

Non Destructive Evaluation of Degradation in Bond Line of Glass Fiber Reinforced Polymer Composite Adhesive Lap Joints

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Abstract

A critical issue in using adhesives for structural joints is the assessment of integrity and reliability of these joints in service. One of the factors limiting the wide spread use of adhesives for structural bonding in industries is the lack of reliable non destructive testing methods to evaluate the bonded joints. Joining of fiber reinforced polymer (GFRP) composite materials by adhesive bonding has to take into account factors such as the adopted bonding process, the interface properties and the control of the adhesive bond line thickness. In this paper, results of experimental investigations on the non destructive evaluation (NDE) of single lap bonded joints made of glass fiber reinforced epoxy matrix composite laminates are presented. Unidirectional glass fiber reinforced plastic substrates were joined using a two part epoxy adhesive system. Specimens with varied bond quality were prepared adding different proportions of poly vinyl alcohol (PVA) to the epoxy hardener mixture. The bonded area of the specimen was subjected to ultrasonic inspection and x-ray radiography. The joints were subsequently loaded till failure to determine their bond strength. Results obtained show a correlation between the amplitude of reflected ultrasonic waves from different interfaces and the bond strength.

1. INTRODUCTION

It is well known that the strength of many engineering structures assembled using adhesives depend critically on the quality of bonding between structural components. Adhesively bonded joints provide many advantages over conventional joints like welding, riveting etc., in terms of uniform stress distribution and reduced stress concentration. Further, adhesive bonding also gives a smooth appearance as there are no protruding fasteners such as screws, rivets and spot-welding marks [1]. Factors determining the integrity of an adhesive bond are, selection of adhesive, joint design, preparation of the bonding surfaces, strict quality control in production and condition monitoring in service [2]. Often these joints are exposed to high temperature and humidity resulting in degradation of the bond line. Defects can also creep into the joined area during its fabrication [3]. Literature shows that work has been carried out previously on non destructive testing and evaluation of adhesive joints [4-15]. The main objective of the current set of investigations presented here was to non destructively evaluate degradation in the adhesive joint which was induced by adding different proportions of PVA into the epoxy hardener mix. The joints were subjected ultrasonic and digital x-ray radiography inspection to obtain a NDE parameter that can reflect the degradation induced. Destructive shear tests were performed to determine the bond strength of these sets of GFRP specimens. Details of the experimental investigations and the results obtained are discussed.

2. MATERIALS AND SAMPLES

Specimens were prepared according ASTM D 5868 standard for lap shear adhesion for fiber reinforced plastic bonding. Single lap shear joint specimens (Figure 1a) were used in the present work. Composite strips were prepared with unidirectional (UD) E-glass fabric as reinforcement and thermoset epoxy

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resin LY556/HY 951 as the matrix. The laminate was fabricated and cured at room temperature with 14 layers of UD glass fabric all laid up in the 0° direction. Spacers were used to maintain a uniform thickness of 2.54 mm.

Surface preparation is one of the basic requirements for a good bond in terms of roughness and cleanliness of the adherend surfaces which can be obtained using emery paper buffing. The roughened and cleaned surfaces facilitate better mechanical interlocking between adhesive and adherend. Thus, surface preparation was carried out according to ASTM D 2093 standard for surface preparation of plastics, the adherend surfaces were buffed using 100 grit size sandpaper, and subsequently cleaned with acetone and dried at room temperature. Bonding was accomplished using a two part epoxy adhesive system -Araldite AV 138M/Hardener HV 998, commonly used in bonding plastics and composites. Adhesive to hardener weight ratio was maintained as 100:40 as per the recommendation of the manufacturer. An area of 25.4mm x 25.4mm was bonded and the bond line thickness was maintained at 0.76mm using a specially designed mold to serve the purpose (Figure 1b). The mold also helps in maintaining a proper alignment of the GFRP adherend strips during the process of curing. The quality of adhesive was degraded using different quantities of PVA which affects the polymerization process in epoxy resin, leading to weak adhesion and cohesion. Five sets of specimens, six samples in each set were used for the experimental study. The set without any PVA content was considered as healthy denoted by the symbol 'H', the remaining samples were represented by the symbol 'P'. The PVA content was varied from 10% to 40% by total weight of the resin system with an increment of 10% in each set. The adhesive in the joint was cured at room temperature for 24 hours at atmospheric pressure. End tabs were bonded to the specimens to facilitate the shear strength tests subsequent to Non Destructive Testing and Evaluation.

