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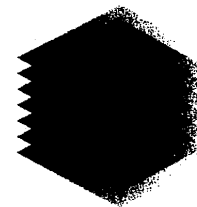
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## Real-time monitoring of an adhesive lap shear test using piezospectroscopy

Amanda L. Stevenson,<sup>1</sup> Ashley Jones,<sup>2</sup> and Seetha Raghavan<sup>3</sup>  
*University of Central Florida, Orlando, FL, 32816*

The stress distribution within an  $\alpha$ -alumina filled adhesive was evaluated using piezospectroscopy. Using optically transparent fiberglass substrates, a single lap-shear loading test was configured and spectral characteristic surface maps of the adhesive were obtained at incremental loads. Corresponding contour plots were created to visually display the stresses within the material due to the applied external load, verifying the capability of a real-time stress monitoring application.

### Nomenclature

$\alpha$	= alpha phase
kN	= kilonewton
nm	= nanometer
MPa	= megapascal
R1	= leftmost (first) R-line curve
R2	= rightmost (second) R-line curve
$\sigma_{ij}$	= piezospectroscopic stress tensor
$\Pi_{ij}$	= piezospectroscopic coefficients
$\Delta\nu$	= frequency shift

### I. Introduction

#### A. Background

The use of adhesives in the joining of aircraft structures has been in effect for over 50 years and is becoming increasingly widespread with their significant advantages over traditional metallic fasteners and rivets. Some improvements in manufacturing due to implementing adhesives include alleviating stress concentrations due to fastener holes and decreasing the overall weight of the aircraft. To fulfill structural requirements, the integrity of these adhesives must be studied extensively to determine their behavior under the loads experienced during operation conditions. Unreinforced polymers or adhesives have inferior mechanical properties compared to that of metallic fasteners and for safety purposes, cannot be used "as-is" in the

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operational field. A solution to this problem is the addition of “modifiers” to these adhesives, which have been shown to increase the adhesion, toughness, and peel strength of the materials (1). Both micron and nano-sized particles have recently been used for this purpose, including such materials as aluminum oxide ( $\text{Al}_2\text{O}_3$ ) and titanium (IV) oxide ( $\text{TiO}_2$ ) (1,2). While it may be more convenient and easier to embed micron-sized particles, the use of nano-particles ensures a higher surface-to-volume ratio, resulting in superior mechanical properties of the polymer matrix (3). Despite the improved mechanical properties, there are limitations to the amount of additive that can be incorporated into a base resin and still achieve a uniform dispersion. According to Gilbert et. al (1), an adhesive containing 10% nanoparticles by weight serves as the maximum mixture ratio. In their work, Munson et. al (4) also defined an upper limit for nanoparticle modifiers as 43% by volume additive. The uniform dispersion of modifiers is necessary as this ensures an even distribution of stress propagation throughout the adhesive during loading. Furthermore, if agglomerations are formed, these areas in the adhesive will be subjected to higher stress, resulting in an unbalance of the stress distribution, affecting the overall mechanical properties of the material.

In addition to improving mechanical properties, alumina nanoparticle modifiers have capabilities through photo-luminescence to provide a wealth of information on the particle dispersion and stress distribution within a composite or adhesive. This phenomenon was recently verified by calibrating the stress distribution in nanoalumina-filled epoxy composites having various volume fractions of modifiers (5). The results are implemented in this study to apply the concept to an adhesive in a single lap shear test. In order to broaden the capabilities of alumina nanoparticle modifiers, this work aims to provide a real-time evaluation of the stress within an adhesive under load using optical spectroscopy. By embedding these photo-luminescent particles into the adhesive, the method of piezospectroscopy can be used to monitor the stress distribution within the adhesive that corresponds to a specified applied external load.

