

SCANNING ACOUSTIC MICROSCOPIC OF SINGLE FIBER FRAGMENTATION

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INTRODUCTION

High performance fibers are reinforced into polymers, metals and ceramic to produce high strength, high modulus and lower density materials. The mechanical properties of the composites materials not only depend on the constituents but also on the properties of the interface/interphase region between the fiber and the matrix [1,2,3]. It has also been established that the interphasial region determines the load transfer from the matrix to the fiber and from broken fibers to the surviving fibers. Since the overall mechanical properties, load bearing and transferring abilities of a composite decides its effectiveness, it is important to measure and understand the properties of the interphasial region. Such a study will guide in tailoring the performance of composite materials. A parameter that is often used in quantifying the properties of the fiber matrix interface is the Interphasial Shear Strength (IFSS). Several direct and indirect measurement techniques [4] have been developed in the last three decades to measure the IFSS in composite materials. Measurement of interlaminar shear strength, transverse shear strength, flexural strength, on full scale composite have been observed to be indirectly related to the interfacial shear strength. Direct measurement of IFSS are usually performed on individual fibers in a matrix. Some of the well established techniques in this category are fiber pull out[5], fiber push in[5], shear debond[5,6], microindentation[7] and the single fiber fragmentation tests[8]. Though these tests provide quantitative results they are dependent on the sample geometry and the model of the stress field around the fiber. Of all the direct IFSS measurement techniques single fiber fragmentation technique is believed to have stress transfer characteristics that are similar to that of full scale composites. Because of its simplicity it is receiving lot of attention.

In a single fiber fragmentation test a sample with a single fiber embedded in a matrix in the shape of a dog-bone is axially loaded. As the tensile load is increased the fiber fractures when the axial stress transferred to the fiber by interfacial shear exceeds the fracture strength (S_f) of the fiber. Continuation of the application of the axial load on the

sample results in the repetition of the fragmentation. Finally a stage is reached, where fiber fragment lengths become so short that the shear stress transfer along their lengths can no longer build up enough tensile stresses to cause further fiber breaks. This final fragmentation length is called the critical fragmentation length (L_c). The average interfacial shear strength (τ) is obtained using [8],

$$\tau = S_f d / 2 L_c \quad (1)$$

where d is the diameter of the fiber.

The main objective in a single fiber fragmentation test is to determine the critical fragmentation length. Optical microscopy with transparent matrices has been used with limited success for such measurements. On the other hand photo-elastic techniques have been used effectively even in partially transparent matrices. But optical techniques simply can not be used in the case of opaque matrices.

As an alternative method for opaque materials acoustic emission technique[9] has been proposed. In this technique acoustic waves generated when fiber breaks is utilized for detection and measurement of fragmentation length. The fiber break generated acoustic waves are detected by placing piezoelectric transducers on the sample. By determining the arrival time of the acoustic signals and with the knowledge of the velocity of sound in the matrix the position of the break can be detected. Simultaneous with acoustic emission and photo-elastic observation of single fiber fragmentation in optically transparent matrices have been used to validate the technique. A good agreement between the two techniques has been observed in the measurements of critical fragmentation length. In spite of such good correlation there are several difficulties in acoustic emission measurements. One of the difficulties is in developing a threshold criterion to isolate and distinguish acoustic emission signals due to fiber breakage from other sources. Another source of difficulty stems from the dimension of the sample. The samples used in these tests are fairly thin (approximately 2mm) and are comparable to the wavelength of sound in the material at acoustic emission frequencies. A combination low frequencies and small thickness enhances the possibility of generating plate mode vibrations that travel with velocities that are complex combination of longitudinal and shear waves. Thus the determination of critical length from the arrival times becomes complicated.

More recently ultrasonic shear back reflection [10] technique has been used to investigate fiber fragmentation in an optically opaque metal matrix composite of large diameter (142 μm) silicon carbide SCS-6 fibers in titanium matrix. Although the fiber fragmentations have been observed, a clear determination of the fragmentation length from acoustic images has been obscured by the low resolution. Severe limitations are encountered in extending the shear back reflection technique to the study fragmentation of smaller diameter (7 μm) fibers in polymer matrix. The frequency of the ultrasonic waves should be at least 200 MHz. At such frequencies the longitudinal wave attenuation in water and the shear wave attenuation in polymers are quite large and reduce chances of obtaining good acoustic images to perform the measurements. In an attempt to overcome the limitations of different techniques, this paper presents application of high frequency Scanning Acoustic Microscopy (SAM), for direct visualization of the fiber breaks and fragmentations, and determination of the critical fragmentation length of a 7 μm diameter carbon fiber embedded in a polymer matrix composite.