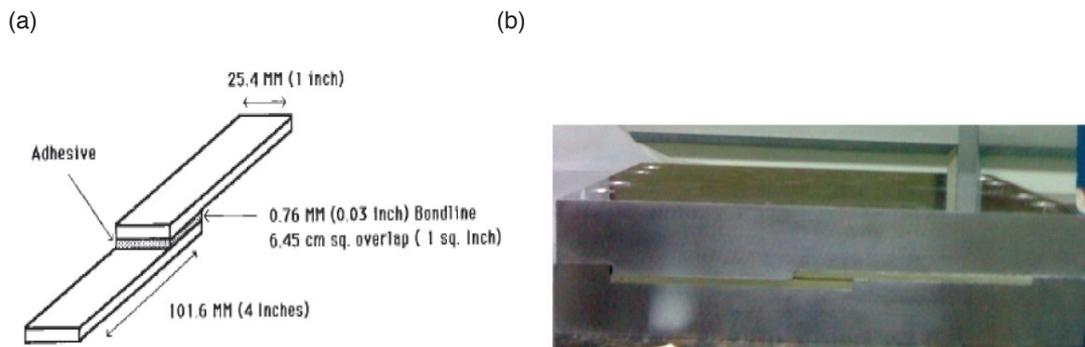


Figure 1. (a) Composite lap shear joint (b) GFRP adherends aligned within the mold

3. EXPERIMENTAL DETAILS

Of different Non Destructive Testing (NDT) tools, ultrasonic methods have generally been regarded as potentially the most useful for NDE of multilayered composite structures and adhesive joints [16-20]. In the current study, the joints were tested using time domain ultrasonic testing methods like pulse echo and through transmission to evaluate their bond quality.

3.1 Ultrasonic Through Transmission Method

Inspection of the bonded region using a focused probe to produce a C-scan image is based on the fact that, greater the polymerization of the adhesive more transparent it is to the ultrasonic wave. The arrangement of through transmission scanning system is illustrated in figure 2. As shown, the system has two coaxially aligned 5MHz focused probes with a focal length of 76mm in water and a diameter of 13mm. One of the probes (pulsar) generate the ultrasonic energy which passes through the specimen, while the other receives (receiver) the transmitted energy. A rectangular pulse, 72ns wide with a pulse repetition frequency of 1000Hz and amplitude of 350 volts was used to excite the pulsar, while the receiver was provided with a gain of 8dB. The entire arrangement of probes and the specimen was immersed in water which acts as a medium for energy transfer. The edges of the joints were completely sealed to prevent the ingress of water into the joint area.



Figure 2. Ultrasonic through transmission scanning set up

The amplitude variation of received ultrasonic signal from the bonded area was mapped to get a C-scan which gives an indication of the adhesive quality in the bond line. The average amplitude of the C-scan image was computed using a simple MATLAB program. To accomplish this, images which were originally with 256 colors were converted to grey scale, as shown in figure 3. White color represents maximum amplitude and its value is taken as 1 where as the black color has the minimum amplitude and its value is taken as 0, all the other shades in the grey scale fall within this range and represent variation in the received signal amplitude.

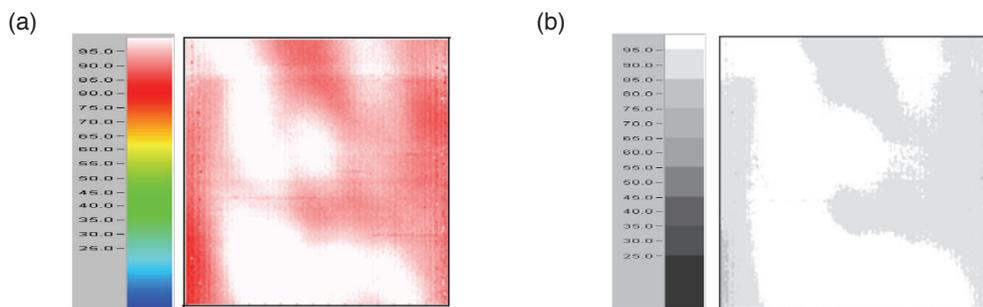


Figure 3. Conversion of color image to grey scale.

Figure 4 shows the grey scale images for a healthy (a) sample and those with different percentages of Poly vinyl Alcohol(b-e). Of these, signal amplitude for the healthy sample is higher which decreases with increasing amounts of PVA.

