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SPECIMEN DEVELOPMENT AND FAILURE MODES INVESTIGATION IN IOSIPESCU INTRALAMINAR SHEAR TEST

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SUMMARY: A method of measuring interfacial strength for bulk polymer composite materials has been investigated in this paper. It was found that there is no such unified sample dimensions existed for all of the composite materials, which can achieve a pure shear failure in between fibers and matrix in the Iosipescu test, since the adhesive force between the fibers and matrix may differ. In our interfacial study, to avoid a non-uniform shear stress with compressive failure in the nylon6/glass fiber composite shear tests, the length between the two notches tips and the other dimensions were varied. The failure modes and mechanisms of the test were studied by using SEM technique. The whole test process was monitored by a microscope to see the loading and shearing failures.

KEYWORDS: failure modes, Iosipescu shear test, specimen preparation

1. INTRODUCTION

The strength of the interfacial bond plays a very important role in determining the properties of composite materials. A strong interfacial bond gives composites high strength and stiffness. There are many mechanical tests, which can be used to characterize the strength of the interfacial bond. The most common are the single fiber pullout/microdrop technique [Broutman, 1969, Gaur et al, 1989 and Baillie and Bader, 1994], the embedded single fiber test [Kelly and Tyson, 1965, Baillie and Bader, 1993 and Buxton and Baillie, 1996] and the microdebonding/microindentation technique [Mandell et al, 1980 and Herrera-Franco et al, 1992]. For bulk composites, there are the short beam shear test [Newaz, 1984 and Vu-Khanh and Denault, 1991], the transverse tensile test [Caldwell, 1990], the transverse flexural test [McHugh et al, 1991] and the Iosipescu shear test [Curtis et al, 1987 and Iosipescu, 1962].

However, selection of the methods to measure the interfacial strength is still a very controversial topic in composite interfacial studies. For instance, the failure mechanisms and failure modes can be very complex in these tests. The test results can be difficult to interpret since failure in the tests may not be due to debonding (adhesive failure) in the interfacial layer, i.e., separation may not occur between the fiber and matrix. Rather, it could occur within a fiber, or within the matrix (cohesive failure). Therefore, none of these tests could really be described as a simple and reliable test, or appropriate for the determination of the interfacial bonding strength for all fiber composites. It is always wise to select more than one method in the interfacial strength study for comparing and verifying the results. Furthermore, SEM technique can be useful to interpret the results and determine the failure mode and analyze the failure mechanisms.

However, from a practical point of view, it may not matter whether the failure during the test is adhesive or cohesive, as long as the strength of the weak link at the interface is being measured. Thus the test result can be denoted interfacial bonding strength, regardless of the failure mode.

In our serial interfacial studies, a longitudinal intralaminar Iosipescu test was employed to measure interfacial bonding conditions of the bulk nylon6/GF composites subjected to different cooling rates [Cartledge and Baillie, 1999]. Values of the shear strength were used to indicate the effect of the cooling rate on the interfacial shear strength of the composites. For comparing the interfacial strength trend results from the Iosipescu test, a transverse flexural test was used in the study as well.

2. IOSIPESCU TEST

The Iosipescu test has been widely used to identify interfacial bond strength of bulk fiber composite materials for more than last two decades. This test method was originally proposed by Nicolae Iosipescu of Bucharest, Romania in the early 1960's [Iosipescu, 1962]. In 1967 [Iosipescu, 1967], Iosipescu published a detailed study involving the development of the asymmetrically loaded notched beam test to measure the shear strength of isotropic materials (metals). His approach involved using a strain gauge at the center of a beam specimen, at the point where the asymmetric loading caused pure shear stress with no bending moment. At this point, 90° notches were introduced at the top and bottom of the beam to modify the classical parabolic shape of the shear stress distribution to a more uniform distribution.

Thomas Place of the Aeronutronic Division, Ford Aerospace and Communications Corporation first applied this test method to composite materials, in the early 1970's. It was used to test three-dimensional reinforced ceramic matrix materials [Place, 1974]. Later, in 1977, the Composite Materials Research Group at the University of Wyoming used this test method to test three dimensional reinforced carbon-carbon composites [Walrath and Adams, 1979]. Since then, Walrath and Adams have used it to test a wide variety of composite materials, ranging from unidirectional glass fiber reinforced epoxy and graphite/epoxy, to chopped glass fiber reinforced polyester sheet molding components, and even materials such as wood and foam. They have successfully introduced a fixture that extended the Iosipescu test method to composite materials [Walrath and Adams, 1983]. During the past decade, the test method has been widely used to measure the intralaminar and interlaminar shear strength [Barnes et al, 1987 and Broughton et al, 1990].

The principle of this test is that a double-V-notch specimen was employed and through the action of a force couples, a state of pure shear in the middle length of the specimen are achieved. The force couples produce counteracting moments and that the moments exactly cancel at the middle length of the specimen. A constant shear force is induced through the middle section of the specimen, thus producing a pure shear loading at that location (see fig1).

