X-RAY DIFFRACTION EVALUATION OF ADHESIVE BONDS AND DAMAGE IN COMPOSITES

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ABSTRACT

Stresses can be measured by X-ray diffraction, not only in metals but also in polymeric composites containing some crystalline filler particles. Diffraction is found effective in disclosing the distribution of stresses over the surface of adhesively bonded joints in aluminum strips when loads well below the yield point are applied. When two 6061-T6 aluminum strips 1/16" or 1/32" thick and 3/4" wide are adhesively bonded in a single lap joint and loaded in tension, maps giving the distribution of the X-ray-measured stresses show clear evidence of the way in which stresses are transferred from one adherend to the other. The maps show the limits of the bonded area with an accuracy about equal to the width (1 mm) of the irradiated area along the specimen. Attendant bending stresses resulting from the loading are also registered. Stress values can be obtained from the observed diffraction angles by calibration with tensile tests of a single unbonded strip. Similar results are obtained for graphite/epoxy laminates adhesively bonded to aluminum when diffraction is from the aluminum, but a much lower accuracy was obtained when diffraction was from the filled composite.

Another X-ray method was developed that appears to be a viable though less accurate method for measuring applied (not pre-existing residual) stresses, and for mapping their distribution around a joint. A thin layer of epoxy paint containing a diffracting filler is applied to a specimen and cured. Diffraction from this paint yields shifts in diffraction angle approximately proportional to the magnitude of applied stresses. A diffracting paint containing, say, aluminum or silver powder can be used on an object that diffracts poorly or in which a filler has not previously been embedded, but the accuracies attainable in stress values are apparently somewhat lower and are being investigated further. When this method is used on the aluminum adherend of a single lap joint and a load is applied, the limits of the bonded area are disclosed by the diffracted beam from the filler in the epoxy paint. This method appears, therefore, to be useful for mapping the areas that are properly bonded and for associated bending stresses, and possibly for non-destructive evaluation of bond defects. Details will be published in Advances in X-Ray Analysis, Vol. 24.

Detection of damage in polymeric composites is possible by X-ray determination of residual stresses. By locally deforming a graphite/epoxy laminate the diffraction from embedded filler powder discloses effects of the deformation not only where damage has occurred but also throughout the immediate neighborhood of the visible damage. Investigation of this method continues with details to be published elsewhere.

INTRODUCTION

X-ray diffraction is widely used for the measurement of residual and applied stresses in metals. A series of experiments has shown that information can also be obtained about residual and applied stresses in polymeric materials, including polymer matrix composites,[1-3] provided suitable diffracting powders are embedded in them before they are cured. Laboratory experiments with an X-ray diffraction method of determining stresses in graphite fiber reinforced epoxy composites have led us to conclude that the method could be extended into one for evaluating adhesive bonded joints. Since the X-ray measurements can reveal the point to point variation in stresses applied to an object, they should serve to map out the areas that are transferring stress from one of the bonded members to another, both with composite and with metallic adherends. A map of an adhesive joint prepared in this way when the joint is loaded should (a) provide evidence of the direct transfer of the applied stress from one adherend to another; (b) serve as a nondestructive method for revealing the outer limits of the bonded area; (c) disclose any unbonded patches within the area that are large enough to significantly alter the stress distribution; and (d) also record any stress components at the surface of an adherend that arise from the bending of adherends as a result of the loading. The method should thus be a direct and quantitative NDE method for comparison with prior discussions of these details in bonded joints,[4,5] including theoretical predictions of the stress distributions.

THE METHOD AND THE SPECIMEN DESIGN

The suggested method as applied to single lap joints reported here, involved measurement of the diffraction angles by two procedures: (1) by fitting a parabola to the diffraction peak in a standard procedure we had previously used;[2] and (2) by a more rapid technique that will be here called a "constant angle" technique. The latter involves measurement of the diffracted beam intensity at a fixed angle on a side of the peak as indicated in Fig. 1, (an angle where the slope is high and the curve is nearly a straight line). Displacement of the peak by stress then is registered as a change in intensity, with the intensity change being approximately proportional to the stress as is seen in calibration runs. A modified version of this technique, (2a), employs measurements at a pair of fixed angles straddling the peak, as indicated in Fig. 1, and taking either the ratio of the two intensities or subtracting one from the other.