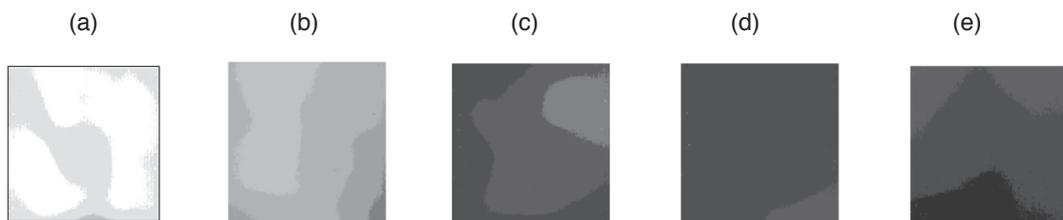


Figure 4. Through transmission C-scans obtained for different specimens (a) Healthy (b) 10 % PVA (c) 20% PVA (d) 30%PVA (e) 40% PVA.

During the curing process the adhesive passes from a semi solid to solid state by the formation of intermolecular bonds with creation of the branched chains of molecules which give the adhesive its strength. Thus, more developed the polymerization process, lesser is the attenuation of the ultrasonic wave passing through what is now a more elastic medium with lower energy dispersion. Accordingly, the amount of ultrasonic energy passing through the healthy adhesive joint is much higher owing to less attenuation. On the contrary, the joints with added PVA suffer from hampered polymerization and leads to more attenuation of ultrasonic energy. An area of 12mm x 12mm covering the central portion of the

bonded region was considered during the calculation of average amplitude to eliminate the distortion effect at the edges of adhesive joints. These amplitude values are plotted against different PVA percentages as shown in figure 5. The maximum amplitude in case of a healthy sample reaches a value close to 1 where as its average amplitude is about 0.78. On the other hand, the sample with 40 percent PVA has an average amplitude of 0.33. There is a sudden decrease in the amplitude value for 10 percent PVA, but the decline becomes smooth and gradual thereafter. Though there is a certain scatter in the receiver signal amplitude obtained which is expected in any experimental investigations, the results show a clear indication of correlation between the transmitted ultrasonic signal amplitude and the degradation in the adhesive layer.

Through-transmission ultrasonic technique promises to be a simple and robust test for evaluation of quality in adhesive joints, but has limited application since this approach requires both sides of the joints accessible which may be rare in most practical cases.

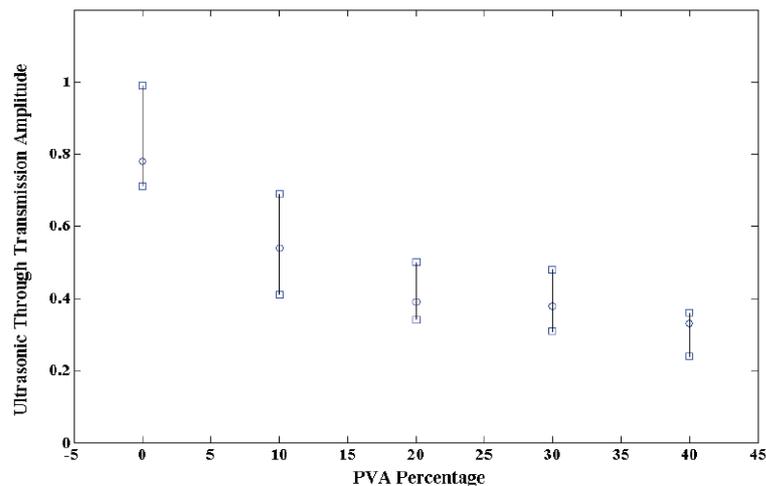


Figure 5. Variation of transmitted signal amplitude with different PVA percentages

3.2 Ultrasonic Pulse Echo Method

Unlike through transmission technique, the pulse echo method uses a single transducer to transmit and receive the ultrasonic energy. The plane of adhesive joint is maintained normal to the 5 MHz focused transducer surface. The incident ultrasonic waves get reflected from each of the interfaces as illustrated in figure 6. The corresponding echoes were identified using the velocity of sound in both GFRP and the

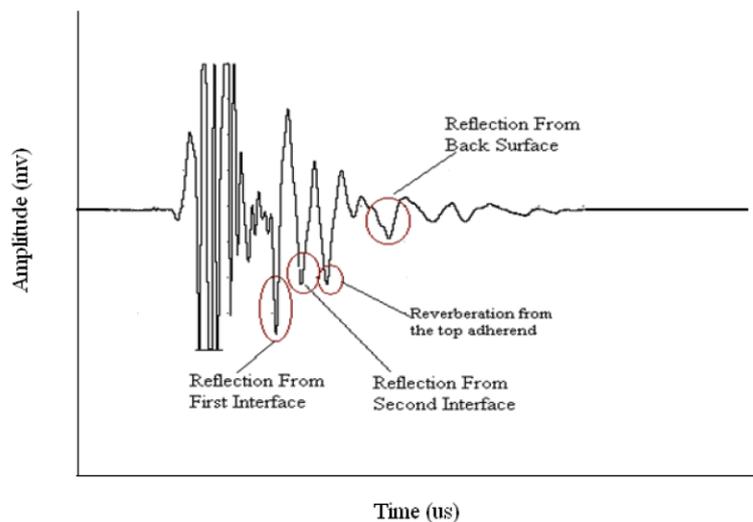


Figure 6. Received signal comprising of reflections from different interfaces.

