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Investigation of Nano-Mechanical Properties of Polyurethane Silver Nanocomposite by Nano Indentation

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Abstract

Polyurethane(PU) is used for huge variety of application to mankind. For example in medical field as catheter and in surgical and medical equipment, in instrumentation and in construction for its toughness properties. Metal nanoparticles in PU would enhance its properties more for specific applications. Hence in recent years, PU-metal Nanocomposite is explored more for its various properties for its potential applications.

PU-Silver Nanoparticle (PU-AgNP) composite synthesized was characterized by different methods such as Scanning Electron Microscopy, UV-VIS Spectroscopy, X-ray Photoelectron Microscopy. Nanoindentation is one of the advanced method which provides a wealth of valuable quantitative information regarding the mechanical properties such as elastic modulus, hardness, stiffness, creep test, fatigue of a variety of materials. Nanoindentation technique relies on the local deformation phenomena of materials at small length in nanometer and micrometer regime and load scales using indenter by application of a load. The indentation depth variation is a combination of both the visco and elastic contribution to the total indentation depth. Hence, the nanomechanical properties of PU-AgNP composite are investigated using nanoindentation. These properties measured using nanoindentation are critically very important for polymer nanocomposites applications such as coatings, food packaging industry, medical implants etc.

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Introduction

Polyurethane (PU) polymers are widely used in various industrial areas such as civil construction, carpentry, coating for various purpose, electric insulation and instrumentation due to their excellent physical and mechanical properties. Polyurethane have been increasingly used in bridges, piers, retaining walls, and storage structures exposed to salts and chemicals. Polyurethane nanocomposites have advantageous properties, such as good mechanical properties, high strength-to-weight ratio, non-corrosiveness, and high energy absorption properties. The mechanical properties of PU nanocomposites have a time dependent nature, i.e. strength and stiffness are time-dependent due to the hereditary nature (viscoelasticity) of polymers. In this context, lifetime models for viscoelastic materials, i.e. fatigue investigation is done. A design life of 10 to 50 years is required for important areas of application of Polymers nanocomposite which include the automotive and aeronautical industry, bridge structures, water and waste systems and more recently in the offshore exploration and oil production. It is not expectable to perform tests on either materials or structures for very long periods to cover the design lifetime. Therefore there is a strong need for accelerated lifetime characterization methodologies which can predict the evolution of stiffness and strength of materials used in construction in order to assure the integrity and safety of structural components. During the last 40 years several lifetime models for viscoelastic materials have been proposed. These models include empirical models, statistical models and theoretical models. The theoretical models are focused on a molecular scale and based on rate theory for the breakage of molecular scale bonds or on a continuum

approach based on the fracture mechanics theory or energy-based criteria. In this work lifetime expressions based on fracture mechanics theory, constant stress rate are found to be satisfactory and performs well in case of loading. Hence a general formulation to predict the fatigue behaviour for arbitrary load ratio (R) is proposed.

Developing polyurethane silver nanoparticle for surface coating technologies provide enhanced toughness and improved viscoelastic characteristics, compared to the historic polyurethane products manufactured using relatively brittle resins (e.g., polyester and vinyl ester). In coating/substrate layered systems, coatings becoming breakable, cracking, and even breaking-off from the substrate, means the failure of structures, equipments or devices. Therefore, the mechanical properties of coatings such as fatigue strength are one of the most important performance indexes. With the development of surface technology for coatings and its applications to various industrial areas, a perfect evaluation of its nanomechanical properties is required urgently. The conventional methodologies testing mechanical properties are to stretch or compress the coatings which are made into standard testing pieces by material testing machine. Since the characterization of mechanical properties of the thin miniature layers is hardly achieved via conventional testing methodologies, and also the properties of the layers are different from those of the bulk state, nanoindentation techniques which is an advance technique is used for its characterisation.

Experimental

The fatigue behaviour of newly developed polyurethane silver nanocomposites is characterized for fatigue life prediction including hardness and elasticity using nanoindentation technique carried on by CSM instruments using Oliver & Pharr (Power Law method).

After calibration of the instrument, a small uniform layer of polyurethane silver nanocomposite with well polished surface was kept on the stage of CSM instrument. A target was fixed with the inbuilt microscopic camera of the instrument. Sample was once intended with spherical indenter with the following parameter given in table 1 for linear loading.

Table 1: Indentation parameters for optimization

Acquisition Rate	10.0 [Hz]
Max load	1.0 mN
Loading rate	2.0 mN/min
Unloading rate	2.0 mN/min
Pause	10.0 s
Fn contact	0.05 mN
Approach distance	1500 nm
Approach speed	5000 nm/min
Retract speed	2000 nm/min
Retract Time	5 sec

With the above parameter, the resultant curve for load versus contact depth is as below in figure 1.