## B. Piezospectroscopy

When excited by a laser, chromium ( $\text{Cr}^{3+}$ ) ions that naturally dope the crystal structure of alumina, transition energy levels. This results in a very distinct photo-luminescent spectrum, containing two prominent and closely spaced peaks, known as the R-lines. Piezospectroscopy directly relates the shift of these two peaks as a function of applied stress, resulting in the ability to determine the stress within a material through the optical spectra. This method is favorable as it is non-invasive. First described as a theoretical relationship by Grabner (6) in his work on sintered polycrystalline alumina, the piezospectroscopic effect (PS) is the relationship between an observable shift in spectral peaks with stress and is governed by the following tensorial expression:

$$\Delta\nu = \Pi_{ij}\sigma_{ij}$$

where  $\Delta\nu$  is the frequency shift,  $\Pi_{ij}$  represents the PS coefficients, and  $\sigma_{ij}$  is the stress tensor.

### C. Deconvolution

The exact peak values of the characteristic R-lines produced from  $\alpha$ -alumina are not available from the raw, experimental data, as they share spectral data due to their close proximity to each other (4). In order to receive accurate and precise peak positions, the spectral data must first be processed using a curve-fitting procedure. Throughout this work, a genetic algorithm (GA) based procedure was used for its capability of global optimization. Previous work by Raghavan et. al (8) on polycrystalline alumina showed that this method provided correct R-line and vibronic sideband peak positions. Four main functions are performed on the raw data for R-line optimization: baseline removal, curve cropping, curve separation, and curve recombination.

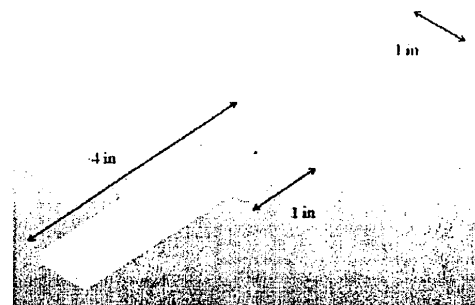
To design and create optimized stress-sensing adhesive materials, a bridge between the well-established compression results of polycrystalline alumina and alumina nanoparticles under loading conditions must be established. This information was obtained in a previous experiment (5) in which three alumina-epoxy nanocomposites, varying from 5% to 38% alumina by volume, were created and tested to investigate the behavior of the photo-luminescent data collected from the alumina particles as they are subjected to compression loads while embedded inside an epoxy matrix. The PS coefficients determined for each nanocomposite ranged from  $3.2 \text{ cm}^{-1}/\text{GPa}$  to  $5.7 \text{ cm}^{-1}/\text{GPa}$  for the R1 line.

## II. Stress-Sensing Alumina-Filled Adhesives

The strength of adhesives is typically evaluated structurally by subjecting the specimen to lap shear tests, either in single or double configurations. For this study, the single lap shear test was selected for simplicity in developing and validating our method. The behavior of joints that can be modeled with the single lap-shear design depend on several factors including the properties and thickness of the adherends, length of the adhesive overlap region, and length of the specimen(10). These factors were taken into consideration when choosing the experimental materials and test configurations.

### A. Materials

The substrates used for the single lap shear test consisted of fiberglass and epoxy, both common materials currently used in the aerospace industry. The dimensions of each substrate were manufactured as 25.4 mm x 101.6 mm (1" x 4") according to ASTM D5868, shown in Figure 1. Tuffbond 317, a high viscous aerospace grade adhesive, obtained from Hernon Manufacturing, was utilized because it bonds well to fiberglass and its components can be mixed in a variety of configurations. Alumina nanoparticles obtained from Advanced Materials, having an average