epoxy resin. Since the thickness of the substrate and the adhesive layer are known, the time of travel for the sound wave can be calculated using the relation $T=2S/V$, where, 'T' is the time of travel, 'S' denotes the thickness of the sample and 'V' is the velocity of sound in the traveling medium.

The separation of the reflected signals in the time domain allows us to identify echoes from each interface and to measure reflection coefficient at those interfaces. Ultrasonic C-scans were obtained for each sample at different interfaces by setting the gate on the time axis at the corresponding echo. For example, C-scan images for the first interface were obtained by confining the gate settings to the reflection from first interface and mapping the amplitude variation of the same over the area. The C-scan images for the first interface are shown in figure 7.

These images reveal that the amplitude of reflected signal from the first interface for specimens with higher degradation is higher as compared to their healthier counterparts. In a two part epoxy resin system when a hardener (HV 998) is added to epoxy resin (AV138M), polymerization of the epoxy monomers take place due to chain reaction. In the absence of any contaminant this process will be complete and the cured adhesive system will have better cohesive and adhesive properties.

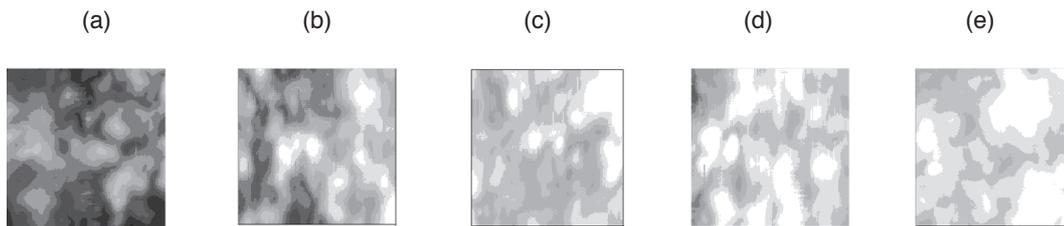


Figure 7. First interface C-Scans for different Specimens (a) Healthy (b) 10 % PVA (c) 20% PVA (d) 30%PVA (e) 40% PVA.

Addition of PVA hampers the polymerization process and creates a weaker interface between the substrate and the adhesive. The amplitude of reflection from an interface depends on the acoustic impedance mismatch between the two mediums. As the amount of PVA is increased, the mismatch becomes more predominant and thus higher amplitude reflections are seen from the first interface of PVA added samples.



Figure 8. Bulk adhesive samples prepared to measure velocity and density variation

To find out the effect of addition of PVA to the adhesive in terms of its acoustic properties, sets of bulk polymer samples with varied PVA content were prepared. Five different samples of size 25 x 25 x 10 mm were fabricated (Figure. 8). One of the samples did not have any PVA content and was presumed as healthy whereas the other samples had the PVA content varying between 10% to 40% similar to that used in adhesive joints. The density and velocity of ultrasonic longitudinal wave for each adhesive sample was obtained using which the corresponding acoustic impedances were calculated from the relation $Z=\rho V$, where, ' ρ ' is the density and 'V' is the velocity of sound in the corresponding medium. The values obtained are tabulated in table 1.

It can be observed from the table that addition of PVA decreases both density and velocity of sound in the epoxy resin. Accordingly, the acoustic impedance also decreases with an increase in the amount of PVA. The reflection and transmission coefficients for the different cases are calculated using the following equations.