The ASTM has now completed work on a standard for the Iosipescu test, ASTM 5379M. However, in practice the specimen geometry dimensions specified by ASTM may not suit all composite materials, since the adhesive force between fibers and matrix may differ. There may be some non-uniform shear stress distribution in the test section, probably arising from small normal compressive stresses. In our study, it was found that it is possible to prevent specimens failing in the compression by changing the geometry dimensions. The length between the two notches' tips and the other dimensions can be varied to achieve pure shear failure in the test, rather than non-uniform shear stress with compressive failure.

3. SPECIMENS PREPARATION AND DEVELOPMENT

The double-V-notch specimen preparation was a very important part of the Iosipescu test. The specimen has to be prepared with the right notch angle, depth and radius to achieve a pure shear failure during the test. Otherwise, the specimens will be compressively crushed at the loading zone as figure 2 illustrates. There are maybe many ways one can achieve a pure shear failure in the test. In our study we used one of them to optimise specimen performance for the particular composite which was to vary notch depth in the specimen, and observing failure modes and performance under a microscope during the test.

The Iosipescu test method is still being modified and developed since it was introduced into the composite shear property study by Place 30 years ago [Place, 1974]. The following few paragraphs are presented to discuss how the Iosipescu specimens were developed to achieve a pure shear failure in our nylon6/glass fiber interfacial study.

The specimens were cut with fibres parallel to the longitudinal axis-0 degree from a bulk 200 mm X 200 mm nylon6/GF composite. The specimens were 80 mm long, 20 mm wide and 4 mm thick. Two 90° notches were cut at the specimen midlength with faces oriented at +45° and –45° to the longitudinal axis. The notches were introduced using a vertical milling machine Pacific FTV-2S with a specially designed parallel fixture. Special care was taken to use optimum cutting rates. A freshly-sharpened cutting tool was used to minimise pre-test damage and produce a sharp notch. The notch tip radius was about 120 μm.

To measure the width between the two tips of the notches accurately two dowel pins were used to fit in the two notches and the length L was measured by a vernier. The width between the two notch tips can be calculated as the geometry shows in the figure 3.

$$L_t = L_d - (b_1 + r_1) - (b_2 + r_2)$$

Where

- L_t = width between two tips of the notches, mm
- L_d = length between two dowel pins in the notches, mm
- r_1 = radius of the dowel pin 1, mm
- r_2 = radius of the dowel pin 2, mm
- $b_1 = r_1 \sin 45°$, distance between the centre of pins 1 to the tip of notch 1
- $b_2 = r_2 \sin 45°$, distance between the centre of pins 2 to the tip of notch 2

The width between the two notches' tips varied from 12 mm down to 3 mm to find the best pure shear failure mode in the test. As it can be seen in figure 4, the width between the two notch tips was 12 mm as ASTM specified. There was a very clear compressive damage line under the loading zone, which was next to the top notch. This means the failure mode of this specimen was compressive failure instead of pure shear failure.

To avoid the compressive failure in the 12 mm tips-width specimen, a thin steel sheet was glued on each side of the loading zone. One was attached on the top of the right side of the notch, and the other one was attached on the bottom of left side of the notch to reinforce the composite as shown in figure 5. However, it still failed in compressive crushing.

Next, a steel channel section sheath was glued on each side of the loading zone, but the specimen still failed in compression (see figure 6). It was found that a few slight shear deformed lines appeared between the tips of the notches and were accompanied with compressive failure. Figure 7 indicates that 9 mm tips-width specimen with no reinforcing fixture attached on produced a similar failure as the specimen in the figure 6.

Reducing the tips-width to 5 mm with no reinforcing fixture attached on, produced shear flowing lines between the two notches' tips, as seen in figure 8. Shear failure appeared first and then followed by an ultimate compressive failure at the loading zones. Therefore the ultimate failure data was still not reliable for measuring the maximum pure shear strength in the composite.

Figures 9 and 10 show that the failure in specimens with tips-width 4 mm and 3 mm, which were no reinforcing fixture attached. The failure modes of the specimens were pure shear failure in the specimens as the photos show. However, it can not be guaranteed that the shear stresses will uniformly distribute in the middle section of the specimens, especially for the 4 mm tips-width specimen. Nevertheless, those specimen geometrises have been found to yield reliable shear strength data falling into the typical acceptable pure shear failure modes as ASTM specified. Thus the optimum tips width for this particular composite should be 3 mm. The shear dislocating lines in this test were found as opposite to the ASTM stated in its failure mode of the unidirectional 0° specimen (ASTM D5379-93, figure 11, 1993). Figure 11 illustrates the difference between this test and the failure mode of ASTM D5379-93.

4. EXPERIMENTAL TECHNIQUE

Eight 0° Iosipescu specimens with the optimum 3 mm tips-width, were tested at a constant cross-head displacement rate of 2 mm/min according to ASTM D5379/D5379M-93 and were carried out on an Instron machine at room temperature. A 10 KN load cell was used to collect the signal of the load and displacement in the test and then logged to a computer to be processed.