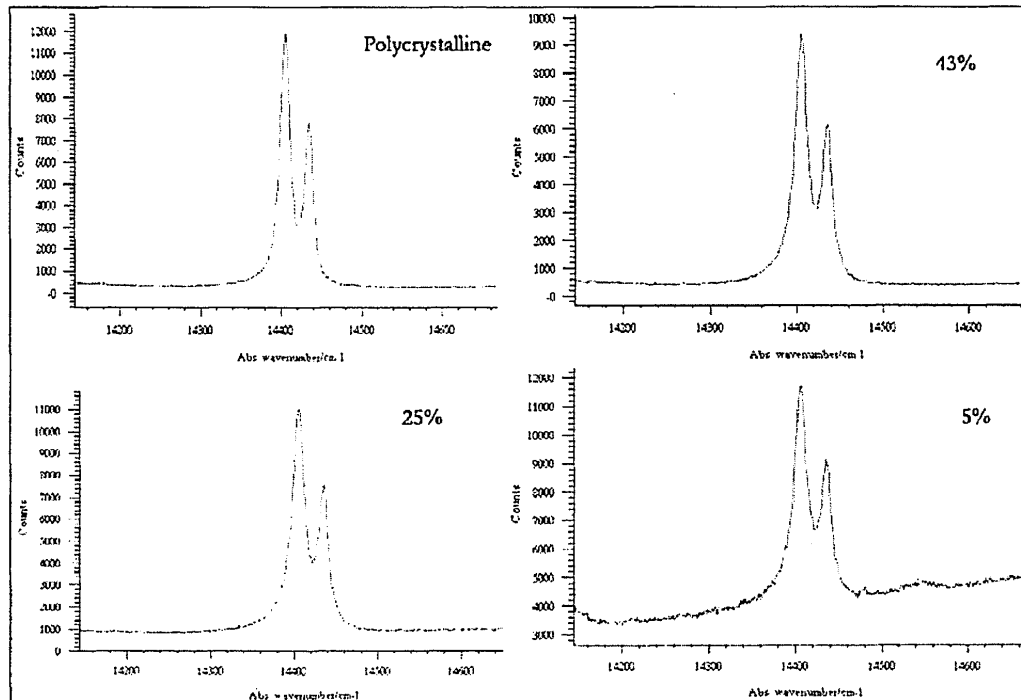


**Figure 1:** Single lap-shear specimen design per ASTM D5868 for fiber reinforced plastic (FRP) bonding.

particle size of 150 nm and purity of 99.85% were used as the filler material to create an adhesive containing 13% by volume particles.

## B. Fiberglass Optical Tests

To ensure that the real-time monitoring method using piezospectroscopy could be applied to this specific lap-shear configuration, the previously described volume fractions of alumina-filled epoxy composites were placed under 0.5 mm thick fiberglass substrates and photo-luminescent data was collected. Alumina photo-luminescence is extremely high, allowing for measurements to be obtained from it even when embedded within a polymer matrix. Typically, these matrices are not photo-luminescent and do not contribute to the resulting spectra. Figure 2 displays the results for this experiment. It is clear that sharp R-lines were obtained, even for the 5% by volume alumina composite. Measurements from the composites through the fiberglass were compared to the measurements from the composites without the fiberglass. A small, consistent decrease in intensity was witnessed for all volume fraction specimens. This trend may be explained through the reduction in transmissibility of the laser through the fiberglass coupled with the difficulty in focusing the laser directly on the alumina composite. While the transmissibility may have contributed to the small decrease in the intensity, it was not sufficient enough to affect obtaining clear and distinct R-lines. The results of these tests verified the capability of real time monitoring of the adhesives through fiberglass substrates.



**Figure 2:** Sharp and narrow R-lines were collected through the fiberglass substrates from 4 different specimens

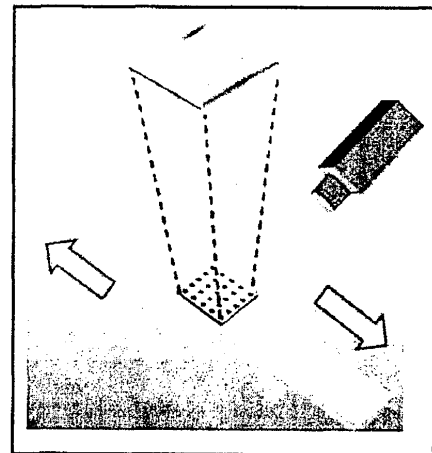
### C. Single Lap-Shear Test

An MTS Insight system capable of up to 50kN force was used for applying specific, incremental tensile loads to the substrates. In a special equipment setup, a fiber optic probe coupled with a Renishaw raman spectrometer was affixed to an x, y, z stage, allowing for specific regions of the adhesive to be examined. The real time monitoring of the adhesive lap shear test using piezospectroscopy was made possible by this novel setup as it is necessary to simultaneously load the specimen and collect photo-luminescent data. An argon laser operating at a 532 nm wavelength was used as the excitation source and designed to remain perpendicular to the adhesive during the application of the tensile loadings. Due to the working and focus distance requirements necessary for the experimental configuration, a long working objective was attached to the probe having 10x magnification. Figure 3 illustrates the single lap shear test set up in detail. A shim was placed within the grips to ensure that the tensile load remained in the same plane as the substrates during the test. According to ASTM D5868, the tensile loads were applied at a rate of 13 mm/min (0.5 in/min) until failure.

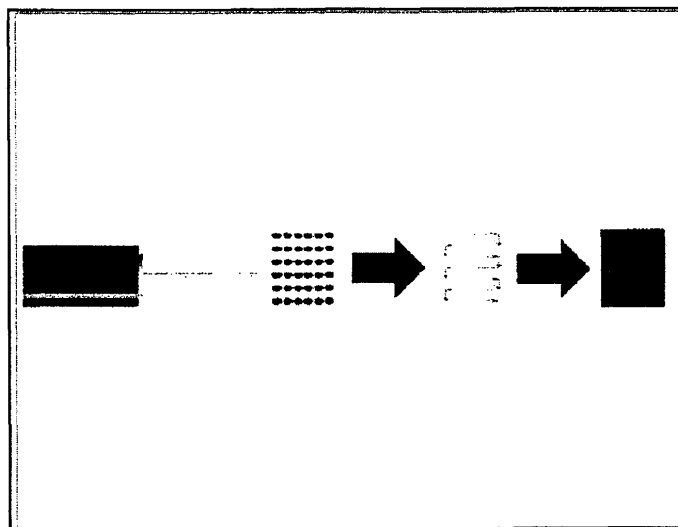
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Spectral characteristic surface maps comparing R-line peak positions were created for three tensile loads: 0 kN, 0.6 kN, and 1.2 kN. The unloaded load, 0 kN, was maintained as the reference load, while the 1.2 kN represented the maximum load. At every 50 MPa (0.18 kN) load, the experiment was held and photo-luminescent data from the alumina within the adhesive was collected in the form of a spectral surface map. To ensure that the optimal focal distance was attained, the x, y, z stage was incrementally moved in the z-direction (away and towards the adhesive) while the intensity of the R-lines were observed. The laser spot was determined to be focused when the R-lines were at maximum intensity. This focal distance was reserved for all data collection points for the surface of the adhesive. There were a total of 36 data collection points; 6 in the x-direction and 6 in the y-direction.

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**Figure 3:** Single lap-shear experimental set up. Depicts the design of real-time monitoring of the behavior of the



**Figure 4:** Design for creating contour maps. Note: Diagram is not to scale

Once all data was collected, it was processed by the previously described deconvolution program to curve fit the spectra. Contour plots comparing the R-line peak positions at every spectral data point were created at each incremental load to visually confirm the stresses throughout the adhesive due to the applied load. This systematic process was repeated until the adhesive failed and debonded from the fiberglass substrates, which occurred at approximately 1.2 kN.

A variety of information can be obtained from the lap shear specimens regarding the behavior of the adhesive during loading. A material experiencing a uniform stress state should produce a contour map that is monochromatic, indicating that the peak positions of the R-lines are approximately the same throughout. This association can be seen when analyzing the stress contour produced under no external load (0 kN). An overall consistent distribution in the R1 peak positions can be observed, as shown in Figure 5. The variances in the peak positions of this contour range from approximately  $14404.9\text{cm}^{-1}$  to  $14404.5\text{cm}^{-1}$ , except for two regions which clearly have a deviation in stress as compared to the rest of the material. These stress variations indicate possible pre-existing residual stresses on the alumina particles, which could be the result of uneven adhesive curing, varying adhesive thickness, non-homogeneous particle dispersion, or agglomerations.

As observed from the other contour maps in Figure 5, there were significant deviations in the range of the R1 peak positions as stress was applied to the adhesive. The overall peak positions ranged from  $14403.5\text{cm}^{-1}$  to  $14404.9\text{cm}^{-1}$ , presenting a frequency difference of approximately  $1.4\text{cm}^{-1}$  from the reference load to the maximum load.



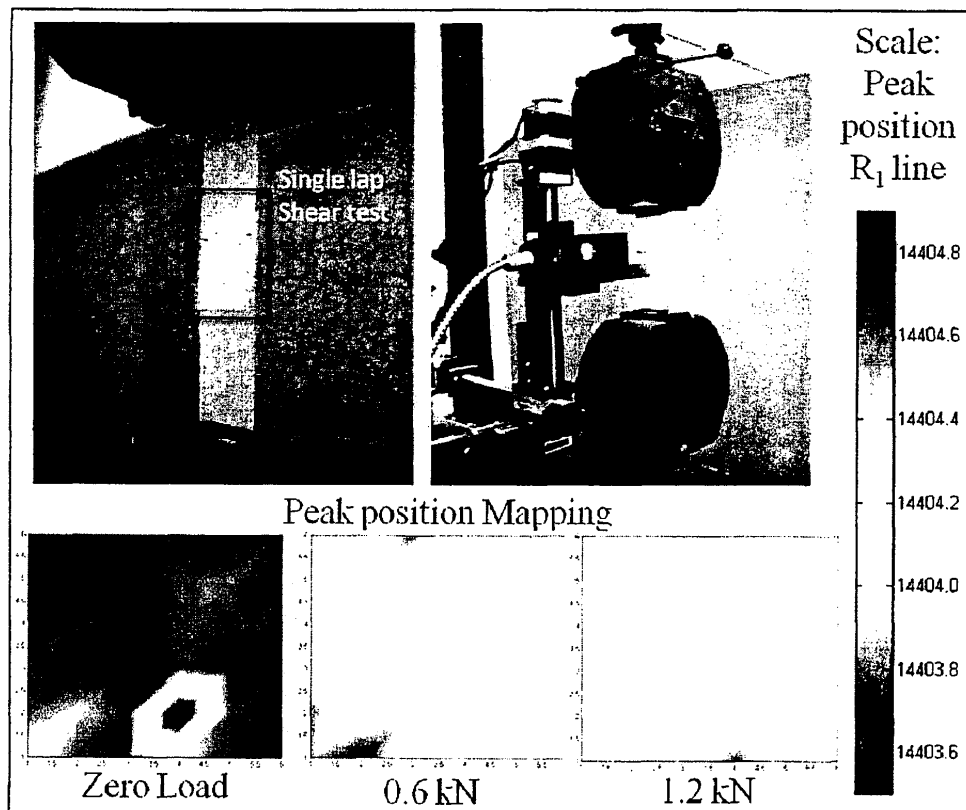


Figure 5: Stress distribution with respect to load presented by the comparison of the R1 peak position

From Figure 5, the apparent frequency shift from 0 kN to 0.6 kN is  $0.45 \text{ cm}^{-1}$  in the mid-region of the overlap. Using previously determined PS calibrations from work on alumina-filled nanocomposites (5), the PS coefficient for 13% alumina volume can be approximated as  $3.36 \text{ cm}^{-1}/\text{GPa}$ . The PS effect equation for these values gives 133.9 MPa as an approximate stress of the adhesive under 0.6 kN load. From the reference load to the maximum load of 1.2 kN, the same calculations can be used to obtain a maximum stress of 148.8 MPa given the apparent frequency shift of about  $0.5 \text{ cm}^{-1}$  in the mid-region of the overlap.

#### IV. Conclusions

The initial results of this work show that piezospectroscopy can be used to analyze the stress distribution within an adhesive in a real time context. This offers further benefits as compared to current methods, such as strain gage rosettes (10) because piezospectroscopy, in comparison, has high spatial resolution, is non-contact, and is not limited to localized regions. Additionally, the ability to receive spectral information from an entire sample surface during in-situ loading has the potential to improve the development of adhesives with optimized mechanical properties. By embedding a polymer with photo-luminescent particles, such as  $\alpha$ -alumina, the material not only strengthens but also provides a non-invasive means to assess stress within an adhesive prior to failure. This method is capable of providing high spatial resolution measurements, and the

resolution of the contour maps can be adjusted to suit the needs of various material dimensions. Development of the testing method for the stress sensing adhesive forms an important motivation for laboratory-based efforts to understand how an adhesive can be improved through the incorporation of filler particles.

### Acknowledgments

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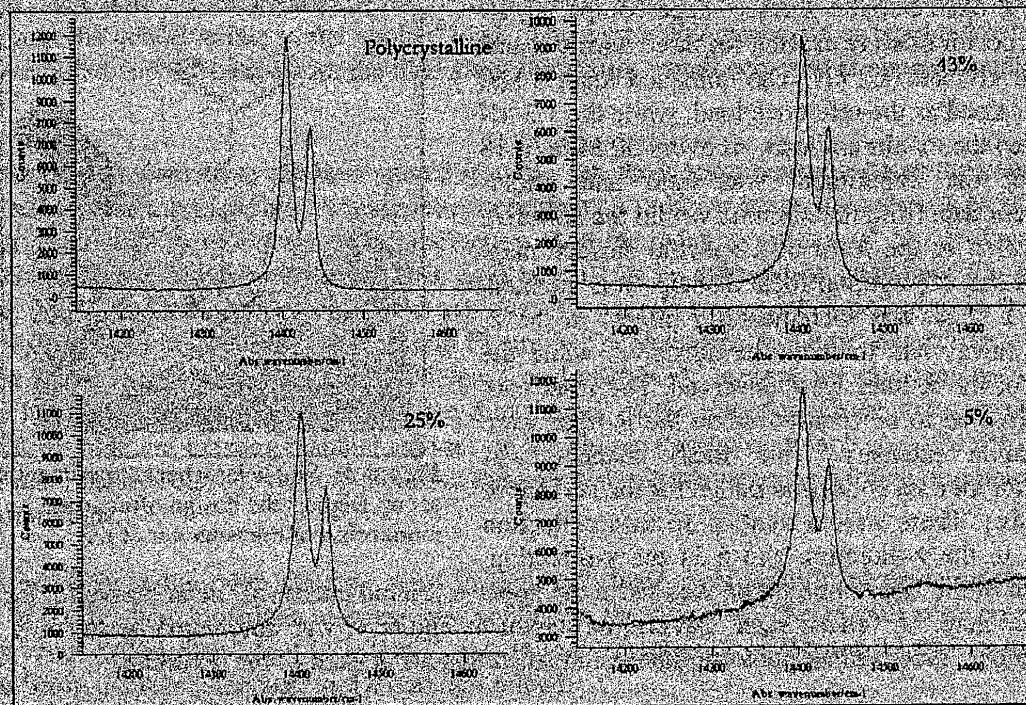