$$R = \frac{Z_2 - Z_1}{Z_2 + Z_1} \quad (1)$$

Table 1. Density, velocity and acoustic impedance values for different bulk adhesive samples

| Sample | Density (kg/m ³) | Velocity (m/s) | Acoustic impedance, Z (kg/m ² -sec) |
|---------------|------------------------------|----------------|--|
| GFRP adherend | 1762 | 3200 | 5.6384 x 10 ⁶ |
| Epoxy +0% PVA | 1548 | 2480 | 3.8390 x 10 ⁶ |
| Epoxy+10% PVA | 1429 | 2342 | 3.3467 x 10 ⁶ |
| Epoxy+20% PVA | 1322 | 2223 | 2.9388 x 10 ⁶ |
| Epoxy+30%PVA | 1213 | 2065 | 2.5048 x 10 ⁶ |
| Epoxy+40%PVA | 1107 | 1914 | 2.1187 x 10 ⁶ |

$$T = \frac{2Z_2}{Z_2 + Z_1} \quad (2)$$

These coefficients for different adhesive joints are as shown in the table below. The addition of PVA increases the impedance mismatch between the GFRP substrate and the epoxy adhesive resulting in higher value of reflection coefficient. The acoustic impedance is a characteristic property of the material being studied and has a bearing on material stiffness. In general, the stiffer the material, higher the ultrasonic wave velocity in that medium. This is evident from the experimental results as a reduction in wave velocity can be observed with addition of PVA.

Table 2. Reflection and transmission coefficients for different adhesive joints

| Adhesive lap shear joint type | Reflection coefficient (R) | Transmission coefficient (T) |
|-------------------------------|----------------------------|------------------------------|
| GFRP + pure epoxy | 0.189 | 0.811 |
| GFRP + epoxy + 10% PVA | 0.255 | 0.745 |
| GFRP + epoxy + 20% PVA | 0.315 | 0.685 |
| GFRP + epoxy + 30% PVA | 0.385 | 0.615 |
| GFRP + epoxy + 40% PVA | 0.454 | 0.546 |

The variation of amplitude of reflected signal from the first interface between GFRP substrate and epoxy adhesive is as shown in Figure 9. The figure also shows variation of the theoretically calculated reflection coefficient plotted using the values of Table 2. While the experimental obtained values follow a trend similar to theoretically calculated, the amplitudes of reflection obtained experimentally is higher compared to theoretical values. This perhaps implies there is an additional factor other than the impedance mismatch which governs the reflection amplitudes from an interface of an adhesive joint. Attempts have been made to non destructively measure the interfacial properties and their influence on the joint quality by Rokhlin et al [21-26].

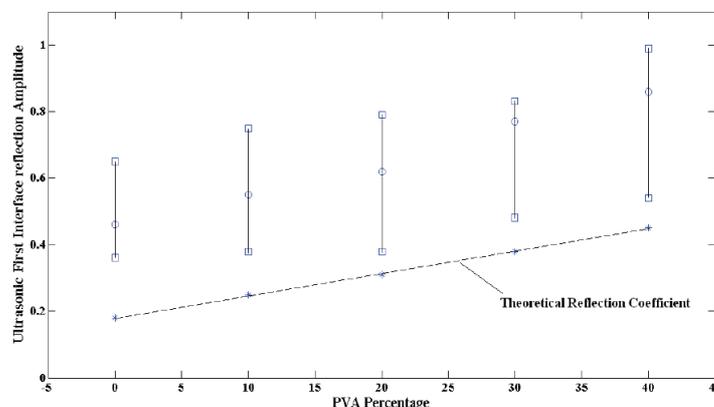


Figure 9. Variation of amplitude of reflected signal from first interface with different PVA percentages

The impedance mismatch between the adherend and adhesive, coupled with high attenuation in the adhesive layer results in smaller amplitude second interface reflections, which can make them difficult to detect [27]. However, since the bond-line used in the present work was sufficiently thick (0.76 mm) the reflection from second interface was obtained without much difficulty. The amount of energy passing through the bond line and reaching the second interface is expected to be more in case of a healthy sample. On the other hand in samples with added PVA significant part of the incident energy gets reflected at the first interface and hence second interface reflections will have lower amplitude. Figure 10 shows the C-scan images obtained for second interface. Variation of amplitude of this reflected signal with different PVA percentages is presented in Figure 11.

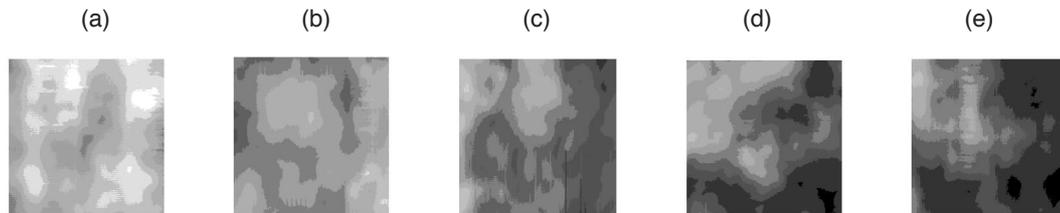


Figure 10. Second interface C-Scans for different Specimens (a) Healthy (b) 10 % PVA (c) 20% PVA (d) 30% PVA (e) 40% PVA.