The test fixture was based on the original Wyoming University test fixture developed by Walrath and Adams, and made by this research centre (see figure 12). Iosipescu tests in this study were performed under monotonously applied compressive loads normal to the longitudinal axis of the specimen. A microscope observed the mechanical dynamic response of the tests.

A state of pure shear was achieved within the mid-span section of the Iosipescu test specimen by applying two counteracting moments, which were produced by two force couples-P. From the simple statics considerations, the shear force acting at the centre of the cross section was equal to the applied force as measured from the load cell. There was no stress concentration at the sides of the notches of the specimen, because the normal stresses were parallel to the sides at that point of the specimen. Consequently, the shear stress was obtained by simply dividing the value of the applied force by the net cross-sectional area between the two notches' tips. The shear stress can be calculated as

$$\tau_i = P / A$$

where

- τ_i = shear stress at ith data point, MPa
- P = load at ith data point, newton
- $A = L_t T_b$, cross-sectional area, mm^2
- L_t = width between the two tips of the notch, mm
- T_b = thickness of the beam specimen, mm

The fractured surfaces were examined by using a Scanning Electronic Microscope. The specimens were sputter coated with a thin layer of gold in order to improve the resolution of the specimen prior to SEM examination. For the low resolution of the GF/PA6 an accelerating voltage was selected at 20 kV and the condenser lens (spot size) was selected as 100 nm. Both backscatter and secondary electron detectors were used.

5. MICROSCOPY OBERVATION OF THE DYNAMIC RESPONSE

A microscope was used to observe the mechanical dynamic response of the Iosipescu test. The dynamic response of the 0 degree oriented specimens was initially linear as figure 13 shows. The on-set of a non-linear behaviour occurred at approximately 0.7 KN. First axial splitting under the notches (at the tips) were observed, manifested by load drop appearing at the load-displacement curves (0.8 KN). The axial crack tended to initiate at the notch root and propagate parallel to the axis on one side of the notch tip and opposite to the loading points (see figure 10). The axially aligned fibres stopped the growth of the cracks in the principal stress plane, 45° to the axis. The shear stress concentration at the notches' tips was primarily responsible for the cracking initiation. These cracks relieved the local stress concentrations at the notch tips.

The actual stresses associated with the initiation and propagation of the cracks in the tips area were complex. At the first stage, the initial crack propagation was primarily associated with mode I fracture. As the crack extended, the mode II contribution to the fracture increased. These cracks were stopped as they propagated into low stress regions.

Subsequently, a few cracks along the fibre orientation appeared between the notches' tips in the specimen mid-section. The load drops again significantly, which was termed as secondary failure at 1.1 KN in the test. The cracks consisted of numerous short interfacial cracks. They were manifested in the SEM photos and will be discussed in the next section. The cracks propagated into both directions along the fibres and finally caused ultimate failure at 1.45 KN in the specimen. The shear strength at the failure points is 91.7 MPa for secondary shear failure and 120 MPa for ultimate failure. The interfacial shear strength from this study and tensile strength from flexural study are listed below:

Sample	Flexural test tensile strength ó_{max} (MPa)	Iosipescu test secondary shear failure strength ô_{s} (MPa)	Iosipescu test ultimate shear failure strength ô_{ult} (MPa)	Flexural test modulus E (GPa)
Nylon6/GF	43	91.7	120	4.3

In the next section, SEM photos will tell us whether the failure was an adhesive failure or cohesive failure at the interface zone. However, the information obtained (strength of the weak link) from the Iosipescu test still can give us a general idea of the mechanical and interfacial bonding conditions of the nylon6/GF composites regardless where the failure occurred.

6. SEM STUDY OF THE FRACTURE SURFACE

From the stress state analysis of the V-notch root axial splitting section, the stress states at this point was tensile stress as mode I fracture dominated the damage zone. It can be seen in SEM photos (see figures 14 and 15). Figure 14 shows that the fracture surface features of the axial splitting section were similar to the flexural fracture surface, which was typical mode I fracture (figure 15). It has been seen in the both SEM photos taken from flexural and Iosipescu fracture surfaces, that the matrix was torn off from the fibre surface vertically to the fibre axis which means mode I fracture contributed to the damage zones in both tests. Thus, the first axial splitting stress may give information of the tensile strength of the interfacial bond of the composites.

Subsequent to the axial splitting, secondary failure and ultimate failure followed with increasing load. The failure modes of the latter (secondary and ultimate) were pure shear failure. It can be verified by the SEM study of the midplane cracking section, figure 16. The surface feature shows typical shearing plastic deformation. The matrix was torn off and plastic flow 45° to the fibre axis approximately as shown in figure 16. Thus, the secondary failure stress and ultimate failure stress may directly indicate the shear strength of the interfacial bond of the nylon6/GF composites.

From the microanalysis of the SEM observations, it is verified that the Iosipescu shear strength could, in a direct way, relate to the interfacial shear strength. However, the tensile strength from this test may relate to the interfacial tensile strength indirectly due to the fact that the axial splitting involved both modes I and mode II fractures in this area. The actual stress state was very complex in the axial splitting section area.