Each specimen consisted of a strip cut from 1/16" or 1/32" aluminum sheet (6061-T6) about 19.7 mm wide and bonded to a similar strip or to a 6-ply

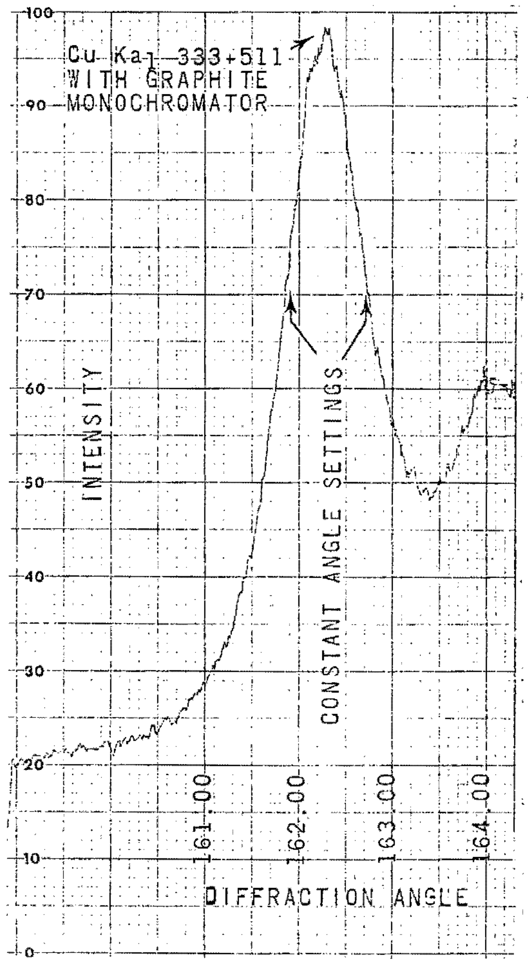

Fig. 1. Profile of diffraction peak used with aluminum. Detector is set at one or both angles indicated, for constant angle method.

unidirectional graphite/epoxy laminate to make a tensile specimen of about 152 mm overall length. Hysol EA9309 epoxy was used at the lap joint and to attach reinforcing tabs at the ends. A teflon film 0.0254 mm thick was inserted at one end of the overlap before curing. The teflon extended across the sample, with its edge perpendicular to the tensile axis, to produce a debond of accurately known position. Tension was applied through clevis grip pins in holes drilled at each end of the sample. A small, manually operated tensile frame held the specimen centered on a Siemens horizontal diffractometer and applied the load used in this and the other similar experiments reported here. An X-ray beam collimated to 0.25° divergence irradiated a spot 1 mm wide on the specimen (or in a few experiments a beam of twice this divergence and width was used). The irradiated spot covered the entire width of the specimen which was 107 mms from the slit and positioned on the diffractometer axis. High angle diffracted rays were passed through a slit 0.610 mm wide at 175 mm from the specimen and into a graphite crystal monochromator and a scintillation counter. The incident CuKα_1 radiation diffracted from the (333) and (511) planes in the aluminum adherends produced the peak recorded in Fig. 1. The one or two constant angles chosen were located about 0.40° from the maximum on the peak.

RESULTS WITH LAP JOINTS

Fig. 2 is a plot of results with the constant angle technique (2): counts per second (CPS) with a load of 10 lbs minus CPS for a load of 400 lbs applied to 1/16" (1.588 mm) thick strips of aluminum (6061-T6) and plotted vs. the X-rayed position on the specimen. The adhesively bonded single lap joint had a teflon debond built in. The 10 lb load was used to assure firm seating of the specimen in the grips and firm centering of the specimen on the diffractometer. The plotted intensities thus represent the effect of an increase in load of 390 lbs, which amounts to a change in stress in each single strip of 7.6 ksi (52.4 MPa). Increasing stress at the surface of the aluminum causes a decrease in the 10 lb minus 400 lb CPS, corresponding to an increase in the diffraction angle and a decrease in interplanar spacing of atomic planes parallel to the surface since these planes were the ones reflecting. The incident beam was collimated to 0.25° and was 1 mm wide at the specimen.