EXPEIMENTAL METHODOLOGY

Single fiber composite test samples in the shape of dog bone, were prepared as described elsewhere[11]. Two samples (Sample A and Sample B) of gauge length 30mm, width 4mm and a thickness of 2mm were selected for the study. Optically transparent matrix was chosen so that photo-elastic and SAM measurements can be compared.

Photo-elastic measurements of fragmentation in Sample A were performed by placing the sample in the grips of a load frame equipped with a calibrated load cell. Observations were made under increasing load until no further fragmentations were detected. For photo-elastic observation thickness and the surface topography, do not cause problems as long as the sample is transparent. On the other hand in acoustic microscopy both surface topography as well as the thickness limit the interior imaging capability. While the dominance of contrast due to surface roughness can mask the visibility of the fiber, large thickness of the sample could restrict the in-depth imaging ability due to high attenuation in the matrix. So, the samples were carefully polished using a special jig designed for dog bone shaped samples, to decrease surface topography and to reduce the thickness.

After photo-elastic measurements one of the sides of Sample A was polished to obtain a smooth surface and to leave approximately 200 μm thickness of matrix above the carbon fiber. Sample B was polished on both sides keeping the carbon fiber in the middle of the sample to a thickness of 400 μm . This leaves 200 μm of matrix material on either side of the carbon fiber.

Scanning Acoustic Microscope (ELSAM, Wetzlar, Germany), with a 200 MHz lens was used to obtain acoustic images. At this frequency the surface resolution of the microscope is better than 8 μm . A drop of distilled water was used between the sample and the lens to focus the acoustic waves on the sample. The distance between the lens and the sample was reduced to focus the acoustic waves at the interface between the fiber and the matrix. Acoustic images at the lowest magnification that correspond to an area of 1mm x 1mm of the sample were acquired.

Acoustic images of the fiber fragmentations in Sample A that was subjected to loading in photo-elastic measurements were obtained without further application of load. For observing the fiber fragmentation in Sample B under scanning acoustic microscope, load frame used in photo-elastic measurements was modified to accommodate thin samples and attached to the specimen stage of ELSAM. Since the scan length of the acoustic lens was limited to a millimeter, in order to obtain acoustic images of the whole sample under tension, a load was applied, held constant, and the whole gauge length was scanned before incrementing to the next load.

RESULTS AND DISCUSSIONS

Fiber fragmentations in Sample A as observed in photo-elastic experimental set up is shown in Fig. 1. Fiber breaks can be recognized due the birefringence effect. In order to detect these breaks the sample must be under load. A bright spot, away from the fiber can also be noticed in this image. This is either a trapped air bubble or a pore that formed during the consolidation of the composite. The first fiber break in this sample was observed at a load of 40 MPa. Several subsequent breaks were observed and the average critical fragmentation length was determined to be $(341 \pm 52)\mu\text{m}$.

Fig. 2 shows an acoustic image of a fiber fragmentation in Sample A of the same region as in Fig. 1. Contrary to the photo-elastic observation there is no necessity of loading the

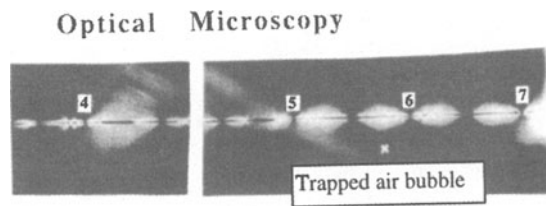


Fig.1 Photo-elastic image of the fiber fragments.

sample to detect the fiber breaks in acoustic imaging. The contrast in the acoustic image, of the break and of the fiber in the matrix are different compared to optical image. In photo-elastic imaging birefringence due to anisotropic stress in the sample helps in detecting the fiber breaks. On the other hand in acoustic microscopy it is the variation in the acoustic impedance along the fiber length that enhances the detection of the fiber breaks. When the lens is over the position of the break the acoustic waves propagating through the matrix encounter a void in the region. The change in the acoustic impedance is very large and also the phase of the incident wave is reversed. So, the fiber break region appears very bright. On the contrary, when the lens is over the region where the fiber is present and still in contact with the matrix, the acoustic waves propagating through the matrix encounter a small change in acoustic impedance and phase change. Since most of the acoustic energy is transmitted through the interface, very little energy is reflected back to the lens. This makes the region appear much darker than the break region. Using the acoustic images the average fiber fragmentation length was determined to be $(235 \pm 56) \mu\text{m}$. The pore or the trapped air bubble observed as a bright spot in photo-elastic image is also observed in the acoustic image. A large change in acoustic impedance and phase reversal makes it appear as a bright spot. The average fiber fragmentation length measurements performed on the same sample using photo-elastic images is much larger than SAM images. This means that the SAM observations showed more fiber breaks. Indeed a careful observation of SAM images show more fiber break like features than photo-elastic images. One such feature is indicated in Fig.2. It is known that if this were to be a fiber break then the photo-elastic images should produce a birefringence contrast. Assuming that these fiber like features observed in SAM are not really fiber breaks, the average critical fragmentation length calculated ignoring these features comes out to be $(356 \pm 56) \mu\text{m}$. this is good agreement with photo-elastic measurements.