7. CONCLUSION

The study of the V-notches specimen development showed that to achieve a pure shear failure in the Iosipescu test, the critical width between the two tips of the V-notch was 3 mm for these particular composite samples. SEM study of the fracture surfaces showed that the shear rupture occurred between the glass fibres and nylon6 matrix in the midplane section.

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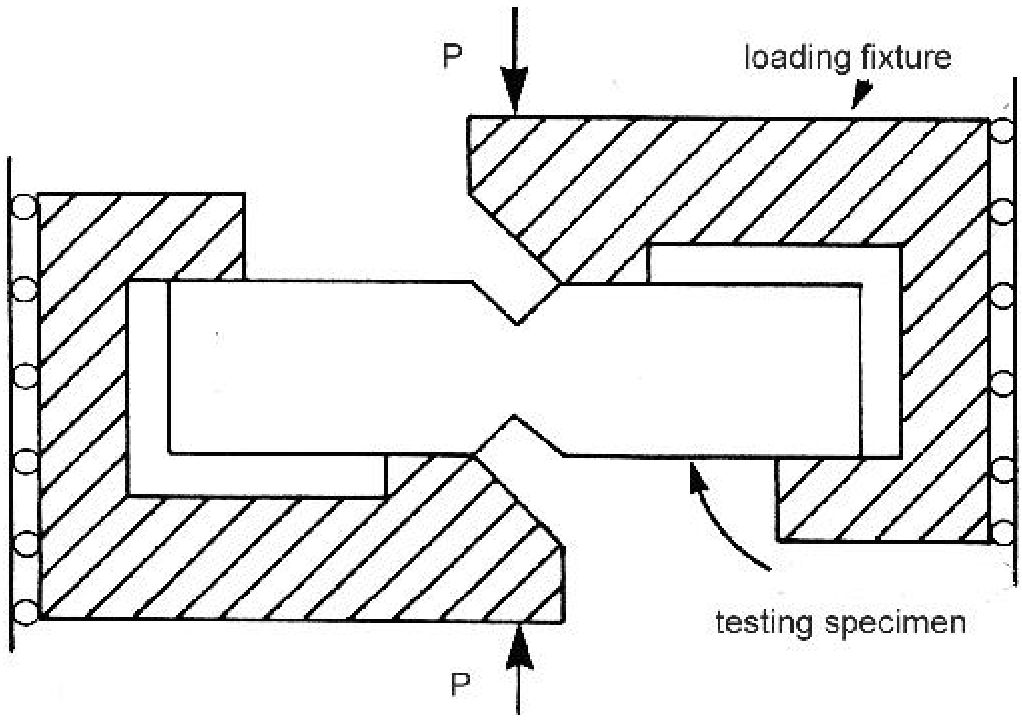

