

## Composite Adhesive-Bonded Joint Reinforcement by Incorporation of Nano-Alumina Particles

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### Abstract

Adhesive bonding technology is being used in a variety of modern industries, including the automotive, aerospace, maritime, construction, defense and so on. On the other side, polymeric nano - composites attracted both academic and industrial interests in the past decades. The scope of this paper is experimental investigation on the effects of the addition of Alpha-alumina nanoparticles to the woven glass / epoxy composite and Araldite 2015 adhesive on the mechanical properties of the composite adhesive bonded joints. In this study, vacuum assisted resin transfer molding was used to fabricate experimental samples and to fabricate composite samples, 6 glass-fiber layers with a surface density of 200 g/m<sup>2</sup> were used. The study of the influences of the addition of Alpha-alumina nanoparticles with different weight ratios to glass/epoxy composites suggests that the maximum values of the ultimate strength, elongation, toughness, and Young's modulus belong to the samples with the weight ratios of 0.43, 1, 1, and 2.1%, respectively. The experimental results from the shear tensile test show that the incorporation of 0.74 wt% of nanoparticles to the adhesive increases the joint strength by about 14%.

**Keywords:** Adhesive Joint, Composite, Nanoparticle, Mechanical Properties, VARTM

### 1. Introduction

The use of adhesive-bonded joints is becoming more common than mechanical joints in engineering applications, as they are associated with further advantages, including lower structural weight, lower fabrication cost, and improved failure tolerances. In recent years, the uses of such joints in fiber-reinforced composite structures are rapidly growing. Traditional joints usually cause fiber cutting and reduce the structural integrity by introducing stress concentrations. In adhesive-bonded joints, the bonded joints are more continuous and have potential

advantages in terms of strength-to-weight ratio, design flexibility and ease of fabrication. In fact, adhesive bonding is widely applicable in various highly advanced technology industries, such as aeronautics, aerospace, electronics, and automotive to traditional industries, such as construction, sport products, and packaging. These structures are made of different fiber types and textures, and resins. Adhesive-bonded joints are expected to sustain many static or cyclic loads for long periods without any adverse effect on the structural load-bearing capacity. The lack of suitable behavioral models and failure criteria has resulted in a tendency to overdesign composite structures.

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The use of particles and fibers in nano-composite structures usually improves the strength of the basic material. When particles and fibers are distributed throughout the basic material, the loads applied to the composite are evenly transmitted to the particles or fibers. Distribution of filling materials across the basic material changes the properties, such as strength, hardness, and porosity, as well as tribological features. The basic material can keep particles apart in a way that crack growth is delayed. In addition, due to the surface interaction between basic material and filling materials, nano-composite components have better properties. The type and amount of interactions have a significant effect on different properties of nano-composites, such as solubility, as well as optical, electrical, and mechanical characteristics.

Bodaghi et al. [1] investigated the effect of adhesive reinforcement in single-lap adhesive-bonded joints under impact and tensile loadings in thermal cycles. They examined the mechanical behavior of epoxy adhesive-bonded joints, which were reinforced with carbon nano-fibers. The amount of carbon nano-fibers in the resin was 1.3 (5% of the resin's weight). Particles had been distributed throughout the resin with a manual stirrer and an ultrasonic device.

Gojny et al. [2] comprehensively reviewed the effect of nano-fillers on the fracture toughness of epoxy resins and related microorganisms. They studied hardening mechanisms from two perspectives: 1) micro-mechanical mechanisms, such as crack deflection at agglomerates, the extension of the plastic deformation zone, etc.; 2) nano-mechanical mechanisms, such as interfacial debonding, pullout and crack bridging in the presence of carbon nanoparticles.

Adding alumina nano-fibers to epoxy adhesives slightly increases the strength of sample aluminum joints (Gilbert et al. [3]; Meguid and Sun [4]); whereas, the effect of nano-reinforced composites on the

toughness of carbon/epoxy composite joint is significant, vary based on the fabrication method (prefabricated versus co-cured). Zhai et al. conducted several studies into the effect of adding alumina nanoparticles to epoxy resin for bonding steel samples [5,6]. Figure 2 presents their experimental findings, along with microscopic images of the alumina nanoparticles.

