1 Introduction:

Adhesively bonded joints (ABJs) are increasingly used in automotive, marine, offshore, and oil and gas industries, to mate both metallic and fiber-reinforced polymer composite (FRP) structural components. Adhesively bonded FRPs offer several advantages such as high strength and stiffness to weight ratios, good fatigue and corrosion resistance, controllable damage tolerance, and high energy absorption capability, which make them more efficient compared to other type of mechanical fasteners [1-3]. Crashworthiness, improved damage tolerance, energy absorption capability, and safety requirements are important factors for the design of lightweight composite structures, especially in automotive and marine vessel applications. However, a major concern in the use of adhesives in those applications has been the lack of adequate database in regards to performance of ABJ at high rates of strain and impact loads. Therefore, mechanical characterization of ABJs at high loading rates is vital for achieving reliable designs [4].

The overall goal of our study is to develop a relatively inexpensive and strong adhesive for common engineering applications. Therefore, various aspects of ABJ are being investigated. In this paper, the effect of high strain rate on the mechanical response of adhesively bonded single lap joint of composite adherends under impact at 2.04E+5 mm/min is investigated. Unidirectional E-glass fiber reinforced epoxy laminate was used as the adherends. The high strain rate tests were accomplished using a modified instrumented pendulum, equipped with a specially designed impact tension apparatus.

The results indicated that ABJs tested under highest loading rate exhibited increased stiffness and strength. Strain rate dependent properties derived from the experimental data will be used in the near future in conjunction with finite element analysis to conduct parametric study and optimize the performance of such joints. The observed failure mechanisms deduced from scan electron microscopic study of the failed specimens will also be presented.

2 Experimental Plan

2.1 Fixture design

The tensile impact fixture’s elements are illustrated separately, as in whole in Fig. 1.
The actual fixture is shown in Fig. 2. As can be seen, the rod used to transfer the impact load is aligned within several roller-bearings. To reduce the errors related to undesirable friction during the impact test, the height of each bolt was adjusted.

A jig was also designed and fabricated for making ABJs consistent and accurately aligned, as shown in Fig 3. Shims were used to obtain the required thickness of the bond line (i.e., 0.25 mm). This thickness was selected based on ASTM D5868-01 standards (2001).

2.2 Specimen preparation

2.2.1 Q-Cell reinforced adhesive

To prepare single lap joints (SLJs), a commonly used thermoset epoxy resin (i.e., West System’s 105 resin and 206 hardener (Bay City, MI)) was used as the baseline adhesive due to their common use and relatively low cost. However, 105 resin’s viscosity is very low, and in order to be able to form practical bonded joints, it has to be thickened (to become paste-like); therefore, Q-Cell filler (obtained from Rayplex, Toronto, ON) can be used to thicken the resin. Q-Cell is inexpensive and lightweight filler that consists of white hollow inorganic microspheres with low bulk density. It is commonly added to resins with the ratios of 0.5% -10% (by weight). It is of course well known that the inclusion of the filler would degrade resin’s mechanical properties. However, no factual information or data on the actual level of degradation resulting from the addition of Q-Cell filler in resin could be obtained from either the vendor or open literature. Therefore, in order to establish the degree of degradation in the resin due to addition of Q-Cell filler, three concentrations of the filler (i.e., 0%, 5%, and 10% by weight Q-Cell) in the resin were investigated. These ratios were selected based on the ease of applying the resin/adhesive in practical applications on vertical surfaces.
2.2.2 Nano particles reinforced adhesive

Since the addition of the commonly used fillers like Q-cell is known to degrade resins/adhesives’ mechanical properties, another filler type material was used with the aim of actually enhancing resin’s mechanical properties. For that, as a means to economically enhance the mechanical properties of the thermoset resin/adhesive (in our case West System 105), attempts were made to use various forms of nano-particles as the filler. However, the uniform dispersion of nanocarbon in resin is quite challenging, time consuming and thus an added cost. The dispersion directly governs the mechanical properties of the adhesive, but more so, GNP agglomeration causes severe statistical inconsistencies in the strength and performance of the adhesive. Therefore, a mechanical stirrer and three-roll mill machine was used to disperse the nano-particles in resin uniformly.

To enhance dispersion, each roller should revolve with a set constant speed. In this study, the roller speed and calendaring frequency were set to maximum speed of the machine (i.e., 174 RPM). To maximize the quality of dispersion the calendaring was conducted seven times. After each calendering, the quality of dispersion was monitored by taking a small sample and examining nano-particles dispersion using a digital microscope, thereby avoiding unwanted agglomerations.

Three different types of nano-particles were selected to be dispersed into the epoxy resin.

a. Graphene Nano Platelet (GNP-M-25) with average diameter of 25 µm and 6 nm thickness and surface area of 100 m²/g obtained from XG Science Ltd. (Lansing, MI).

b. Multi Walled Carbon Nanotubes (MWCNTs) with outer diameter of 5 to 15 nm, and more than 95% purity (obtained from the US Research Nanomaterials, Inc., Houston, TX).

c. Graphitized Carbon Nano Fibers (CNF) with outer diameter of 200 to 600 nm, and more than 99.9 % purity (obtained from the US Research Nanomaterials, Inc., Houston, TX).

The nano-particles were first distributed in the resin using a mechanical stirrer set at speed of 2000 rpm for 10 min.

Next step was calendering the nano-particles-resin slurry using the three roll mill. The roller gap was set at 20 um using a filler gauge, for 0.5% (by weight) concentration of CNF, MWCNT, GNP (see Fig. 4).