The incident ultrasonic energy into the bonded lap-joint area goes through partial reflection and partial transmission at each of the interfaces before reaching the back surface of the joint. Hence, properties and condition of each layer of the bonded joint have their effect on the amplitude of the back wall echo.

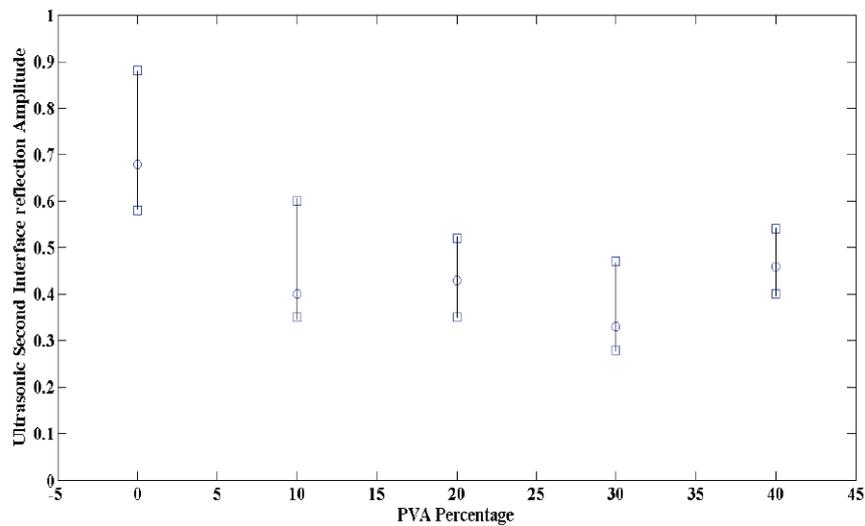


Figure 11. Amplitude of reflected signal from second interface versus PVA percentage

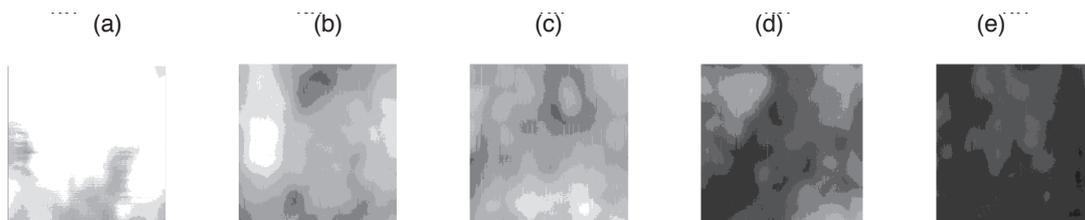


Figure 12. Back wall echo C-Scans for different Specimens (a) Healthy (b) 10 % PVA

Thus, the amount of ultrasonic energy reaching the back surface in a healthy joint is certainly higher than in that of PVA samples due to better polymerization and weak interface reflections. Hence, the

corresponding amplitude of reflection from back wall is higher as shown by the C-scan images for different samples presented in Figure 12. Figure 13 shows the plot of this amplitude versus PVA content.

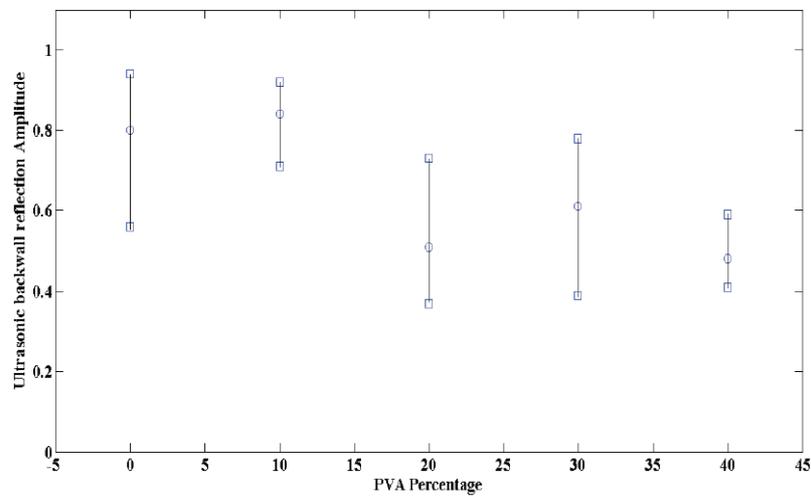


Figure 13. Variation of Amplitude of reflected signal from bottom surface versus PVA percentage

4. DIGITAL X-RAY RADIOGRAPHY

The digital x-ray radiography setup utilized consisted of a portable x-ray source and silicon based flat panel detector. The adhesive joint to be tested is placed in between the source and the detector. Though conventional Radiography is known to be insensitive to disbonds and high absorption in adherends can mask defects in metal to metal joints [28-30], the absorption in case of a glass fiber reinforced composite substrate is much lesser compared to metals.