Yu et al. [7] developed carbon nanotubes epoxy nano-composite adhesives, to be utilized in aluminum joints. It was found that the durability of epoxy-based adhesive joints under water at 60 °C was greatly improved for nano-filler concentrations up to 1 wt%. Xi et al. [8] analyzed the electrical conductivity and the shear strength of polyurethane adhesives filled with different kinds of modified graphite, finding that the strength of the adhesive joints to aluminum increased up to a filler content of 20 wt%.

Tessema et al. [9] Using a unidirectional heat transfer apparatus investigated the roles of nanoparticle geometry, loading, dispersion and temperature on the thermal conductivity of polymer nanocomposites. The polymer nanocomposites (PNC) consist of epoxy matrices filled with silica nanopowder and carbon nanotubes, respectively, as well as poly (2-vinylpyridine) (P2VP) matrices loaded with silica nanoparticles.

Kaboorani et al. [10] investigated the feasibility of improving the mechanical properties of a high solid content UV-curable acrylic coating system through the addition of cellulose nanocrystal (CNC). The CNC was rendered hydrophobic by using a cationic surfactant, hexadecyltrimethylammonium bromide (HDTMA), to make it compatible with the matrix polymer and facilitate proper dispersion. Two different amounts (1 and 3%) of modified CNC were added to the coating system. Tensile strength and modulus of elasticity (MOE) of coating films were affected

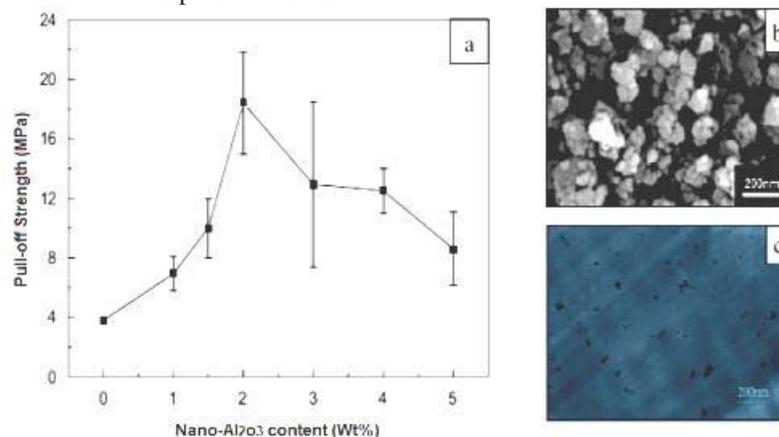


Figure 2: Effect of nano-Alumina on pull-off strength [5].

positively by the addition of CNC. Hardness, measured both by König pendulum and pencil methods, was found to increase as CNC loading increased in the coatings. Mass loss due to abrasion resistance tests was reduced by the addition of CNC. The results of this research showed that CNC is an ideal reinforcing nanoparticle which can positively affect the mechanical performance of coating systems.

Bian et al. [11] fabricated a novel PP nanocomposites containing graphene/SiO<sub>2</sub> hybrid (GOS) via melt blending. It showed that GO has been chemically functionalized by selective monomers. Due to the existence of “heterogeneous nucleation effect”, the melting temperatures and thermal properties were improved, as verified by thermal analysis. GOS has been dispersed uniformly in the PP matrix due to the “barrier effects” between SiO<sub>2</sub> and graphene sheets.

Following tables show the experimental results reported by other researchers concerning the addition of nanoparticles and its effect on mechanical properties

of adhesive-bonded joints (epoxy resin) for lap shear, pill stress, and double cantilever beam (DCB) tests.

In this study, Araldite 2015 was reinforced with nanoparticles, aiming to investigate the effect of nanoparticles on mechanical properties of adhesive-bonded joints. Moreover, the effects of adding nanoparticles on mechanical behavior (tensile strength, failure strain, Young’s modulus) of nanocomposite samples were evaluated. These experimental data can be used to estimate mechanical properties of nano composite materials and strength of single lap joint in different applications including aerospace, marine, and automotive industries. The novelty of this work is using nano-alumina particles to improved mechanical features ( ultimate strength, failure strain, toughness, and Young’s modulus) of woven Glass/Epoxy made by VARTM process and used a creative method to add nano particles to the Araldite 2015 adhesive to enhance joint strength.

