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Online structural health monitoring of polymer composite structure using gold nanoparticles (AuNPs)

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K E Y W O R D S	ABSTRACT				
Gold Nano Particles	A mechanism for online structural health monitoring of composite structure				
Synthesis	was established by developing highly sensitive strain gauges based on colloidal Gold Nano Particles (AuNPs). They were synthesized using well				
Structural Health Monitoring	known Turkevich-Frens method followed by phase transfer in chloroform				
Smart Sensor	using surfactant Cetyltrimethylammonium bromide (CTAB). Flexible Strain				
Polymer Composite	gauges / smart sensors were formed by mixing colloidal AuNPs-chloroform solution in polystyrene (PS) – chloroform solution and depositing their thick				
	paste on hand-made flexible thermoplastic polyurethane (TPU) film. These sensors were used as strain gauges on composite substrates for tensile testing				
	The developed AuNP based strain gauges were found to have 10 times higher				
	sensitivity than normal metal foil strain gauges.				

1. Introduction

Composite materials have wide range of applications from ordinary household to high end usage like in aerospace industry [1]. The world is shifted towards composite due to its unique feature of high strength to weight ratio, low production cost and enhanced mechanical properties [2]. They are now use in aerospace, medical instruments, structures, sports equipment, and vehicle's parts etc. [3]. They have heterogeneous behaviour which makes them difficult for in-situ monitoring [4]. Real time SHM of composite is very important in this regard. Lot of work is being done in integrating the sensor within composite especially in case of aerospace industry where we need to monitor parts in its working condition. This will not also improve their shell life but also ensure the safety of onboard passenger [5]. So, there is a need of their self-sensing in which sensor is integrated with in the structure and eliminate the usage of external sensor.

Strain gauges for the purpose of detecting deformations and flaws are the farthest important for

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structural health monitoring (SHM)[6]. Highly sensitive, low fabrication cost, smaller size and flexibility in usage are the most desirable features. Normal metal foil strain gauges can't fulfill all of these necessities with their gauge factor G= $(\Delta R/R0)/\epsilon$ where ($\Delta R/R0$ is the relative electrical resistance variation ϵ is the strain) of about 2 to 4. Whereas semi conductive based strain gauges have gain of 20 to 100. Though, they have little disadvantages as well; their sensitivity decreases with the increase in applied strain [7].

Recently, lot of work has been done on metallic nanoparticles based smart sensor [8]. They exhibit the same sensitivity as of semi conductive based gauges especially in case of tensile strain. The highly sensitivity is mostly dependent on tunnelling effect between two adjacent nanoparticles [9]. Controlling the size and shapes on nanoparticles via chemical synthesis is very interesting. This provides the opportunity to further optimize the sensor performance such as in using different capping agents, chemical concentration and by varying temperature and speed of stirrer [10]. Different techniques is being used for nanoparticles suspension on flexible substrate like air-brush, spraying [11], layers by layers deposition [12] and micro contact printing [13]. Nanoparticles also embedded with in thermoplastic like carbon blackpolystyrene in our previous work to form semi conductive paint [14]. However, there is always slight window open to improve the sensor performance by some changes in nanoparticles chemical synthesis, deposition technique and data acquisitioning method.

Our aim is to measure the response of Glass fiber reinforced polymer (GFRP) composite using smart sensor based on AuNPs for in-situ SHM of loaded specimen and correlate the Normalized strain (Δ R/R) with the applied strain to check the predictability and validity of the sensor.

2. Material and Methods

2.1 Synthesis of Gold Nanoparticles

Gold chloride (HAuCl4, ~50% Au basis, Aldrich) and Sodium citrate (Assay 99%, Deajung) were used for synthesis of AuNPs. Following Turkevich-Frens method [15], 50 ml of 38.8 mM Trisodium Citrate was added in boiling 500 ml of 1 mM Gold Chloride solution. Solution became colorless to dark and finally pink after some time. Pink color indicates the formation of AuNPs. At this point the heating was stopped, and the solution was allowed to cool down to room temperature.

Phase transfer of AuNPs was carried out by following the procedure described elsewhere [16]. 400 ml of chloroform was added in colloidal AuNPs solution and stirred for a minute. 800 mg of CTAB was then added and stirred for about 1 to 2 hours. AuNPs settle down at the bottom of beaker in chloroform phase which are then separated and mixed with PS-chloroform solution, calcinated and pasted on TPU films to form stretchable PS-AuNPs/TPU smart sensors.

2.2 Vacuum Bagging

GFRP composite laminates were manufactured using vacuum assisted resin transfer molding (VARTM) [17] by epoxy/hardener as matrix and glass fabric were reinforcement and was cut into specimen of standard D3039 for tensile testing.

2.3 Electro-Mechanical Tensile testing

GFRP sheets were cut into 5 specimens of 250 mm X 25 mm each, following ASTM D-3039 standard, for the purpose of electro-mechanical characterization of developed sensors. Smart sensors were pasted on GFRP specimens in the center. The

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specimens were subsequently tested on UTM in tensile loading mode. The electromechanical response of sensors was registered using Keithley KUSB-3100 data acquisition module in conjunction with a Wheatstone bridge and an instrumentation amplifier for data amplification and noise reduction. The sensing layer deposited on the GFRP specimens was connected to conductive wire using silver conductive paint in order to reduce contact resistance and make robust connections. The wires were connected such that they were 20 mm apart.3.