The surface stress in the upper adherend of Fig. 2 decreases as the bond is approached, because of the elastic bending which tends to throw the X-rayed surface into compression. This bending stress reaches an abrupt maximum at the edge of the bond as expected[4] and the CPS reaches a very sharp minimum here, showing that this X-ray method can locate the edge of the bond with an accuracy about equal to the width of the X-ray beam. Bending stresses also extend throughout the bonded area as can be seen in a rubber model such as the one reproduced in Fig. 3, and are superimposed on the stresses that would be transferred from one adherend to the other if bending was absent.

Figure 4 records a similar experiment on thinner aluminum strips, with data from each of the adherends. The results of Fig. 1 and the conclusions regarding the stress components arising from bending were confirmed. Again the limits of the bonded area are well marked. In this experiment merely the 400 lb count rate was measured at constant angle. The beam width was 1 mm. Irregularities in the curve within the bonded area are seen which may indicate bond defects, but no other NDE method is yet available to us to check this possibility. The 400 lb load corresponded to double the stress in each adherend that was used for Fig. 2.

The main features shown in Figs. 2 and 4 were also obtained in experiments with the sample of Fig. 2 in which the diffraction peak shifts were determined by technique (1), 5 point parabola fitting. This technique is more laborious than the constant angle technique and fewer data points were obtained, so that evidence regarding irregularities in the bond was inconclusive. However, the minima in count rate at bond limits were clearly present.

Fig. 5 records results with a 1/16" thick aluminum adherend joined to a 6-ply unidirectional graphite/epoxy laminate adherend in which aluminum powder was embedded between the first and second plies nearest the X-rayed surface. Technique (2a) was used, with CPS at 10 lbs minus CPS at 400 lbs load, and with a 1 mm wide beam. The curve of beam intensity (counts in 40 seconds) for the aluminum adherend again shows the minima at bond limits and some unidentified irregularities

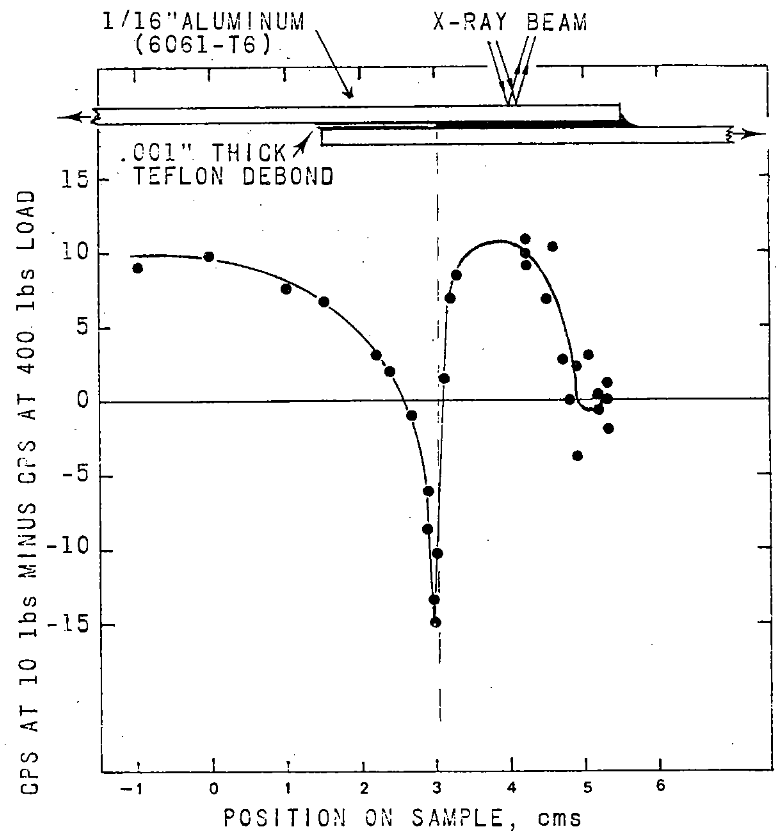

Fig. 2. Counts per second (CPS) vs. position for adhesive joint between aluminum strips 1/16" thick, with teflon debond. Constant angle method, with change in load of 390 lbs (7.6 ksi, 52.4 MPa). Increasing CPS at 10 lbs minus CPS at 400 lbs corresponds to increasing longitudinal tensile stress