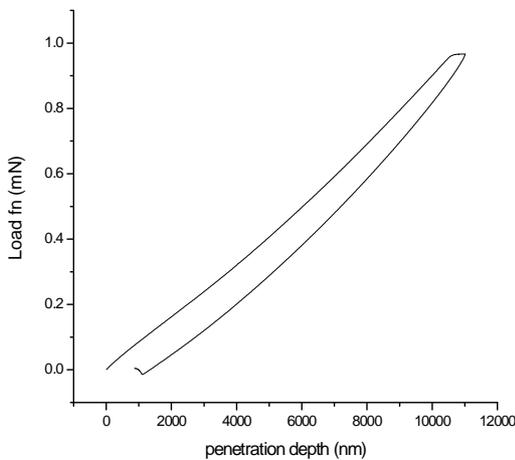


Figure 1: Load F_n (mN) versus penetration depth (nm) for single indentation

From the above graph analysis, following result in table 2 was interpreted for nanomechanical properties.

Table 2 : Result for nanomechanical properties interpretation

Properties	Unit	Result for single indentation	
Indentation Hardness	HIT	MPa	1.0534
Indentation Elasticity	EIT	MPa	3.0757
Indentation Creep	CIT	%	4.64
Contact Stiffness	S	mN/nm	0.0001
Elastic work done	W_{elast}	pJ	4086.71

From the above indentation, all the parameter was optimized for fatigue testing. An Indentation is done for 25 cycles for fatigue analysis with the optimized parameter given below in table 3 with the constant periodic cycle as seen in the graph given in figure 2 .

Table 3: Optimized Indentation parameter

Acquisition Rate	10.0 [Hz]
Max load	0.8mN
Unload to	0.5mN
Loading rate	1.6mN/min
Unloading rate	1.6mN/min
Pause	5.0 s
Fn contact	0.05 mN
Approach distance	1500 nm
Approach speed	5000 nm/min
Retract speed	2000 nm/min
Retract Time	5 sec

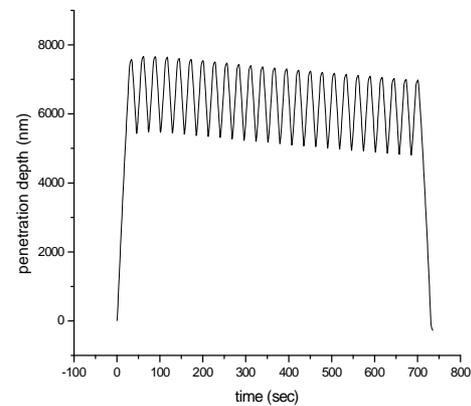


Figure 2: Penetration depth versus time

The graph of load versus penetration depth is plotted for fatigue analysis which is given in figure 3 which shows that the material is having good elasticity.

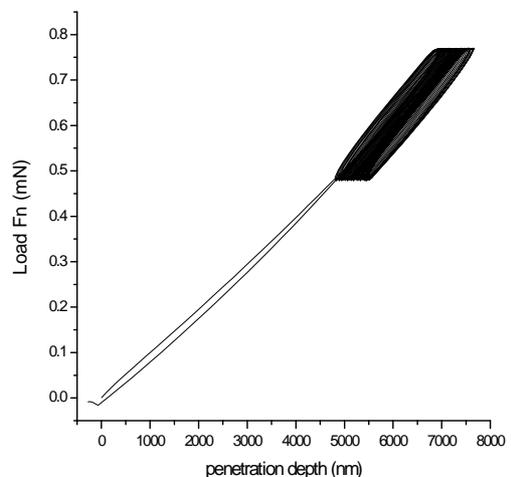


Figure 3: Load F_n (mN) versus penetration depth (nm) for 25 cycle

The change in nanomechanical properties can be seen from the result acquired from the above graph analysis which is summarized in a tabular manner in the given below table 4.

Table 2 : Result for nanomechanical properties

Nano mechanical Properties	unit	before fatigue	After fatigue	
Indentation Hardness	HIT	MPa	1.7651	3.1280
Indentation Elasticity	EIT	MPa	5.0628	6.1209
Indentation Creep	CIT	%	3.7	3.11
Contact Stiffness	S	mN/nm	0.0002	0.0001
Elastic work done	W_{elast}	pJ	1296.69	2396.2

From the above result interpretation, we can say that hardness and elasticity got increased and stiffness and creepiness got decreased after fatigue. For further investigation of these nanomechanical properties changes throughout the cycles, We studied the changes in each parameter individually with reference to the cycle number.

a) Variation of Indentation Elasticity wrt cycle number

From the following graph given in figure 4, it can be seen that Indentation Elasticity increases with the increase in cycle number.