Figure 1. Diagram of Iosipescu test for 0 degree GF/PA6 composite specimen

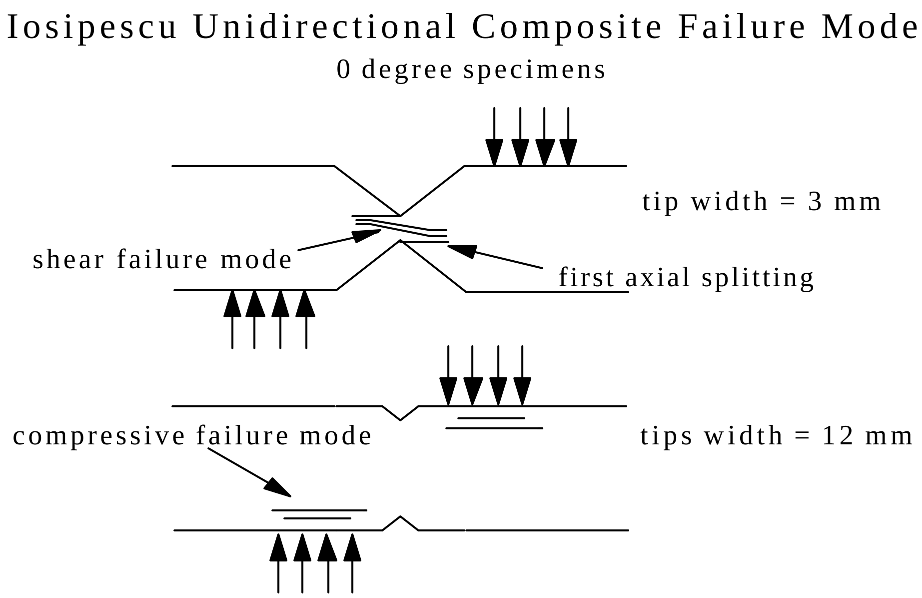

Figure 2. Illustration of Iosipescu test failure modes with different tips width

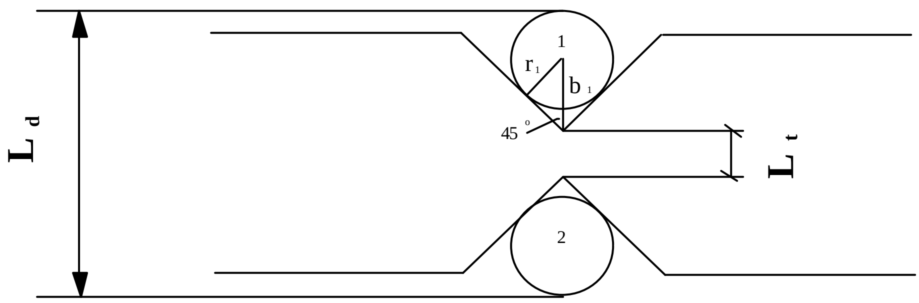

Figure 3 Measurement of the tips width of Iosipescu specimen

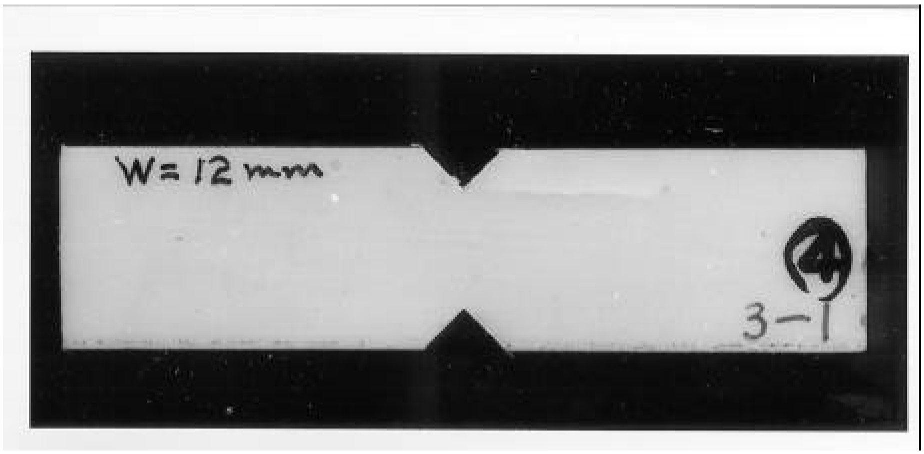

Figure 4 Iosipescu specimen with 12 mm notches’ tips-width and without reinforcing fixture

Figure 5 Iosipescu specimen with 12 mm notches tips-width and a thin steel layer reinforcing fixture glued on each side of the loading zone

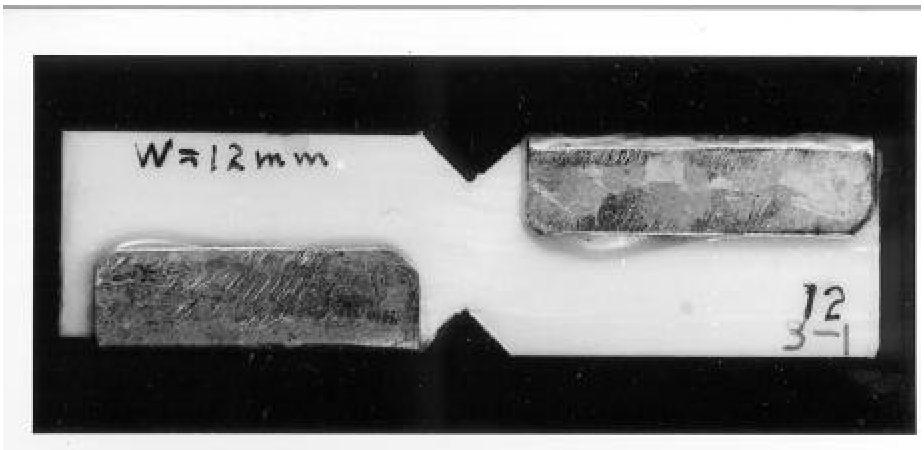

Figure 6 Iosipescu specimen with 12 mm notches tips-width and a thick steel channel section sheath glued on each side of the loading zone