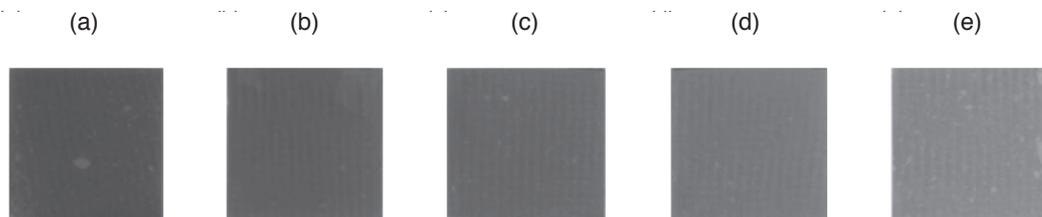


Figure 14. X-ray radiography images of different specimens (a) Healthy (b) 10 % PVA (c) 20% PVA (d) 30%PVA (e) 40% PVA.

The absorption of x-rays in a material depends mainly on three factors, atomic number, density and thickness. Since thickness of sets of samples is maintained constant, intensity of X-rays transmitted across a bonded joint depends on density variation of adhesive in the bond line. Density of adhesive in a healthy sample is higher compared to that in a PVA sample, leading to more absorption of X-rays. Accordingly, the images for healthy samples appear darker as can be observed from Figure 14.

The variation of average X-ray intensity transmitted across the bonded joint with the amount of PVA is shown in Figure 15. The dotted line is the linear best fit which shows the average intensity of the images increasing with the increase in the amount of PVA. However, this approach of x-ray inspection gives a cumulative effect of all the layers involved and overall degradation in the adhesive area but fails to identify contributions of individual layers or parameters.

5. TESTING OF ADHESIVE JOINTS FOR DETERMINING BOND STRENGTH

The adhesive lap-joint specimens were tested according to ASTM D 5868 standard for bond strength determination in a computer controlled test system (Fig. 16). Care was taken to minimize the effect of bending moment due to eccentricity in the joint using appropriate spacers [32].

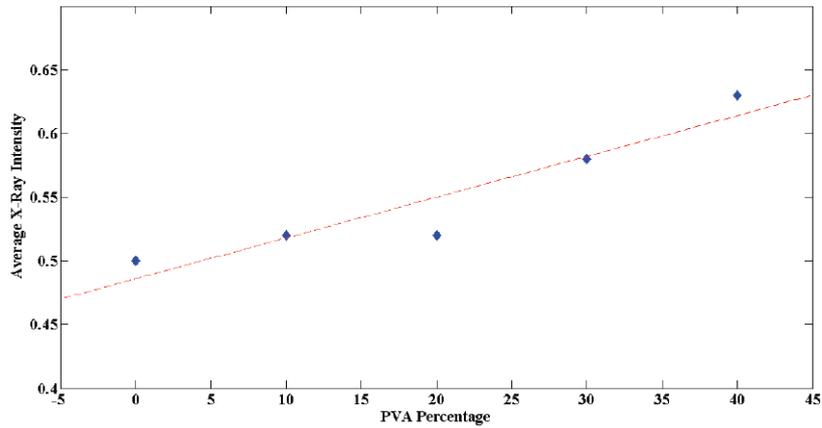


Figure 15. Variation of average intensity of the digital X-Ray images with respect to different PVA Percentages.



Figure 16. Test set up for determining the bond strength.

The shear strength for each sample was obtained and plotted against the PVA percentage (Fig. 17). It can be observed that the shear strength of the adhesive joint decreases with increase in PVA content. The healthy samples had average shear strength of 6MPa; while the 40% PVA samples had average bond strength of 3.65 MPa. There is a severe degradation of bond strength for 40 percent PVA samples. Table 3 shows the mechanical test results. Though there is significant scatter in the test results within each set of samples owing to different material and test parameters such as properties of each adherend layer, surface conditions of the bonded area, bond-line thickness, curing conditions of adhesive joints etc., the change in bond strength of different sets of samples clearly indicate the loss of strength due to degradation caused by addition of varied PVA content.