Although ignoring the break like features in SAM images and measuring the fragmentation length gives good agreement with photo-elastic measurements the contrast of these features confuse the identification and determination of the critical length. Hence we decided to follow the processes of fiber fragmentation while loading the Sample B in the scanning acoustic microscope so that their origin could be investigated. Acoustic images of Sample B obtained without any load and load smaller than 15 MPa showed uniform fiber matrix interface without any features. As the load was increased features similar to the breaks appeared at several locations simultaneously. From photo elastic measurements it is known that the first fiber break occurs at 40 MPa. Also, only one break is possible at a given load. A load of 15 MPa is too small to produce a single fiber break and it should be impossible to produce multiple breaks. Hence the features observed in the acoustic images are not due to fiber breaks. One of the reasons for the appearance of these features is probably due to the debonding of the fiber matrix interface. It is possible that in some regions along the fiber matrix interface the fiber may be just in

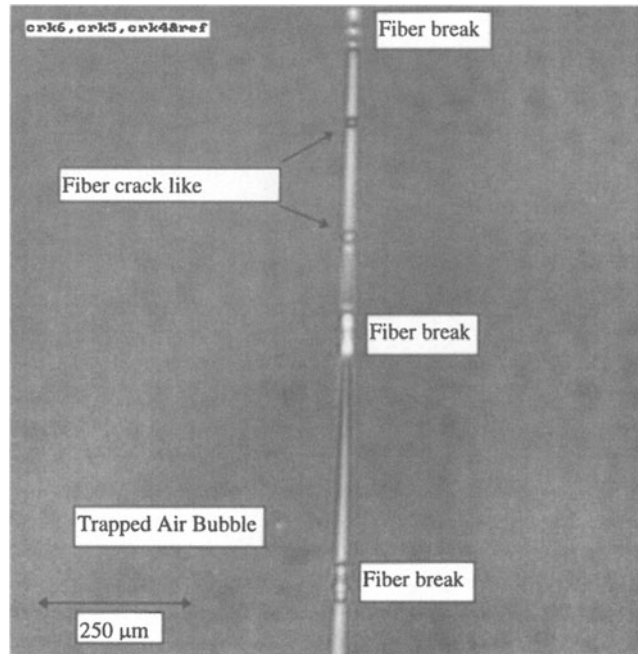


Fig. 2. Acoustic image of the fiber fragments.

physical contact with the matrix and there may be lack of chemical bonding. This is commonly known as “kissing bonds” in the field of ultrasonic examination of adhesive bonded structures[12]. Because of the physical contact the stress transfer is relatively good and this makes the detection of such disbonds extremely difficult in an ultrasonic examination. However, the situation changes as soon as a load is applied. The two regions get separated and an interrogating ultrasonic wave encounters a huge change in acoustic impedance. A similar situation is observed in the SAM examination of Sample B. The regions along the fiber matrix interface which were not chemically bonded were separated at a load of 15 MPa. At these regions the focused acoustic waves encountered large changes in acoustic impedance and phase reversal. These regions look as bright spots in acoustic images.

Further increase in the load does not show change in the dimensions of these features. When the load reached about 40 MPa, new features appeared and this was identified as the first fiber break. Observation of the fiber matrix debonding before the occurrence of the first fiber break in a single fiber fragmentation test is very important for understanding the acoustic images. Because of this reason the average critical fragmentation length in Sample A determined from SAM images were found to be in good agreement with the photo-elastic measurements when the fiber like regions were ignored.

CONCLUSIONS

This paper demonstrates the applicability of scanning acoustic microscope to study single fiber fragmentation tests. The critical fragmentation length measured using scanning acoustic microscope is in good agreement with photo-elastic measurements. SAM can reveal the fiber matrix debonds that may be present in the sample and which may open up during the loading processes. Though the experiments have been performed on transparent samples SAM technique can be applied for characterization of opaque materials.

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