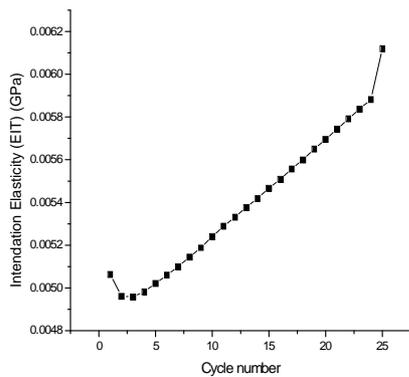


Figure 4: Indentation Elasticity versus cycle number

b) Variation of Indentation Hardness wrt cycle number

From the following graph given in figure 5, it can be seen that Indentation Hardness increases with the increase in cycle number.

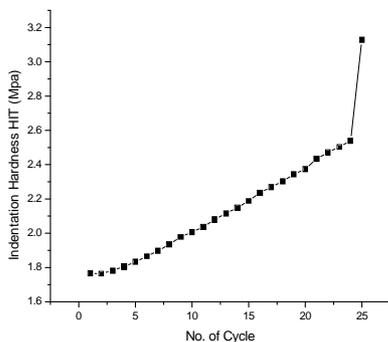


Figure 5: Indentation Hardness versus cycle number

c) Variation of Indentation Creep wrt cycle number

From the following graph given in figure 6, it can be seen that Indentation Creep initially decreases very fast then soon gradually

increases with the increase in cycle number. This initial decrease in creep may be due to some grain boundaries defects getting dislocated in polymer matrix due to indentation load.

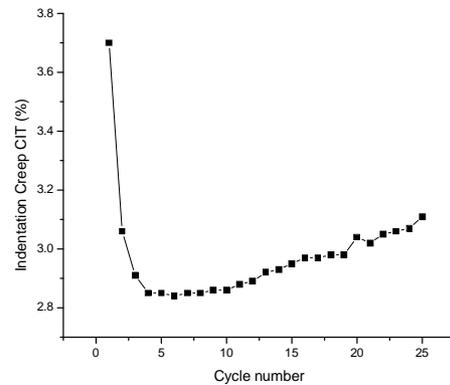


Figure 6: Indentation Creep versus cycle number

d) Variation of Indentation Creep wrt penetration depth

From the following graph given in figure 7, it can be seen that Indentation Creep decreases gradually till good depth then suddenly increases with the increase in cycle number. The initial gradual decrease in creep shows too many of dislocation of grain boundaries near the surfaces of polymer due to indentation

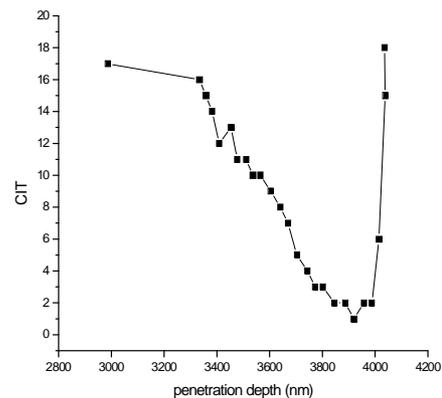


Figure 7: Indentation Creep versus penetration depth

e) Variation of Ratio of HIT/EIT wrt penetration depth

From the following graph given in figure 8, it can be seen that with the increase in penetration depth hardness becomes more prominent than elasticity because probability of dislocation of grain boundaries decreases

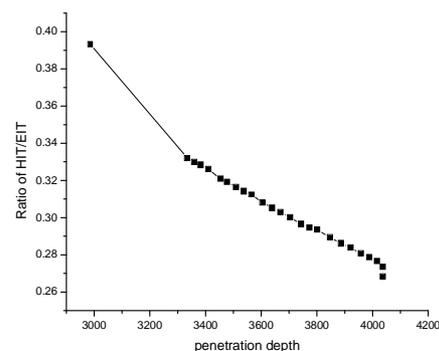


Figure 8: Ratio of Indentation Hardness to Indentation Elasticity versus penetration depth

Conclusions

Fatigue testing analysis of synthesized polyurethane silver nanocomposite gave interesting results which has created lots of curiosity to study it more and has evoked for carrying out more number of experiments. Increase in hardness and elasticity after fatigue make this material of great interest for tough use in construction for better life time.

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