Table 1: Effect of nano-particles on Lap shear strength of different adhesive joints

Nano-particle	Adherent	Neat	Nano-reinforced	Variation (%)	reference
Al <sub>2</sub> O <sub>3</sub>	Al	237 MPa	273 MPa	+15	Gilbert [3]
	Al	25.5 MPa	28.5 MPa	+12	Klug [12]
SiO <sub>2</sub>	Al	20.8 MPa	23 MPa	+11	Kinloch [13]
	Ti	25 MPa	40 MPa	+60	Bhowmik [14]
POSS	Al	21 MPa	24 MPa	+14	Dodiuk [15]
MWCNT	CF/epoxy	-	-	+46	Hsiao [16]
Al <sub>2</sub> O <sub>3</sub>	Al	237 MPa	265 MPa	+12	Gilbert [3]
	Al-CF/epoxy	-	-	+30	Meguid [4]
CNF	PMMA	28 MPa	32.5 MPa	+16	Xu [17]
	CF/epoxy	11.9 MPa	12.8 MPa	+8	Prolongo [18]

Table 2: Effect of nano-particles on Peel strength of different adhesive joints

Nano-particle	Adherent	Neat	Nano-reinforced	Variation (%)	reference
Al <sub>2</sub> O <sub>3</sub>	Al	87 N	130 N	+49	Gilbert [3]
SiO <sub>2</sub>	Al	3.1 N/mm	5.5 N/mm	+77	Kinloch [13]
POSS	Al	0.19 N/mm	0.49 N/mm	+158	Dodiuk [15]
Al <sub>2</sub> O <sub>3</sub>	Al	87 N	119 N	+37	Gilbert [3]

Table 3: Effect of nano-particles on Mode I fracture toughness (G<sub>IC</sub>)

Nano-particle	Adherent	Neat	Nano-reinforced	Variation (%)	reference
Al <sub>2</sub> O <sub>3</sub>	CF/epoxy	0.47 kJ/m <sup>2</sup>	0.85 kJ/m <sup>2</sup>	+81	Gilbert [3]
SiO <sub>2</sub>	CF/epoxy	0.59 kJ/m <sup>2</sup>	0.74 kJ/m <sup>2</sup>	+25	Klug [12]
	Al	1.2 kJ/m <sup>2</sup>	2.3 kJ/m <sup>2</sup>	+92	Kinloch[13]
Al <sub>2</sub> O <sub>3</sub>	CF/epoxy	0.47 kJ/m <sup>2</sup>	0.79 kJ/m <sup>2</sup>	+68	Gilbert [3]

## 2. Materials and Methods

### 2.1. Materials, Geometry, and Test Parameters

To fabricate composite samples, 6 glass-fiber layers with a surface density of 200 g/m<sup>2</sup> were used. In this study, Alpha-alumina nanoparticles with 99% purity, average nanoparticle size (ASP) ~80nm, and specific surface area (SSA) smaller than 10m<sup>2</sup>/g (Notrino Company) were used. The selected polymer resin was comprised of two components mixed with the weight ratio of 12%: (1) epoxy-based EPL 1012, and (2) EPH 112 as a hardener (Table 4).

In this study, vacuum assisted resin transfer molding (VARTM) [19] was used to fabricate experimental samples. In the following, the composite sample fabrication stages are described using VARTM:

1. Alpha-alumina nano-powder was heated in an oven at 80°C for 150 minutes and then at 120°C for another 150 minutes to ensure complete moisture loss.
2. To fabricate nano-composite samples, nanoparticles were first mixed with the hardener (due to lower viscosity of the hardener) with 0.2, 0.43, 2.1, and 4.1 wt% of the total weight of the resin, using a magnetic stirrer for 20 minutes and rotation rate of 1800 RPM. Then, the obtained mixture was sonicated for 15 minutes, using the Hielscher ultrasonic device UP400S set to Cycle 0.8 and Amplitude 80 μm.
3. The mixture was placed in a laboratory container and mechanically stirred with the resin for 5 minutes at the room temperature.
4. The glass die was cleaned using detergents like acetone.
5. The separating layer in the die was created with RENLEASE QV 5110.
6. The preform was comprised of 6 layers of glass fibers (200g; 30×20cm), a layer of Dacron fabric strips, and a distribution layer.

7. By preparing and connecting the hydraulic system, sealant, and vacuum bags, the die was prepared for injection of the resin.
8. The system was connected to a vacuum pump and the resin was injected into the preform and die under the vacuum pressure of -0.8 bar.
9. Figure 5 shows the different components of the VARTM fabrication process.

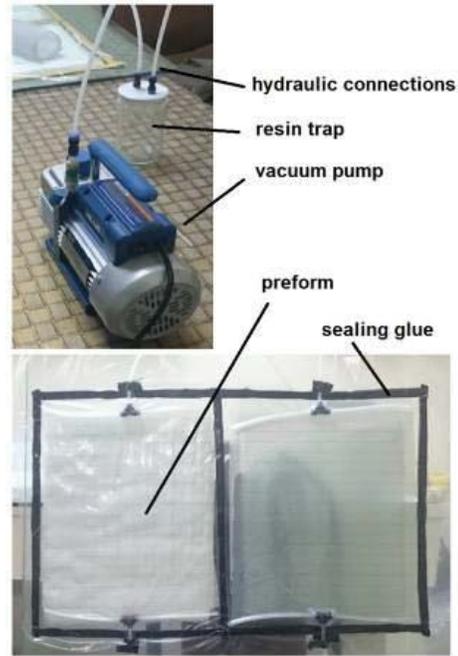


Figure 5: VARTM process.

The Araldite 2015 has two components and is cured in a short time after being mixed with nanoparticles, therefore, that part with lower viscosity rate was selected and nanoparticles were mixed with it, using a mini-stirrer (a DC motor and a miniature arm) for 30 minutes. It was then mixed with the second part of the mixture for bonding.

Fig.6 presents scanning electron microscope (SEM) images of the sample with 1wt%, showing good nanoparticle distribution in the matrix.

### 2.2. Tensile test

After curing and preparing nano-composite

Table 4: Mechanical properties on resin and hardener (Factory Data Sheet).

Viscosity at 25°C(mPa.s)- EPL1012	900-1100	Shore Hardness	82
Viscosity at 25°C(mPa.s)- EPH112	30	Mix Ratio	12/100
Young's Modulus (GPa)	2.73	Gel Time	24 min
Tensile Strenght (MPa)	74.62	Full Cure	7 Days

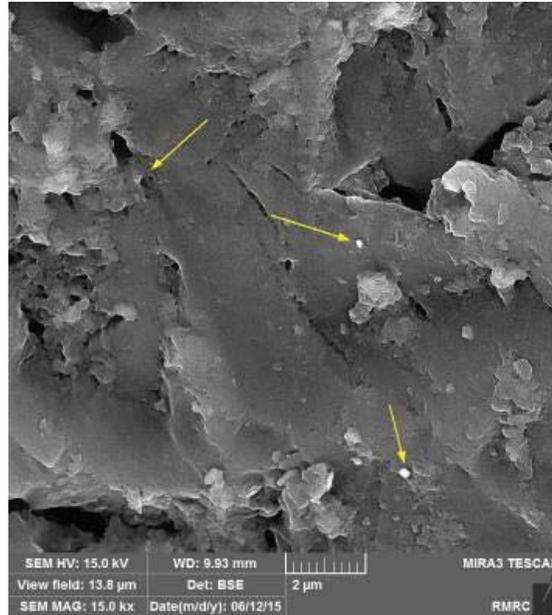


Figure 6: SEM image for distribution of nano-particles in epoxy matrix.

(25×250mm) of each sample were obtained, using a water jet cutter according to ASTM D3039 standard. To obtain mechanical specifications, different pieces underwent tensile testing. Results are presented in Table 5. Tests were conducted by a hydraulic machine (WDW-300E; load accuracy: 0.001mm; load cell: 15 kN; UTM-300E controlling system).

Table 5: Mechanical properties of nano-composite samples (ASTM D3039).

Sample	Wt.%	Tensile Strength (MPa)	Elongation %	Young's Modulus (GPa)
1	0	279.7	0.03	9.48
2	0.2	289.4	0.031	9.33
3	0.43	312.7	0.03	10.42
4	1	308.9	0.033	9.36
5	2.1	301.2	0.027	11.36
6	4.1	297	0.027	11

### 2.3. Single-Lap Adhesive-Bonded Joint

In this study, Araldite 2015 (Huntsman) was used to bond nano-composite samples. Physical specifications of this adhesive are presented in Table 6.

Table 6: Mechanical and physical properties of Araldite 2015. (Data Sheet).

Tensile modulus (GPa)	2
Tensile strength at 23 C (ISO 527)	30 MPa
Specific gravity	1.4
Viscosity at 25°C (Pas)	thixotropic

Nanoparticle-free composite samples were fabricated using VARTM, and cut according to ASTM D5868-01 standard (Fig.7). Then, nanoparticles with 0, 0.25, 0.5, 0.61, 0.74, 0.83, 1, and 1.23wt% were added to the adhesive and eight single-lap adhesive-bonded joints with the same overlap length (30mm) and thickness (0.5mm) were fabricated.

As mentioned, Araldite 2015 (Huntsman) was used to bond Nano-composite samples. Due to the high viscosity of this adhesive, the addition of nanoparticles to it via magnetic stirrer and ultrasonic device was not possible. In some studies, the addition of nanoparticles to adhesive was done by the dispersion of nanoparticles in acetone or alcohol, using aforementioned methods. Then, the mixture was added to the adhesive and stirred mechanically. Due to the volatility of the employed solvent, it evaporated in a short time, leaving nanoparticles dispersed in the adhesive. As a fundamental shortcoming, this method changes adhesive characteristics because of the adhesive-solvent reaction. In this study, the addition of alcohol changed the quintessential features of the adhesive. Therefore, following technique was used to add the nanoparticles to this adhesive with high viscosity. Araldite 2015 is comprised of two components and cured in a short time after being mixed with nanoparticles. Therefore, that portion with lower viscosity rate was selected and nanoparticles were mixed with it, using a mini-stirrer (comprising of a DC motor and a miniature arm) for 30 minutes. It was then mixed with the second part of the mixture for bonding. Figure 8 presents the single-lap bonded joints

with composite adhesive and nano-adhesive. After the implementation of the axial tensile test with WDW-300E, the maximum load-bearing capacity (kN) of each sample was obtained (Table 7).

### 3. Results and Discussion

In this study, the strength of the single-lap adhesive-bonded joint in nano-adhesive samples under tensile loading, as well as the mechanical behavior of composite sample in the presence of alumina nanoparticles were evaluated.

#### 3.1. Single Lap Joint

The below figure shows the experimental results for the maximum load-bearing capacity of the single-lap adhesive-bonded joint based on adhesive nanoparticles. Adding nanoparticles to the adhesive decreases the joint's load-bearing capacity from 4.89kN to 4.49; whereas, adding nanoparticles (up to 0.74wt%) increases the load-bearing capacity by 14.6% as compared to the initial sample (nanoparticle-free adhesive). Adding more nanoparticles (>0.74wt%) decreases the load-bearing capacity. This is because of an increase in nanoparticle content and greater probability of nanoparticle agglomeration in the adhesive, and thus deeper stress concentration in agglomerated points.

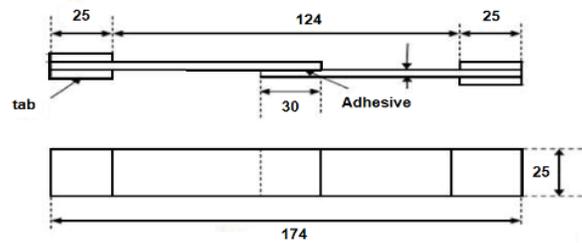


Figure 7: Dimensions of tensile test specimen as ASTM D 5868-01 (mm).



Figure 8: Single lap samples.

Table 7: Experimental results for nano-particle, adding to Araldite adhesive.