Fig. 3. Rubber model of an adhesive joint with load applied, showing bending of the adherends

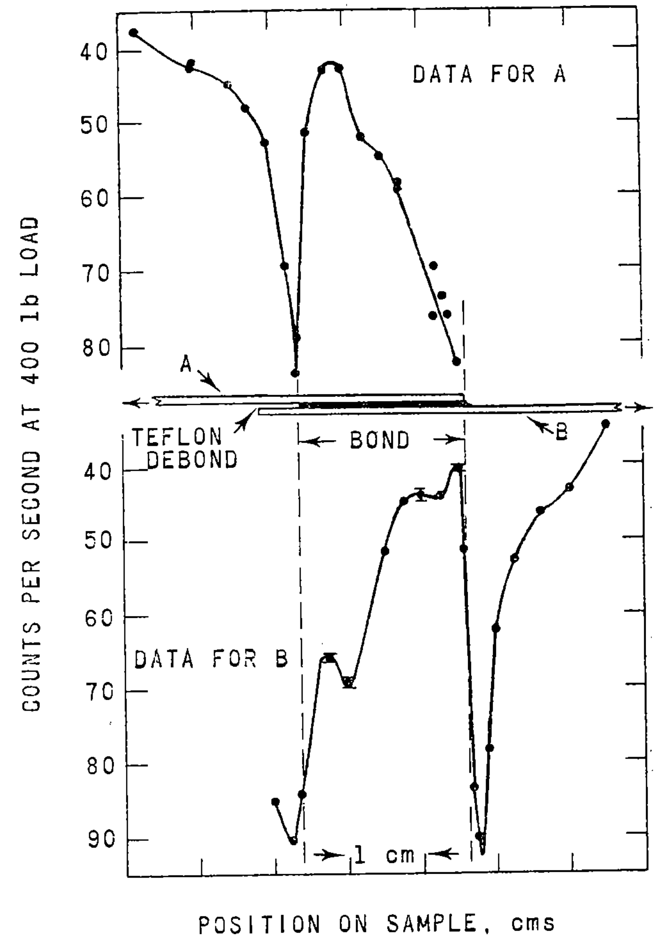

Fig. 4. Diffracted intensities for surfaces A and B of adhesively bonded 1/32" thick aluminum strips. Longidutinal tensile stress increases with decreasing count rate. Constant angle method with 400 lb load.