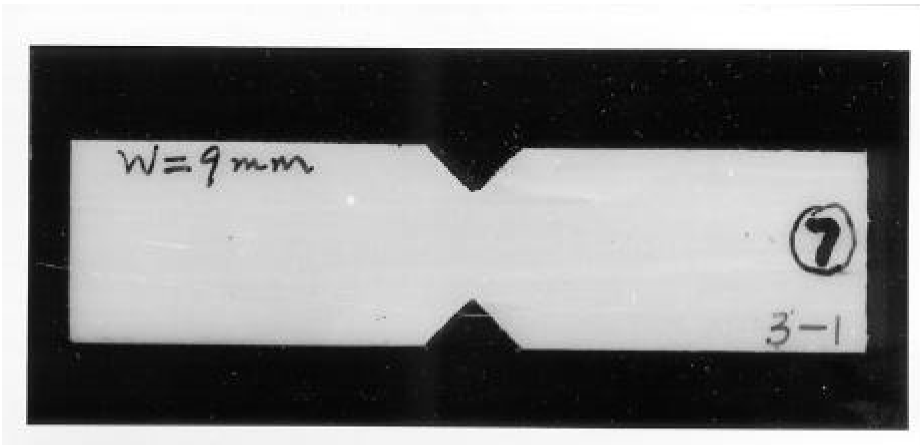

Figure 7 Iosipescu specimen with 9 mm notches tips-width

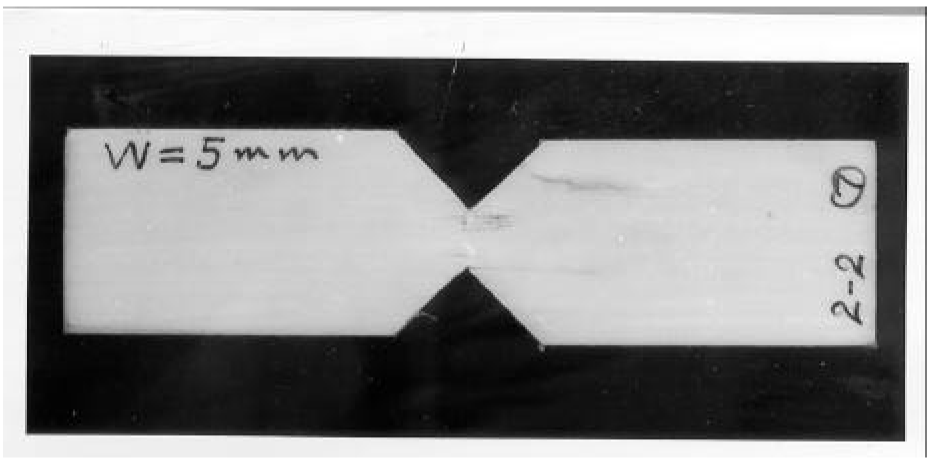

Figure 8 Iosipescu specimen with 5 mm notches tips-width

Figure 9 Iosipescu specimen with 4 mm notches tips-width

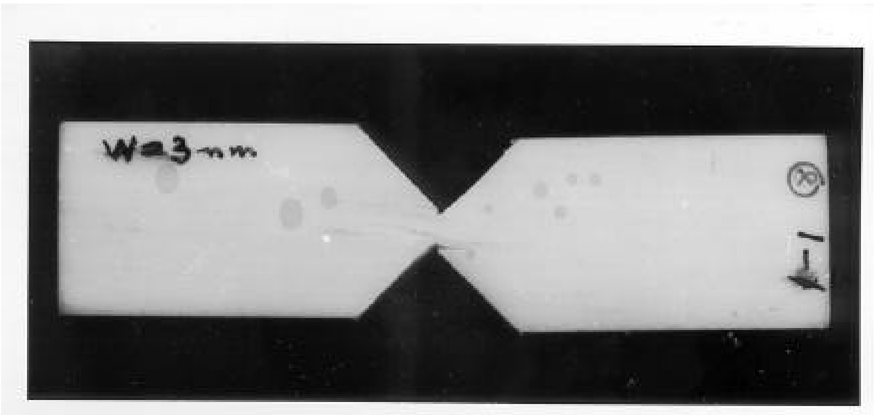

Figure 10 Iosipescu specimen with 3 mm notch tips width

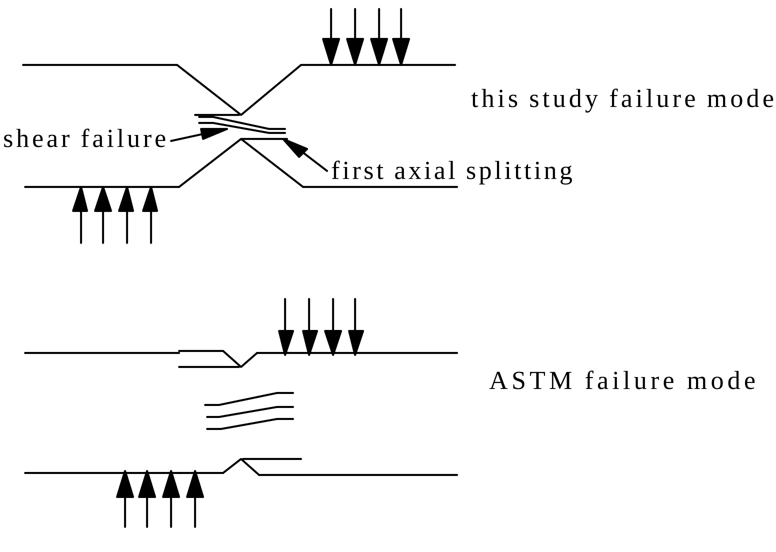

Figure 11 Comparison of unidirectional 0° composite specimen failure modes in Iosipescu test

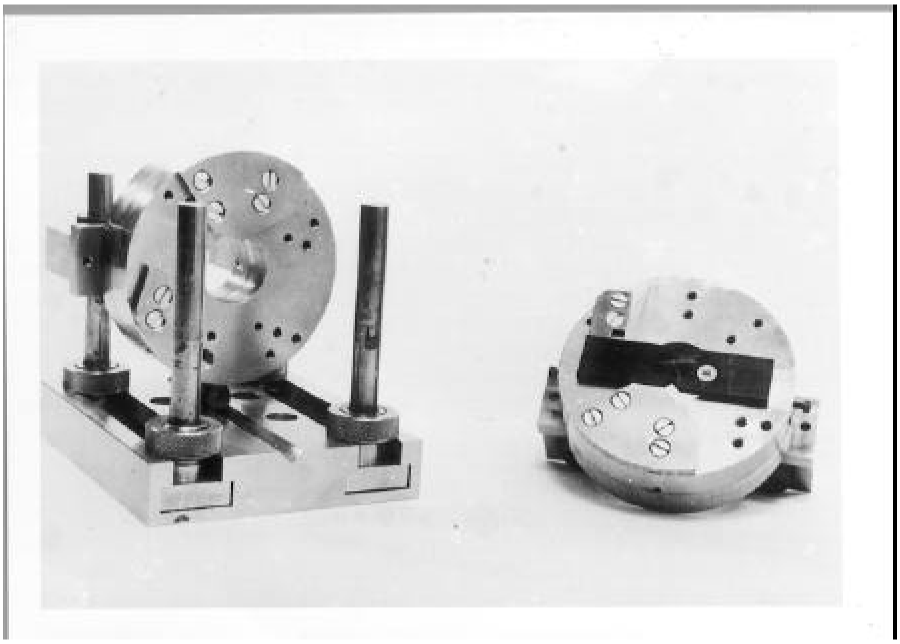

Figure 12 Iosipescu test fixture

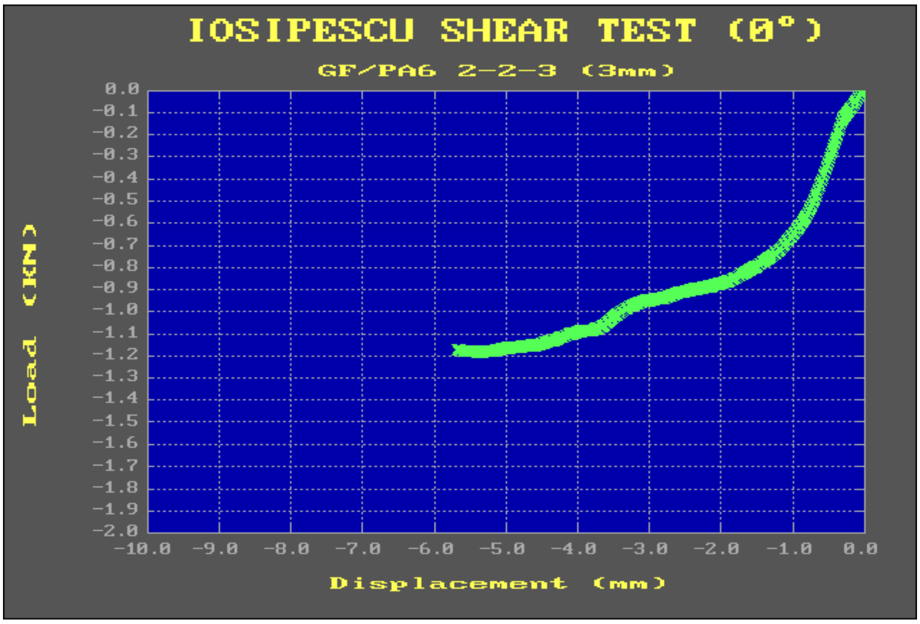

Figure 13 Load-displacement curve of air cooled 0 specimen in Iosipescu test

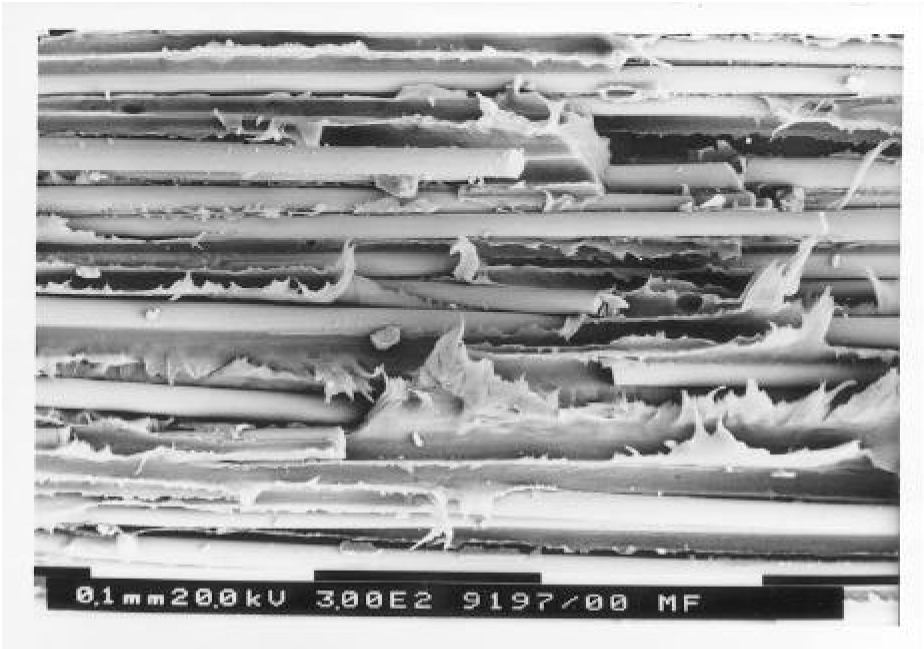

Figure 14 SEM photo of Axial splitting section fracture surface of Iosipescu test

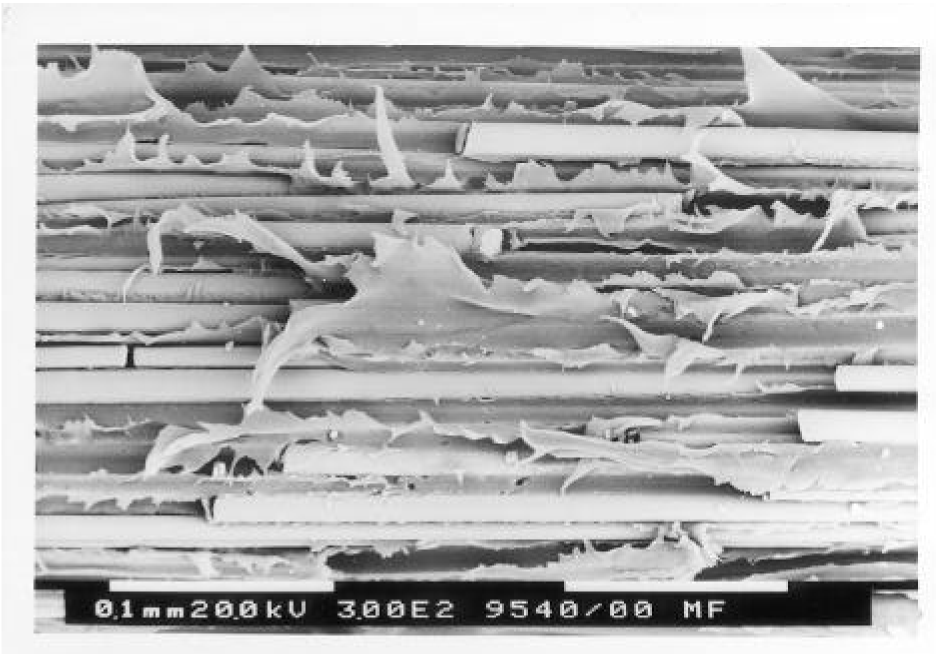

Figure 15 SEM photo of midspan section fracture surface of flexural test

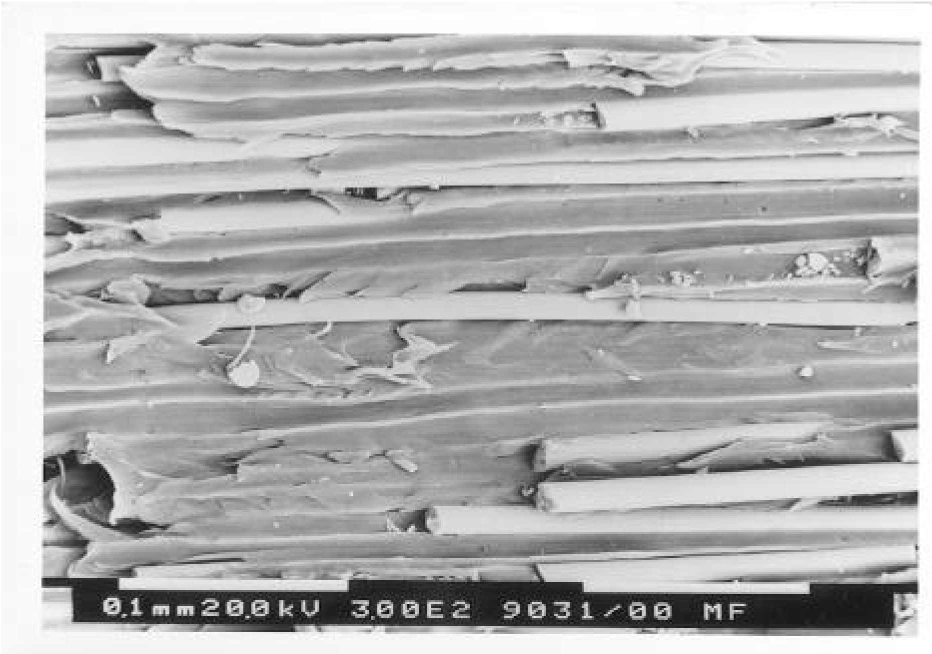

Figure 16 SEM photo of midplane section fracture surface of Iosipescu test