Sample	Wt.% of Nano-particle	Ultimate Force (kN)
1	0	4.89
2	0.25	4.77
3	0.5	4.49
4	0.61	5.07
5	0.74	5.73
6	0.83	5.42
7	1	5.35
8	1.23	5.1

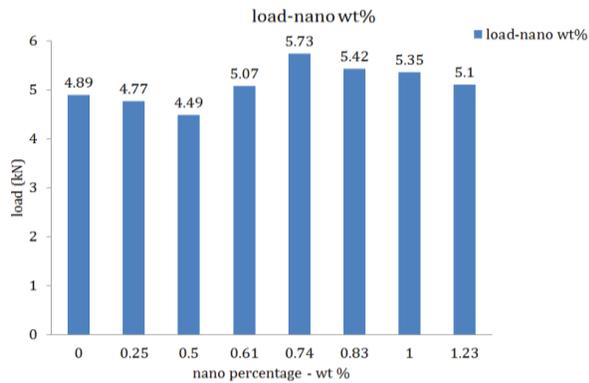


Figure 9: Experimental results for single lap joints strength. Nano-composite Samples

Figure 10 presents stress-strain curves for nano-composite samples with different percentages of alumina nanoparticles. Figure 11 presents ultimate strength, failure strain, toughness, and Young's modulus for nano-composites.

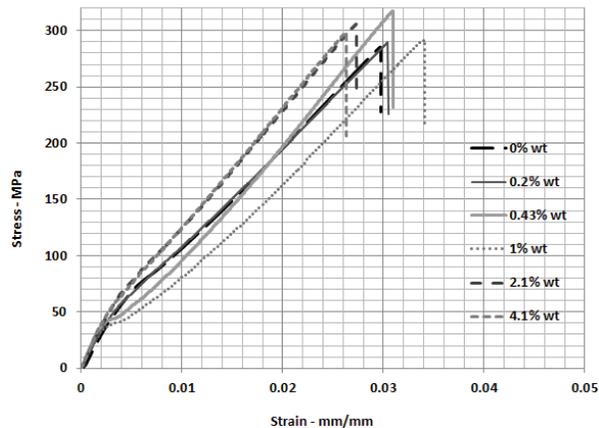


Figure 10: Stress-Strain curves for nano-composites.

The elastic modulus in a sample containing alumina nanoparticles (2.1 wt%) increased by 16.5% as compared to the primary sample (without nanoparticles). Adding alumina nanoparticles with 0.2 wt% and 1 wt%, slightly decreases elasticity modulus by less than 2%. The increased elastic modulus in weight ratios greater than 1% is due to better dispersion of nanoparticles within the polymer matrix. Increased nanoparticle content, along with good dispersion of them improves the interfacial bond and adhesion of nanoparticles and polymer matrix. This phenomenon limits the movement of polymer chains during loading, resulting in an increased elastic modulus of nano-composite [20].

Increased nanoparticle content to 0.43 wt% of the total weight of composite improves tensile strength by

11.7%; however, a higher ratio of alumina nanoparticles decreases tensile strength. The presence of well-dispersed nanoparticles in polymer matrix decreases polymer matrix movement on the nanoparticle-matrix interface and glass fibers-polymer matrix interface. This reduction improves stress transmission to the glass fibers, which consequently increases the strength of nano-composites. When alumina nanoparticle content exceeds a certain level (0.43 wt%), nano-composite strength decreases with increasing the amount of nanoparticles. This can be attributed to the accumulation of nanoparticles with increasing nanoparticle content, which causes crack growth and stress concentration in nano-composite and material failure under lower stress.

Much change was not observed in failure strain with increasing nanoparticle content to 1 wt%; however, in high weight ratios, nanoparticles prevent the formation of a continuous network in a polymer matrix and cause a 10% reduction in failure strain rate.

The concurrent effect of elastic modulus, tensile strength, and failure strain of composites can be seen in the surface area under the stress-strain curve (or material toughness). The greatest increase in toughness (23.5%) was observed in the sample with alumina nanoparticle content of 1 wt%. The samples with 1.2 wt% and 1.4 wt% had the equal toughness with a 3% reduction as compared to the primary samples.