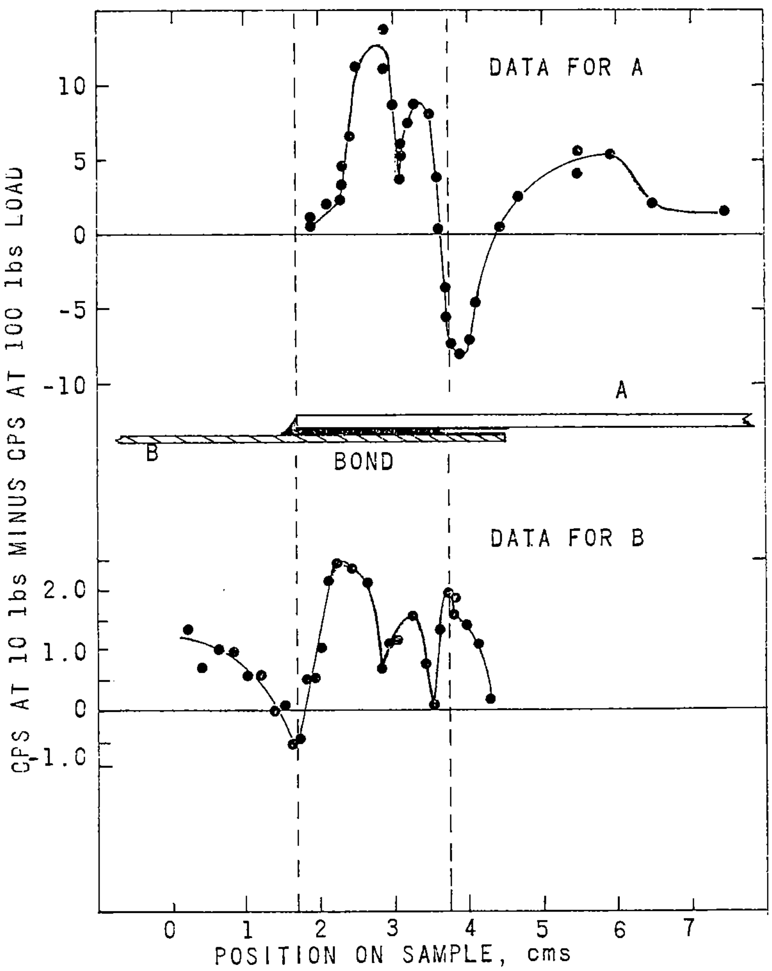

Fig. 5. Diffracted intensities for a 1/16" aluminum strip joined to a 6-ply unidirectional graphite/epoxy composite. Constant angle method.

within the bond. But the data from the aluminum-filled graphite/epoxy are very irregular. Only the minima opposite the end of the aluminum strip and the end of the bond to the composite are prominent.

Fig. 6 shows results from constant angle technique (2) on 1/32" strip bonded to the 6-ply graphite/epoxy laminate and loaded to 400 lbs. The surface stress distribution in the aluminum adherend implied by the upper plot is drawn as a solid line in the lower plot and is compared with a schematic dot-dash curve representing the stress that would be expected in the absence of any bending, if averaged throughout the thickness of the adherend. The displacement of the solid line to lower tensile stress than the dot-dash line corresponds to the stress component from bending, which is longitudinal compressive. The solid line lies above the dot-dash line where bending contributes a longitudinal tensile component. The rubber model of Fig. 3 illustrates these bends in a matched joint; Fig. 6, however, is an unmatched joint with the modulus of the composite much greater than that of the aluminum. The horizontal portion of the dot-dash lines in Fig. 6 represent the CPS obtained from a single aluminum strip, unbonded, subjected to the same load, without bending stresses.

The following paragraphs review briefly some of our research currently underway that is related to the subjects presented here.

STRESS MEASUREMENT WITH DIFFRACTING PAINT

Another X-ray method is being developed that appears to be viable for measuring applied (not pre-existing residual) stresses, and for mapping their distribution around a joint. A thin layer of epoxy paint containing a silver filler was applied to an aluminum specimen and cured. Diffraction from this paint yielded shifts in diffraction angle approximately proportional to the magnitude of applied stresses. A diffracting paint containing a diffracting filler, such as aluminum or silver powder, can be used on an object that diffracts poorly or in which a filler has not previously been embedded. The accuracies attainable in stress values in this way are apparently somewhat lower and are being investigated further. When a diffracting paint was applied to the aluminum adherend of a single lap joint and a load was applied, the limits of the bonded area were disclosed by the shifts in the diffraction angle. The onset of delamination as loads were increased was observed in a tensile