As stated, after each calendering, the quality of dispersion was monitored by taking a small sample and examining nano-particles dispersion using a digital microscope (see Fig. 5).
minutes. The mixture was degassed under 28” Hg vacuum for 2 to 3 minutes. Degassing duration depends on the gel time of the resin. After degassing, the mixture was poured in appropriately shaped molds, and let cure for 12 hours at room temperature. Typical final products in the form of dog-bone coupons, with dimensions as per ASTM D638-94B, are shown in Fig 6.

![Fig. 6 Representative tensile coupons neat, Q-Cell filler, and nano-particle reinforced resins as per ASTM D638-94B (Dimensions in mm).](image)

2.2.3 Adherends preparation
To manufacture the adherends, laminate plates with dimension of 350 mm ×350 mm were fabricated by the vacuum resin injection technique (VRIT). Appropriate size coupons were then extracted from the plates, followed by the surface preparation process applied to the bonding regions. To meet ASTM D5868-01 requirements, the glass/epoxy plate was made from 12 unidirectional plies. Unidirectional E-glass fabric and Huntsman’s Araldite LY 564 epoxy resin with Aradure 2954 hardener (West Point, GA) were used to fabricate the laminate plates.

2.2.4 Single Lap Joint preparation
The single lap joints (SLJs) were prepared from glass/epoxy laminate adherends, and the adhesive containing different amounts of micro and nano-particles, using the described jig. A typical SLJ specimen is illustrated in Fig. 7.

![Fig. 7 Typical Glass/Epoxy single lap joint specimen](image)

Depending on the nature of each tests, and to ensure a concentric load pass through adhesive FRP tabs with appropriate thickness were affixed to adherends’ ends (see Fig. 8).

![Fig. 8 Typical single lap joint specimens (dimensions in mm).](image)

2.3 Characterization of the mechanical properties of neat adhesives
The prepared dog-bone shaped specimens were tested in tension using an Instron servo-hydraulic universal test machine equipped with 8500+ electronics. The specimens were subjected to displacement controlled tensile loading as per ASTM D638, to establish the stress-strain curve of each adhesive. An Instron extensometer was used to record the gauge-length displacement (hence, the strain) of the specimens. Tensile tests were performed at room temperature at cross head speeds of 1.5 and 3 mm/min (for the static, and quasi static tests), and 2.04 E+5 mm/min (for the impact tests), based on ASTM D 897, and ASTM D 950, respectively. [19-21]. Details of the impact (high-strain) loading will be given in section 4.1.
Using the recorded load and gauge length displacement, the stress-strain curve of each adhesive was constructed and their elastic modulus was assessed.

3 Experimental investigation of ABJs
The experimental part consists of two phases, which will be discussed in below.

3.1 Static and Quasi Static tests:
To perform the static and quasi static tests on the ABJs, the aforementioned loading rates were used. The applied load was recorded directly through the Instron machine electronics and indirectly using the National Instrument DAQ system equipped with Lab View software. Gauge length displacement was also captured using a laser extensometer, through the same DAQ system.

3.2 Impact (high strain rate) tests:
In this category of tests, the applied load was measured using a PCB dynamic load cell (Depew, NY), positioned at the tip of the tensile carriage fixture (see Fig. 10(b)). The relative displacement of the overlap region was captured by a dynamic linear variable differential transducer (DLVDT) (Data Instruments, Acton, MA) (see Fig. 10(c)).
Following the main objective of this research, in an attempt to produce a strong and viscous adhesive from a commonly used and relatively inexpensive room-cured resin, the Q-Cell filler and various nano-carbons were added to the neat resin (West System 105). Result of the tensile tests showed that although adding Q-Cell filler increased adhesive’s workability (viscosity), however the mechanical properties of the adhesive was degraded. The observed decrease in Q-Cell added adhesive renders the adhesive unsuitable for practical bonding.

In contrast to the above, very good results could be obtained when the least expensive nano-carbon (i.e. graphene nano-platelets) were added to the resin. Various researchers have also observed that addition of Carbon Nano Fibers (CNF), or Multi Walled Carbon Nano Tubes (MWCNTs) to resins produced enhancement of resin’s mechanical properties and fracture toughness [9, 12]. However, very limited data exists for the level of enhancement that could be expected by addition of GNPs to resins. As stated, GNPs are considerably less expensive than CNFs and MWCNTs. The results indicate that not only does the inclusion of GNP improves the mechanical properties of this adhesive, but it also enhances resin’s viscosity, hence making it suitable for use as an adhesive, especially suitable for marine and other applications. In fact, the shear strength of the GNP reinforced adhesive was improved by 33% when SLJs were tested at high rates of loading, and the increase was approximately 20% when SLJs were subjected to quasi-static loading rate (See Fig. 11).

Fig. 12 illustrates the SEM images of the neat resin as well as the GNP and MWCNT reinforced resins. As can be seen in Fig. 12 (b), the GNPs are piled on top of one another like a colony. Due to higher surface aspect ratio of the GNPs, they offer added strength to micro-cracking, and in the event of cracking, they effectively bridge the micro-cracks. In contrasts, MWCNTs dispersed in the resin are filamentous and furcated as can be seen in Fig. 12(c).
4.2 Influence of loading rate on the mechanical response of nano-particle reinforced SLJs

As it was discussed in previous section, the results showed that inclusion of the GNP in resin could significantly improve resin’s stiffness and strength. The average ultimate shear strength of SLJs was increased as high as 33% when SLJs were subjected to the high loading rates, and about 21% under quasi-static rates. Fig. 13 shows that increase in the strain rate affects the average ultimate shear stress of the adhesive in a nonlinear manner.
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References: