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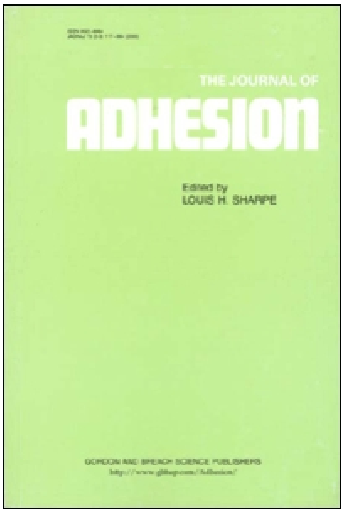

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# NOTE

# A Preliminary Study of the Use of Kynar® Piezoelectric Film to Measure Peel Stresses in Adhesive Joints†

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## INTRODUCTION

A variety of test techniques have been developed to test the performance of adhesives bonded *in situ* within joints. Most of these techniques measure strength, fracture toughness, or adhesive modulus of the bonded joint. Techniques to measure actual stress or strain values within a bonded joint are quite few in number. The Krieger gage[1] is able to measure the average shear displacement along a 12.5 mm. gage length of a thick adherend joint. It has been used primarily to measure *in situ* shear moduli of adhesives. Brinson and his colleagues[2] proposed bonding strain gages within adhesive joints to measure strains within the adhesive. Unfortunately, these gages are only sensitive to the lateral strains and not shear or peel strains. Because the lateral strains are dominated by the behavior of the adherends rather than the adhesive, the information which can be gained is incomplete.

Other useful approaches for determining adhesive deformation fields are high-precision optical techniques. Post and his colleagues[3,4] have applied high frequency cross gratings to the edges of lap joints and measured displacements in two perpendicular directions with Moiré interferometry. From these deformation fields, they could compute strain fields on the edge of the entire joint. Liechti, *et*

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*al.*[4–7] have employed classical interferometric procedures to measure the opening mode displacements for debonds along an interface between two materials. In this approach the bond is viewed from a position perpendicular to the bond plane. The technique has been applied to several material systems to determine crack opening displacements and strain energy release rates. Knauss[8] has used a similar technique and carefully analyzed the entire outer surface displacement of the adherend in an attempt to measure *in situ* stress-strain relations throughout the rupture process. Although these optical techniques can provide a great deal of information about adhesive joint displacements, their nature restricts them to laboratory environments.

In an effort to develop a technique which is not limited to optical isolation tables, KYNAR® piezoelectric film, manufactured and courteously supplied by the Pennwalt Corporation, is currently under evaluation. In current embodiment, the technique is very sensitive to peel stresses within bonded joints. This is highly desirable because peel stresses are commonly believed to be responsible for joint failure in many applications. Reasons for this belief include the fact that peel stresses are often quite large even in lap-type joints, and that the critical strain energy release rates for peel ($G_{Ic}$) are often smaller than for shear ($G_{IIc}$ and $G_{IIIc}$).[9] DeVries, *et al.*[10] have even suggested that the peel stresses in certain double lap joints are large enough to induce permanent yielding (bending) in sufficiently thin adherends.

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The piezoelectric film is sensitive to changes in load (and temperature) and is especially applicable to cyclic, impact, or other dynamic loading situations. The manufacturer claims that the film has a useful frequency range of 0.005 Hz. to the order of gigaHz.[11] This dynamic nature implies that the utility for measuring residual stresses would be very limited. Nonetheless, the technique has shown considerable potential for measuring mechanically-induced peel stresses. Because no other technique with such versatility currently exists, it offers the potential to fill a portion of the significant void which now exists in experimental adhesive joint evaluation. The following results are preliminary, but do demonstrate the potential for the technique.

## BACKGROUND AND DEVELOPMENT OF THE PEEL STRESS SENSOR

KYNAR piezo film consists of a polyvinylidene fluoride (PVDF) polymer which has been stretched and then poled in an extremely high electrical field at elevated temperatures to exhibit significantly greater piezoelectric properties than are obtainable with other polymers.[11] A very thin layer of metallization is deposited on each side of the film to provide a conductive path. The film is commercially available from the Pennwalt Corporation in a variety of thicknesses from 9 to 800 $\mu$m and with a variety of metallizations. When subjected to a normal strain in any of the three principal directions, an open circuit voltage is created between the two metallizations. Theoretical considerations reveal that the films should not be sensitive to shear deformations.[12] By attaching leads to the film and then subjecting the film to a changing load or temperature, a relatively large output voltage develops across the leads. For the 28 $\mu$m film, for example, a 1 MPa stress normal to the film should produce an open circuit voltage of 9.5 V.[11]

KYNAR piezoelectric film has been widely used in a variety of applications. A summary of many of these applications and patents is presented in Ref. 11. The material may be used as either a sensing unit or as an electromotional device. Of special note is an acoustic emission study by Stiffler and Henneke[13] where they used different backing configurations to tailor the sensitivity of the film to the type of wave they were trying to measure, thereby demonstrating some of the versatility of the film. The most relevant use of the piezo film to our discussion was a study by Chou and Ekstrom[14] in which they utilized KYNAR strips to measure contact pressures between laminated plates. By recording data during unloading steps, they were able to obtain good agreement with theory and pressure probes. Their successful results have helped provide the impetus for this current study.

One especially interesting feature of the KYNAR peel stress sensor is that it can be calibrated to be a "stress gage" rather than a "strain gage". This is especially advantageous for the highly constrained conditions imposed on the adhesive by the adherends which tend to limit the strains significantly. The film is quite resistant to moisture and other chemicals and, in fact, the polymer is used as a sealant for corrosion protection in some applications. This suggests that the film should be quite durable for certain applications, although the long-term integrity of the metallization layers and the film-metallization bonds would be questionable. One problem with the film is that the PVDF loses its piezoelectric properties when exposed to high temperatures. The film cannot be used above 100°C, and does exhibit a time-dependent loss of properties at temperatures as low as 60°C.[11] This temperature effect is a serious limitation to widespread use for adhesive joints and composite materials because the film cannot be used with high temperature cure adhesives. PVDF has a glass transition temperature of −40°C.

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The volume resistivity of the polymer is quite high (15 TΩ · m), so even in the very thin films, the resistance to electron flow which discharges the metallizations is normally much higher than the impedance of ordinary instrumentation. The capacitance of the film is not negligible, and must be taken into consideration when designing any electrical circuitry for measuring the created voltage.[11]

Piezoelectric constants are third-order tensors which relate stress or strain, which are second-order tensors, to the electric field or electric displacement which are first-order tensors. For our purposes, we will express the piezoelectric constants in contracted notation with the first subscript denoting the orientation of the electric field, and the second subscript taking on values of 1 to 6 to reflect the components of the symmetric stress tensor.[14,15] The values of the piezoelectric constants are given in Table I.[10] As can be seen from the table, the sensitivity in the 1- and 3-directions are similar, but the sensitivity in the 2-direction is about one order of magnitude less. Since the electrical output of the film is

$$V = tg_{3i}\sigma_i$$

where $V$ is the generated open circuit voltage, $t$ is the film thickness, $g$ is the

TABLE I
Piezo properties [Pennwalt Corporation][11]

| | | | |
|---|---|---|---|
| Piezo stain constant | $d_{31}$ | $23 \times 10^{-12}$ | (m/m)/(V/m) |
| | $d_{32}$ | $3 \times 10^{-12}$ | or |
| | $d_{33}$ | $-33 \times 10^{-12}$ | (C/m$^2$)/(N/m$^2$) |
| Piezo stress constant | $g_{31}$ | $216 \times 10^{-3}$ | (V/m)/(N/m$^2$) |
| | $g_{32}$ | $19 \times 10^{-3}$ | or |
| | $g_{33}$ | $-339 \times 10^{-3}$ | (m/m)/(C/m$^2$) |
| $(d_{34} = d_{35} = d_{36} = g_{34} = g_{35} = g_{36} = 0)$ | | | |

piezoelectric stress constant and $\sigma$ is the applied stress (indicial summation notation is assumed), the output voltage is the sum of the contributions from the normal stresses in the three principal directions. Because the compliance of the film is similar to that of typical adhesive layers and because it is very thin, it adds little perturbation to the surrounding lateral strain field. For adhesive joints consisting of adherends which are relatively stiff compared to the adhesive, the lateral strains in the adhesive are essentially the same as those in the adherends. If these strains can be estimated, it is possible to assess the errors which would be anticipated due to the in-plane deformations, providing fully-characterized elastic and piezoelectric properties are available. The available data do not seem complete at this time, however, suggesting the need for more calibration studies.

To minimize the film's sensitivity to stresses or strains in the in-plane directions, one should orient the film so that the 2-direction is parallel to the largest anticipated strains. For example, for the case of single and double lap joints, the film is placed with the 2-direction along the loading axis. Because the piezoelectric sensitivity in the 2-direction is an order of magnitude less than the sensitivity in the 1- and 3-directions, the adherend bending will have little effect on the output voltage from the sensor. For butt joints with relatively rigid adherends, the strain in the plane of the film is negligible and the output voltage may be isolated from an in-plane effects by properly calibrating the material for mounting on a rigid substrate.

One advantage of the piezoelectric film technique is that it is an imbedded technique rather than an edge technique such as Moiré. This permits one to measure peel stresses over any small discrete region within the adhesive layer. This can be achieved by properly etching the film to make the film sensitive to a certain region. The output voltage is only sensitive to the average stress level over the region of the film where both metallizations are intact. By etching away one or both metallizations, the region does not respond to the piezoelectric input. Using multiple leads and sampling points, a stress sensor capable of measuring peel stresses at numerous discrete locations is possible. Etching is easily performed by using standard circuit board etchant kits.

Another technique being considered for making more refined grids is photolithographic pattern transfer and etching. At present this method has not yet been successful. In order to activate the chemical photoresist, an ultraviolet light is used to expose the coated film. Due to the thinness of the film metallization

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surface, the light passes through the side facing the light source and exposes the back side of the film as well. Thus, the same pattern is generated on both sides after etching. Or, in our case, where both sides of the film are eventually subjected to the light, only the overlap locations would survive the etching process. If a photolithographic technique is successful, the peel stress sensor could conceivably be produced on a microscopic scale. Although the sensors discussed herein were at least $10^{-6}\,m^2$ in area, the manufacturer reports using sensing areas as small as $10^{-12}\,m^2$. These dimensions could allow one to measure even the order of singularity at bond terminations.

## EXPERIMENTAL RESULTS

Our first multi-point sensors were made with nine sensitive regions for insertion in lap joints with a 25 mm square overlap. The original configuration is shown in Figure 1a. It was thought that by connecting the readout device to the two leads which intersect at the desired location, the peel stresses could be measured. The signals obtained were found to be in error because the other sensitive regions were acting as a complex network of capacitors, thereby reducing the output signal. To avoid this type of capacitance cross-talk, it is necessary that each stress sensor be electrically isolated from all other stress sensors. Figure 1b schematically illustrates the improved configuration. Care should be taken in the design of the sensors to eliminate the parasitic capacitance effects.

The results reported herein were obtained by connecting a Tektronix Model 5113 dual beam storage oscilloscope to the output leads. Because the impedance of the module is only 1 MΩ, the decay times were quite short. Nonetheless, the outputs were very repeatable and calibration revealed quite linear output *vs.* input traces. Improved design will call for a higher impedance instrument such as a specially designed op amp or FET circuit or a charge amplifier, although high-impedance measuring devices can be more noise sensitive and may reduce the magnitude of the output signal. The relatively low impedance of our instrumentation explains the relatively large difference from the calibration curves and the tabulated piezoelectric constants. At this time, we have estimated the output by visual inspection of the oscilloscope trace.

The first experiment was employed to test both the linearity of the applied dynamic load/voltage output relationship and the effect of metallization overlap area on the voltage output. Two film sizes, 89 mm × 89 mm and 89 mm × 6.4 mm, were placed between two steel plates of dimensions 89 mm × 89 mm × 12.7, and loaded in an axial MTS testing machine using compression–compression square-wave cyclic loading. A piece of felt was placed on each film metallization surface in order to insulate it from contact with the steel slabs and to provide. a unifrom stress field. The cyclic tests were performed at a minimum load of 20 lbs and a maximum load varying from 40 to 120 lbs in 10 lb increments. The results are shown graphically in Figure 2. From these results, one can conclude that a linear relationship exists between the applied dynamic force and the output voltage and,

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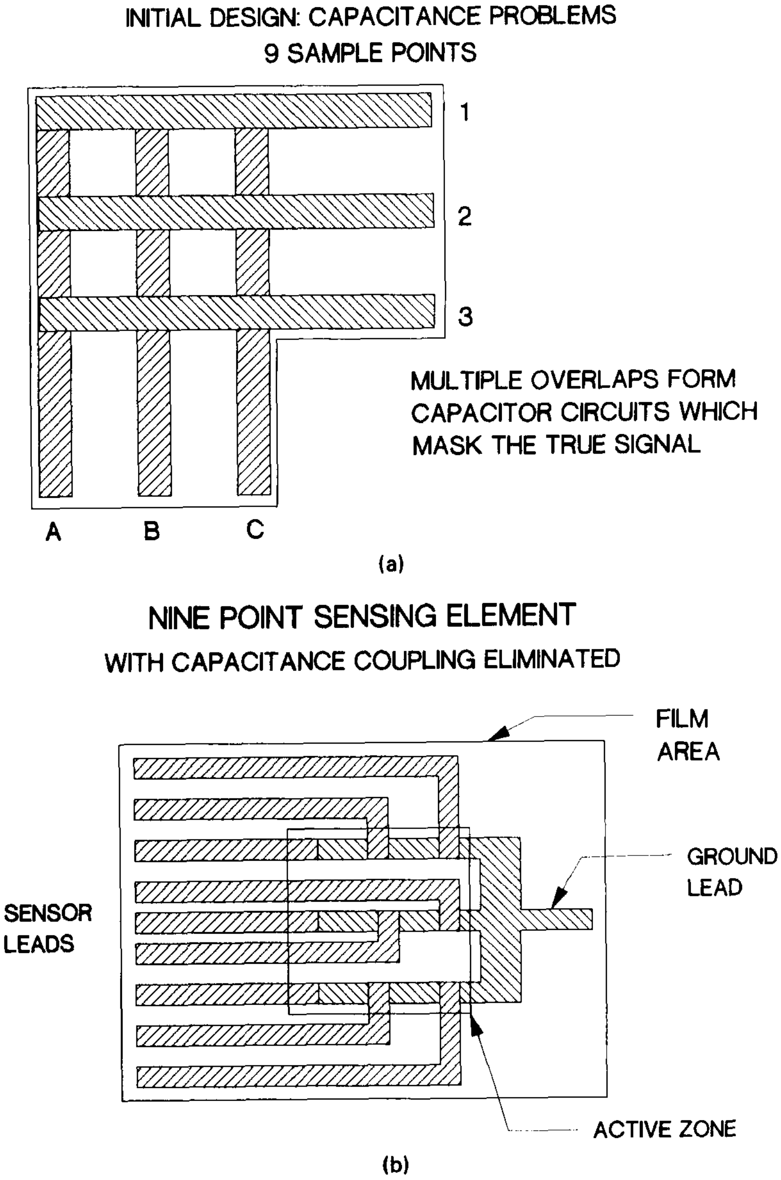

FIGURE 1 Schematic of nine point sensing grid a) with parasitic capacitance, b) without parasitic capacitance.

at least in square wave loading, the output voltage is independent of the area of metallization overlap.

The film was then used to determine the peel stress variations in single and double lap joints. Grids similar to those shown in Figure 1b were etched in the film, and the film was bonded into the lap joints made from 6061 aluminum

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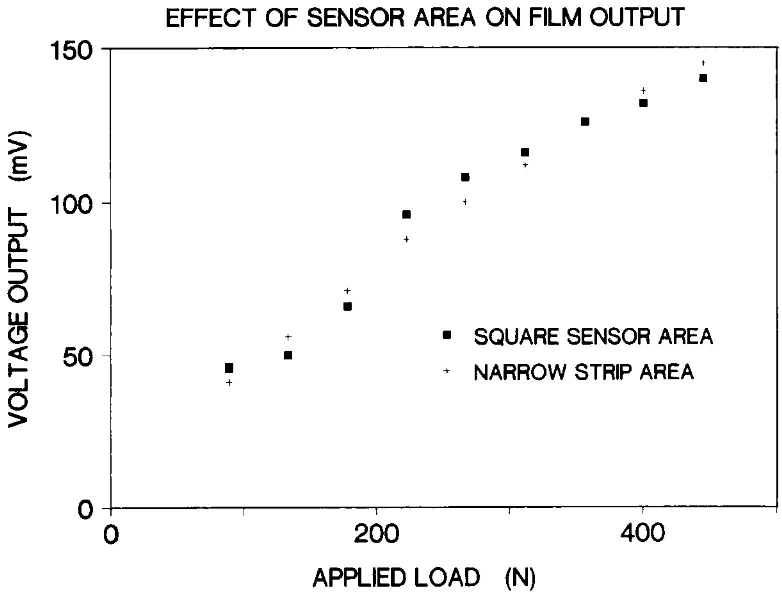

FIGURE 2 Comparison of output for the two film sizes under square wave loading.

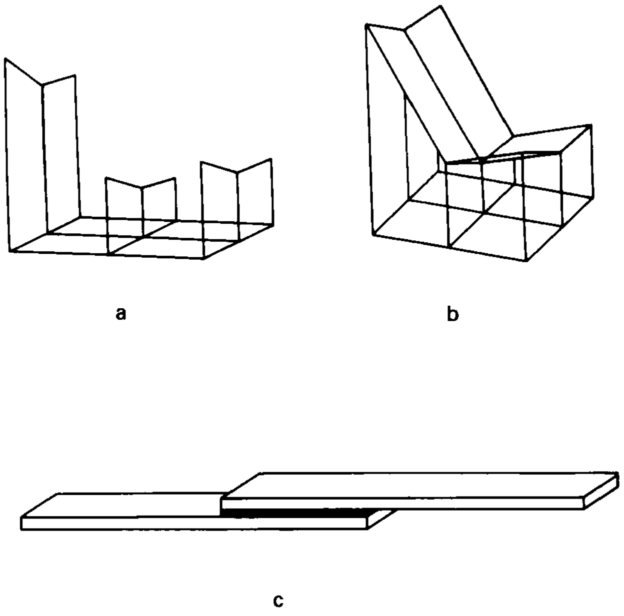

FIGURE 3 Single lap shear joint results from 1.03 MPa stress amplitude. a) 3-D point graph, b) 3-D surface chart, c) location of the stress profile.

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adherends of dimensions 101.6 mm × 25.4 mm × 3.18 mm. Care was taken to assure that the less-sensitive two-direction was oriented along the loading axis in order to lessen the effect of adherend bending on the output voltage. The adhesive used was a commercially-available, room-temperature-curing, two-part epoxy. Figures 3 and 4 graphically demonstrate the obtained results. In both cases, the results give a fair representation of the known peel stress trends.

The normal stress distribution in an elastomeric butt joint was also obtained using KYNAR film stress sensors. A circular elastomeric disk of radius 3.81 cm was bonded between two Plexiglas adherends. An etched film was bonded into one of the bondlines. Due to the axisymmetry of the specimen, a simple grid was employed consisting of a radial metallization strip on one side of the film and six strips perpendicular to the first on the opposite side of the film. The six perpendicular strips led from the radial strip beyond the bond of the specimen. This resulted in six stress-sensitive regions at differing radial positions. The elastomeric butt joint was chosen due to its known parabolic normal stress distribution.[17] The joint was loaded in compression using a sinusoidal wave input of 20 Hz, average mean stresses of 0.487 MPa and 0.731 MPa, and average stress amplitudes ranging from 0.292 MPa to 0.536 MPa. The data for 0.731 MPa appear in Figure 5. Note the parabolic stress variation which is consistent with theory.

In order to correlate the voltage outputs to dynamic stress amplitude, it was necessary to calibrate the piezoelectric film. Earlier efforts employing the same experimental method as was used to test dynamic stress/voltage linearity and overlap area dependence showed the resulting voltages to depend not only on dynamic stress amplitude but also on the mean stress level. Since this result was contrary to tests performed by the manufacturer, it was assumed that either the felt used to insulate the film metallization from the steel platens was acting as a buffer to dampen the dynamic load or the platen alignment was not exact. These

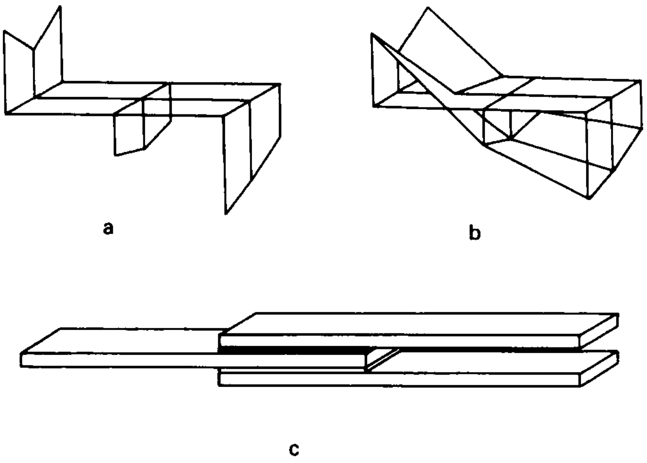

FIGURE 4 Double lap shear joint results from 1.38 MPa stress amplitude. a) 3-D point graph, b) 3-D surface chart, c) location of the stress profile.

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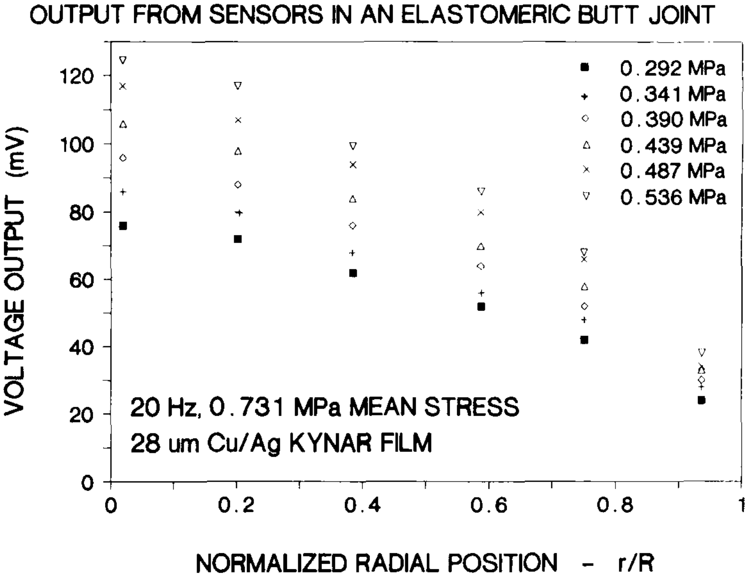

FIGURE 5 Data for elastomeric butt joint subjected to 20 Hz sine-wave loading at a mean stress of 0.731 MPa.

two possible error sources were overcome by bonding the film sensor between steel platens, thereby making a tensile butt joint. The active sensor area was small in comparison with the platen diameter, and was centered over the platen cross-section. By performing the bonding operation within the MTS testing device, proper alignment was guaranteed. Since the epoxy used has a Poisson's ratio near 0.35, the stress at the center of the bond was very close to the average stress.[9] The film was then calibrated in tension and compression using sinusoidal wave loading at 10, 20, and 30 Hz. Mean stresses of 0.139, 0.278, 0.417, and 0.556 MPa were used with the dynamic stress amplitude varying between 0.083 and 0.5 MPa in 0.028 MPa increments. Calibration curves for the three frequencies are shown in Figure 6. It is observed that throughout the entire range of mean stresses from compressive through tensile, no mean stress effect is present. The scatter in the data is believed to be caused by the instrumentation which will be improved in the future.

Using the calibration performed, the experimental elastomeric butt joint data were compared to theoretical predictions. It was found that the experimental data were nearly two times larger than the predicted values. A probable source of this difference is due to the use of metallic platens in the calibration procedure. Since the bond thickness between the steel platen and the film metallization was small, parasitic capacitance between the film metallization and the platen could be drawing off a portion of the generated voltage, an effect similar to the initial crisscrossing grids. The use of glass platens for calibration should eliminate this source of error.

In an effort to assess the effect of the imbedded film on joint strength, tests

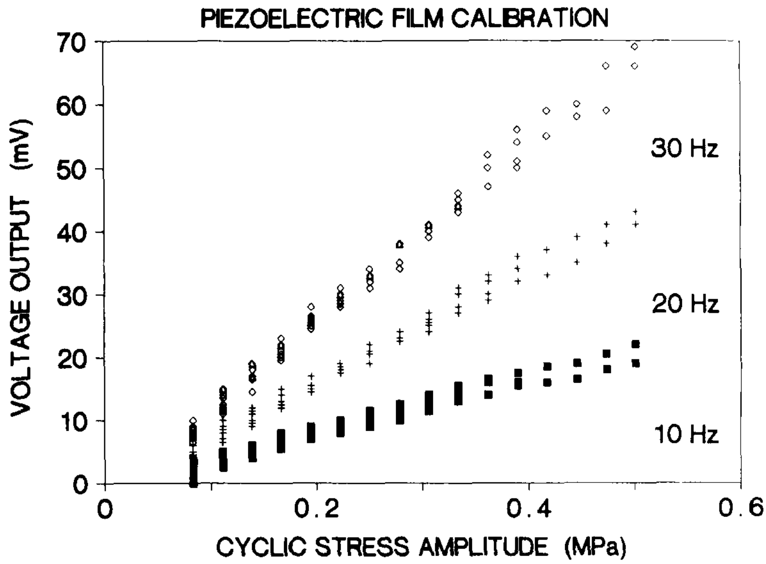

FIGURE 6 Comparison of calibration data at 10, 20, and 30 Hz sinusoidal loading.

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were performed using single lap shear joints to measure the difference in joint strength due to the imbedding of the piezoelectric film. Four specimens containing the film and four specimens without the film were stressed to failure in the MTS machine. The adherends of the specimens were abraded slightly with 340 grit sandpaper. The film could not be pretreated physically or chemically due to the delicate nature of the metallization surface. The average bond strengths, with the respective standard deviations in parentheses, for the two sample types were: 2.53 MPa (0.35) for the bonds without film imbedded and 1.81 MPa (0.32) for those with film. All failures for the samples without the film appeared to occur interfacially between the adhesive and the adherend. All failures in samples containing the film appeared to occur at the interface between the adhesive and the piezoelectric film. The low value for the samples without film could be improved by using an anodization surface pretreatment, but the even lower value for the samples containing the film represent a very restrictive load limitation for imbedded film adhesive joints. Testing may need to be done at quite small load levels or in compression to avoid failure at the KYNAR film interface.

## SUMMARY AND CONCLUSIONS

Peel stresses are often believed to be the most critical stresses responsible for failure of a variety of adhesive joints. Metallized polyvinylidene fluoride films have been etched to provide multipoint sensors which can be imbedded within adhesive joints to measure these peel stresses. Preliminary data suggest that the technique may offer potential to partially fill the significant void now existing in experimental adhesive joint testing. Although the approach is a dynamic technique and does have temperature and load restrictions, it has successfully illustrated the peel stress trends expected in single and double lap joints and in butt joints. Calibration studies have revealed that the output is quite linear and is independent of the mean stress level in tension and compression. Output has been measured with an oscilloscope, although improved circuitry is currently being designed to improve the decay time of the output voltage. Fairly large errors exist between calibration results and known stress fields, but the errors are believed to be caused by the existing instrumentation. This should be corrected by improved measurement capabilities. Photoresist patterning techniques have not yet been entirely successful, but may offer the potential to miniaturize the sensors to microscopic dimensions, thereby allowing peel stresses near steep gradients to be measured.

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