Table 3. Mechanical Test Results

| Sample | Maximum Shear Strength (MPa) | Minimum Shear Strength (MPa) | Average Shear Strength (Variation) |
|---------------|------------------------------|------------------------------|------------------------------------|
| GFRP adherend | - | - | - |
| Epoxy +0% PVA | 7.2 | 4.6 | 6.0 (±20%) |
| Epoxy+10% PVA | 5.8 | 4.9 | 5.4 (±8%) |
| Epoxy+20% PVA | 5.5 | 4.6 | 5.2 (±11%) |
| Epoxy+30%PVA | 5.2 | 4.2 | 4.8 (±12%) |
| Epoxy+40%PVA | 4.1 | 3.1 | 3.6 (±14%) |

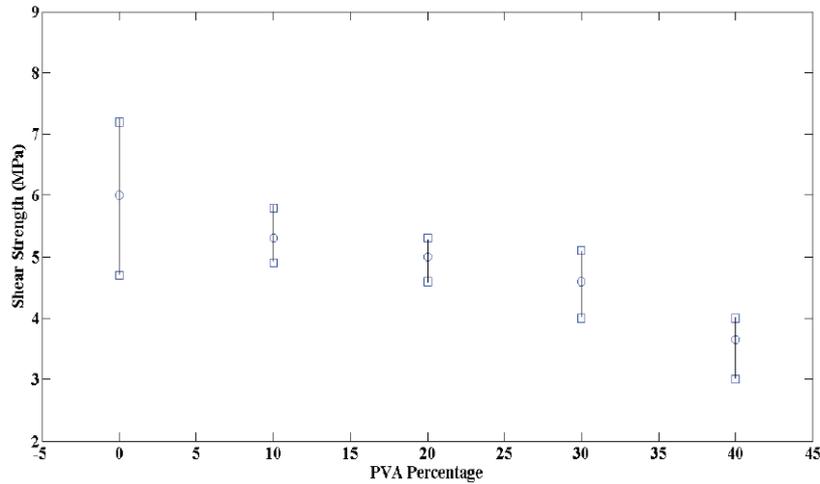


Figure 17. Variation of Shear Strength with different PVA Percentages.

The failed adhesive joint surfaces were visually inspected to examine the failure mode. Joints having higher percentage of PVA showed mixed mode of (adhesive and cohesive) failure parts of adhesive layer stuck to each of the adherend surfaces. Where as joints in healthy set showed a pure adhesion failure (Fig. 18) with higher bond strength.



Figure 18. Failed Adhesive joints of a) Healthy Sample and b) PVA added sample.

A linear correlation was found between the coefficient of reflection from the first interface and the joint strength (Fig. 19), the dashed line is the best fit linear curve for the experimental values. It shows that as the coefficient of reflection from the first interface decreases the shear strength of the adhesive joints increases

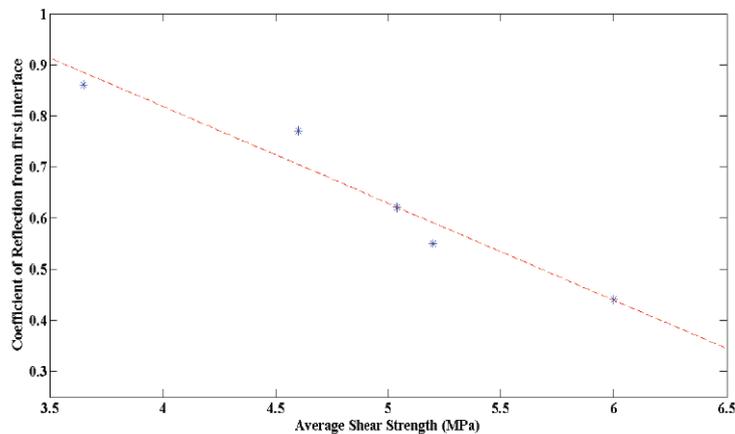


Figure 19. Correlation between Amplitude of reflection from first interface and Average shear strength of Lap joints

6. CONCLUSION

The bond strength of an adhesive joint is governed by various factors like bond line thickness, joint geometry, surface preparation, type of adhesive used, curing process etc. Each factor has its own influence on the bond strength. An NDE approach has been adopted through a series of experimental investigations to evaluate the bond quality and strength of glass fiber reinforced composite adhesive lap joints. An attempt also has been made to obtain correlation between the bond strength and the NDE measurements. Ultrasonic and X-ray imaging have been utilized for evaluating the bonded joints. The effort has yielded encouraging and interesting results indicating that these methods can be effectively used to evaluate the quality of adhesive joint. Further investigations along the same line with larger set of samples to cater for statistical variations can lead to proper quantitative correlation between NDE parameters and bond strength of such adhesive composite joints.

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