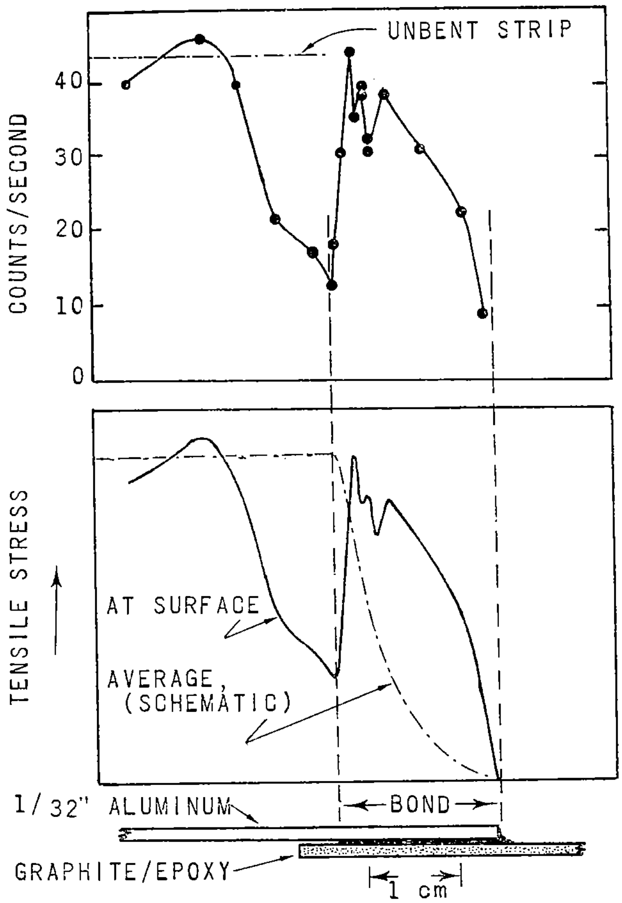

Fig. 6. Adhesive joint between 1/32" aluminum and 6-ply graphite/epoxy unidirectional composite. Upper plot: aluminum diffraction in counts per sec by constant angle method. Lower plot: surface stresses from the diffraction data compared with average stress throughout adherend cross section in the absence of bending (dot-dash line, schematic)

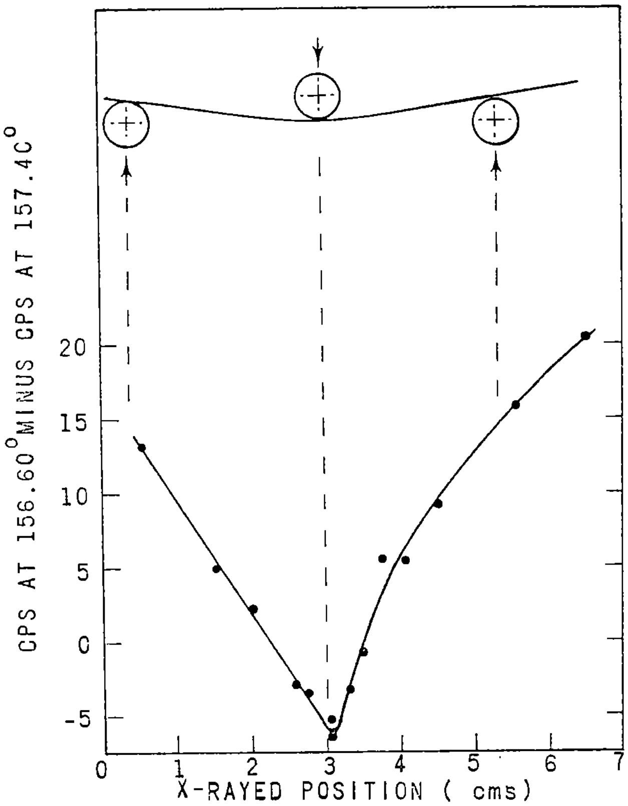

Fig. 7. Diffracted intensities vs. position on 6-ply graphite/epoxy composite after 3-point bending to initial fracture in jig sketched at the top. Constant angles method. Embedded silver powder. X-ray beam 2 mm wide.

experiment on a single composite sample. This method appears, therefore, to be useful for mapping the areas that are properly bonded and associated bending stresses and possibly for non-destructive evaluation of bond defects and of delamination. Further discussion of the method is scheduled for presentation at the 1980 Denver X-ray Conference and inclusion in its proceedings (Advances in X-Ray Analysis, vol. 24).

DETECTION OF DAMAGE IN POLYMERIC COMPOSITES

Our earlier work had suggested that if the filler yield point in a polymer composite was sufficiently low, areas of the composite where applied loads had exceeded the filler yield should be detectable from X-ray measurements of the residual stresses in the filler. A rapid test of this possibility was made by locally deforming a 6-ply unidirectional graphite/epoxy sample in 3 point bending in a jig sketched in Fig. 7. The sample contained Ag filler between the first and second ply on the X-rayed side of the bend. The deflection was increased until the first crack was heard. The sample was immediately removed, and was X-rayed on the compression side 24 hrs later and again 48 hrs later. There was no visual evidence of damage or residual bend anywhere on the compression side, but on the tension side there was a 1.5 cm longitudinal split and a short (2 mm) transverse crack and delamination. The delamination was shallow and not in the plane of the filler particles. The effects on the diffraction angle caused by the deforming were large and clearly showed the region where bending had taken place, with a maximum at the position of the sharpest bend and with effects extending well beyond the crack on the compression side, as shown in Fig. 7. A decrease in CPS in Fig. 7 corresponds to an increase in longitudinal residual tension in the filler.

Since these results show that this method has potential for revealing something of the stress history of composites and for detecting damage, it is being studied further and will be discussed in greater detail elsewhere.

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