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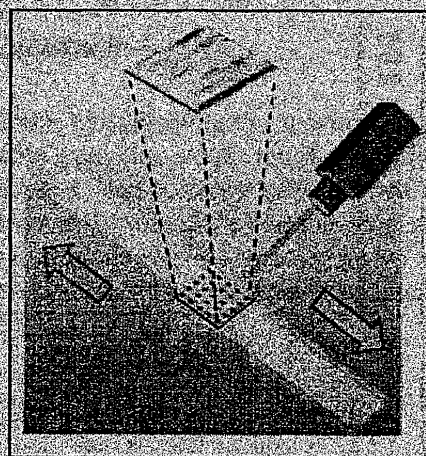
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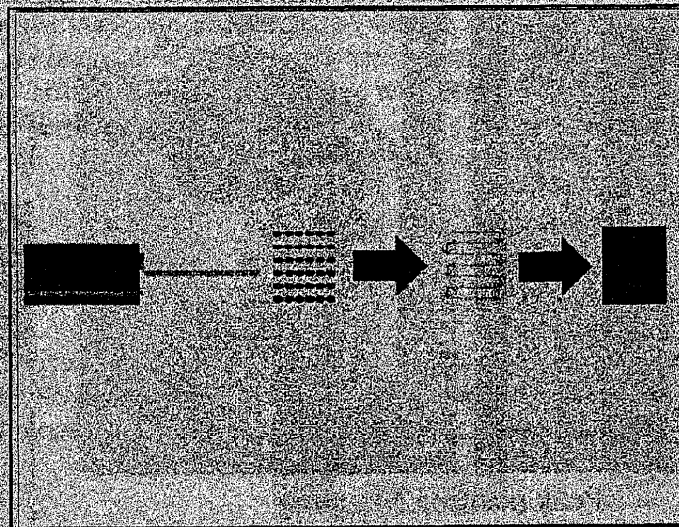
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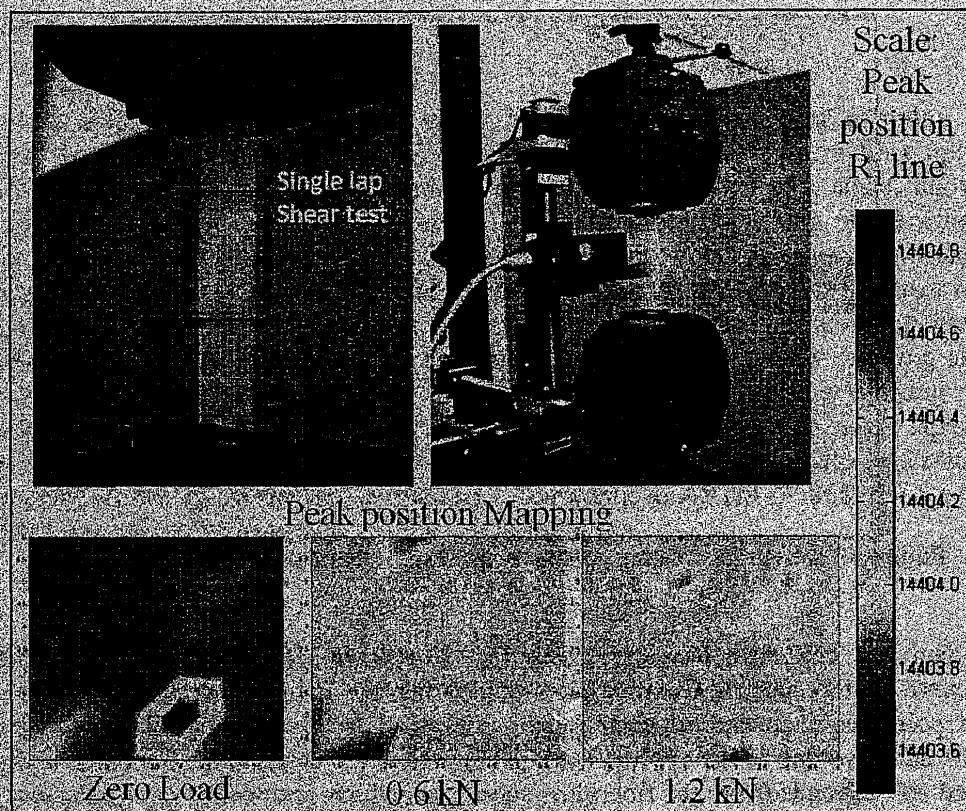


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From Figure 5, the apparent frequency shift from 0 kN to 0.6 kN is  $0.45 \text{ cm}^{-1}$  in the mid-region of the overlap. Using previously determined PS calibrations from work on alumina-filled nanocomposites (5), the PS coefficient for 13% alumina volume can be approximated as  $3.36 \text{ cm}^{-1}/\text{GPa}$ . The PS effect equation for these values gives 133.9 MPa as an approximate stress of the adhesive under 0.6 kN load. From the reference load to the maximum load of 1.2 kN, the same calculations can be used to obtain a maximum stress of 148.8 MPa given the apparent frequency shift of about  $0.5 \text{ cm}^{-1}$  in the mid-region of the overlap.

#### IV. Conclusions

The initial results of this work show that piezospectroscopy can be used to analyze the stress distribution within an adhesive in a real time context. This offers further benefits as compared to current methods, such as strain gage rosettes (10) because piezospectroscopy, in comparison, has high spatial resolution, is non-contact, and is not limited to localized regions. Additionally, the ability to receive spectral information from an entire sample surface during in-situ loading has the potential to improve the development of adhesives with optimized mechanical properties. By embedding a polymer with photo-luminescent particles, such as  $\alpha$ -alumina, the material not only strengthens but also provides a non-invasive means to assess stress within an adhesive prior to failure. This method is capable of providing high spatial resolution measurements, and the