubes seemingly being more than the optimal concentration for the nanospheres or indicating the influence of nanoparticles shape.

Tribological properties of Epoxy - IF/INT-WS₂ nanocomposite



Prof. L.Rapoport, Tribological lab., HIT, June 2010

Fig. 11. Histogram summarizing the wear measurements of Epon 828/Versamide140 epoxy compounded with INT-WS₂ and IF-WS₂.

4. Conclusions

WS₂ nanotubes can now be synthesized in a pure phase and in substantial quantities. This allows them to be dispersed in nanocomposites and tested. Indeed in the current study, INT-WS₂ were dispersed in an epoxy matrix and a number of mechanical properties were measured to characterize the adhesion to a substrate, tribological properties as well as the fracture toughness of the nanocomposites. DMTA tests provided thermal stability assessment. The maximum effect of the nanotubes on all mechanical properties and on the glass transition temperature is achieved at a relatively low nanotube concentration (0.5 wt%). The SEM studies of the fractured specimens showed evidence of the energy absorbing failure mechanisms, the most dominant of which being bridging and pullout at the nano-scale. Crack pinning/deflection was also observed. The combined effects of the INT-WS₂ on the mechanical properties of the nanocomposite, i.e. an increased strength, strain and toughness along with improvement of the thermal properties shows promise in the field of nanocomposited adhesives and coating, as well as for structural applications.

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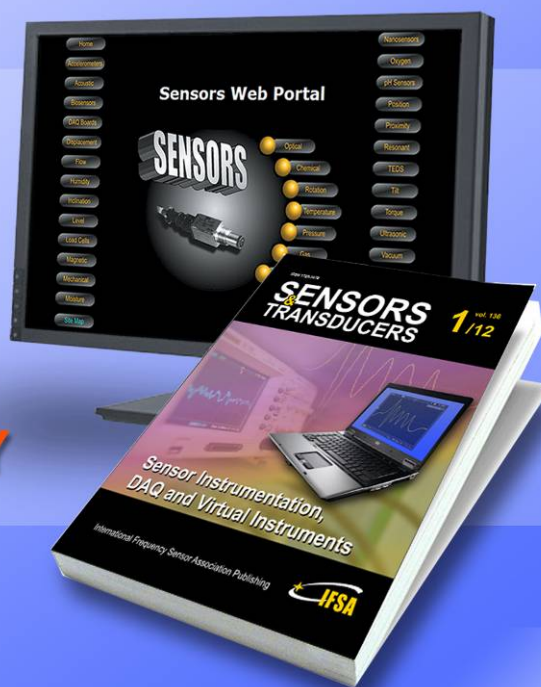
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