3. Results and Discussion

The SEM-EDS (Scanning Electron Microscope-Energy Dispersive Spectroscopy) performed using VEGA TESCON3 confirmed the presence of gold (Fig. 1)



Fig. 1. SEM-EDS Image of AuNPs

After the phase transfer of AuNPs from aqueous to chloroform had been achieved by adding CTAB, polystyrene (PS) was added in AuNPs-chloroform solution to form thick reddish wine-colored paste upon drying. This paste was deposited on stretchable thermoplastic polyurethane (TPU) film to form stretchable smart sensor for strain monitoring.

3.1 Vacuum Bagging

GFRP laminated composites were manufactured by VARTM (Vacuum assisted resin transfer molding) of the woven glass fiber reinforcement. The lay-up parameters of the laminated composite are given in the Table 1. The tensile test specimens were cut according to the dimensions specified in ASTM D3039. Metallic strips for strong gripping and uniform load distribution were pasted onto the specimen extremities to serve as tabs.



Fig. 2. Vacuum Bagging Set-Up



Fig. 3. GFRP Specimen

3.2 Electro-mechanical Testing

For measuring resistance change Keithley KUSB-3100 is connected with Wheatstone bridge. In order to amplify the output some sort of amplifier like INA 118 is used as:



Fig. 2(a). Circuit Diagram of Wheatstone Bridge With Amplifier INA 118



Fig. 2(b). Keithley With Wheatstone Bridge and Amplifier



Fig. 3(a). SHM Set Up Circuit Diagram



Fig. 3(b). SHM Setup

For Balanced condition of Wheatstone bridge

Vout = 0 $V_{in} \frac{R_1}{R_1 + R_2} = V_{in} \frac{R_3}{R_3 + R_4}$ $\frac{R_1}{R_2} = \frac{R_3}{R_4}$

As R1 and R2 is fixed, so any change occurs in R4 (gauge resistance), R3 has to be changed in order to balance the bridge i.e. to maintain Vout at zero. So variable resistance is placed over R3 during the formation of circuit. The change in R4 has to be rationalized to be able to compare the different test results when plotted against strain. If R4/-R4 is the change in resistance, then the normalized resistance would be according to

Normalized Resistance = $\frac{\nabla R}{R} = \frac{R_4^{\prime} - R_4}{R_4}$

For strain gauges, with high gauge factor, simple voltage divider circuit is not good enough due to its high sensitivity due to strain that will result in noise. In order to amplify the output voltage and to reduce noise, instrumentation amplifiers are mostly used. Instrumentation amplifiers are the ICs which amplify the output signal without the risk of unbalancing the bridge circuit.

The complete SHM set up was shown in Fig. 3. The GFRP specimen were subjected to tensile loading and their online response was monitored and recorded by Keithley KUSB-3100 DAQ (data acquisitioning) set-up and were compared with actual response of specimen.

The geometry and sensor characteristic of all the specimen is shown in Table 1.

Sensor and specimen characteristics

#	Specime	Gaug	Area	Senso	R0	Gain
	n	e	(mm2	r wire	(KΩ	resist
	Name	lengt)	apart)	ance
		h				
		(mm)		(mm)		
1	T1.1	150	45.49	20	1.6	Open
						circui
						t
2	T1.2	150.1	48.24	20	5.9	Open
		3				circui
						t
3	T1.3	147.8	43.54	20	5.5	Open
		3				circui
						t

The response of all the specimen comparison with the actual response was shown in Fig. 4.



Fig. 4(a). T1.1 Stress-Strain-Normalized-Strain-Curve



Fig. 4(b). T1.2 Stress-Strain-Normalized-Strain-Curve



Fig. 4(c). T1.3 Stress-Strain-Normalized-Strain-Curve



Fig. 5. SEM Image of AuNPs

As you can see, some points on the curve of normalized resistance strain and coincides. indicating the sensors are responding well. Especially the sensor T1.2 which behave well even after fracture. GFRP Composites have unusual behavior compared to metals. They broke fiber by fiber. High gain of sensors has little drawbacks as well. They detect electric motive force (emf) of the machine and produces noise. This is somehow prevented by using capacitors in circuit and common grounding. Fig. 5 shows the particles size well below 40 nm.

4. Conclusion

It was observed that size and distribution of GNPs play an important role in behavior of strips i.e. smaller the nanoparticle's size, more easily they phase transfer and more uniformly it suspends in nanoparticle's smart strip. Smart strips pasted on GFRP composite specimen behave like strain sensor and respond well in tensile loading. Capping agent prevents the particles form oxidation and create tunneling which is required for Piezo-resistive semiconductive behavior. Higher strain gauge factor resulted increased sensitivity which can be beneficial for high precision application especially in biosensor. Deposing GNPs on flexible substrate make them useful for conductive surfaces as well, unlike carbon, GNPs are not health hazard and freely used in biomedical.

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