#### 4. Conclusion

In this study, the effect of adding Alpha-alumina nanoparticles to glass/epoxy composites with woven fibers on mechanical properties was investigated. In addition, the effect of adding nanoparticles to Araldite 2015 on the strength of a single-lap joint of composite samples was studied. In general, experimental results from the axial tensile test and the formation of the force-displacement curves for each sample show that adding a small amount of nanoparticles to adhesive increases the maximum load-bearing capacity by 14%. The effect of adding Alpha-alumina nanoparticles with different weight ratios (0, 0.2, 0.43, 1, 2.1, and 4.1 wt%) to glass/epoxy composites suggests that the maximum values of the ultimate strength, failure strain, toughness, and Young's modulus belong to the samples with the weight ratios of 0.43, 1, 1, and 2.1%, respectively. Alumina nanoparticles can effectively improve the mechanical performances of Araldite adhesives and glass/epoxy composites, by increasing their mechanical properties such as ultimate strength, toughness etc.

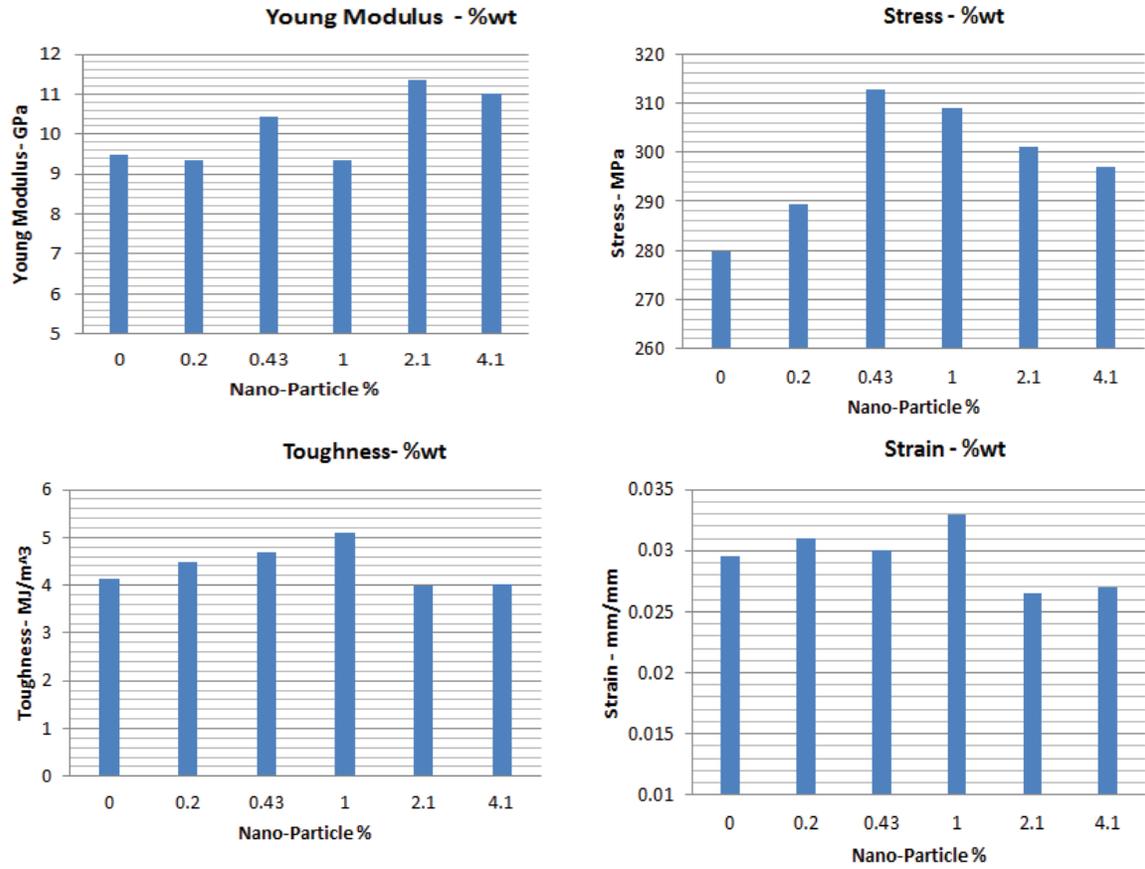


Figure 11: Mechanical properties of nano-composites.

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