FABRICATION AND NDE OF MULTI–WAVE THERMOSET GR/EP COMPOSITES

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INTRODUCTION

The use of composite materials is proliferating these days. Their excellent strength–to–weight ratio has been the major reason for their popularity. Other reasons include: tailoring of desired properties, excellent corrosion properties, etc. Use of composites in underwater applications such as submarine hulls, due to their stealth properties, is becoming very attractive. Composites absorb sonar rays, making it difficult to detect submarines. Composites are also being used as pressure vessels and missile bodies. In these applications the structural shape is cylindrical or a surface of revolution. Very large composite cylinders with thickness of 3–4 inches have been manufactured for a variety of applications. These cylindrical shapes, in general, are manufactured by filament winding process. Continuous fiber is dipped in the adhesive and is wound on a mandrel. The fiber has to be under tension for good adhesion and reduced porosity. The cure process is an exothermic reaction and due to fiber tension and cooling, compressive stresses develop in the cylinders. It has been observed that due to these stresses the cylinders develop waviness in the fibers. It is not difficult to see that this waviness in fibers will result in reduced strength, of composites in compression, Garala [1], and increased fiber matrix debonding in tension. Hyer et al. [2] and Telegadas and Hyer [3] have studied the effect of fiber waviness on the stress state in hydrostatically loaded cylinders. The sample used by them had a single wave in a thermoset composite. The civil structural industry is finding new ways to use the composites. Small 3 inch long glass fiber wavy composites, wave length about 1 mm, are being tested as inclusions in reinforced concrete. The motivation for this stúdy comes from the above applications of composites.

We describe here the development of a technique for the fabrication of multi–wave graphite/epoxy composites. These composites samples were fabricated by a two–step cure process. In the first step a wavy laminate was produced and was partially cured so that it retain the wavy shape. In the second step the wavy lamina was assembled with flat composite panels on both sides and co–cured to produce the laminate with one wavy sublaminate. In the first step of fabrication the partial cure temperature was decided based on a series of fracture testing of flat samples which were fabricated by the two step process but with varying first step temperature. The fracture tests were performed to arrive at the best partial cure temperature. Due to the partial polymerization in the first step, the second step cure between the plies may not be as good as the one cycle cure. This can result in a sample which would not be a true representative of the ac-

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tual material. The fracture toughness of the two–step cure interface is one criteria which can be used to estimate the 'goodness' of the interface. The complete two step fabrication process of the composites and the results of the G_{IC} testing of the flat samples are presented here.

After it was assured that the composites were of satisfactory quality, they were subjected to damage by compressive loading. Micrographic pictures show damage as crushing and debonding in the cross–plies.

The samples were tested by ultrasound before and after damage and a very interesting picture emerges. The waves in the composites can be seen very 'clearly' by the ultrasonic C–scan and as the damage is introduced, the picture becomes complicated. We present here the theoretical results of the wave propagation in a wavy composite. Some results of the compression testing and ultrasonic nondestructive testing of wavy composites are also presented.

SAMPLE FABRICATION

The samples were fabricated in a two step process. In the first step a zero degree laminate with waviness is produced. This is done by the fixture as shown in Fig.1.

The fixture has wires of diameter 1.2 mm laid out straight with a spacing of 12 mm on an aluminum plate of 12 mm thickness. The wires are kept tight and straight by fixing them with screws on the side of the plate. The two plates are aligned together by two guide pins on the opposite corners of the plate. Five 150 mmx150 mm unidirectional laminas are prepared and stacked together with all fibers running in the same direction. Next, this layup is put between two of the fixtures such that the fibers are oriented normal to the direction of the wires thus producing the waves in the fibers. A piece of bagging material is placed between the layup and templates to prevent the adhesion between the two. At this stage no bagging is needed because the temperature is not increased to an extent that the epoxy may start flowing. The entire assembly is cured at an intermediate temperature. The value of this intermediate temperature will be discussed later. At this stage the wavy lamina, Fig. 2 a, is in a semi cured state and easy to handle. Two more laminates, each 150 mmx150 mm, 5 ply thick but flat are prepared. The waves in the wavy laminate are filled with uncured strips of unidirectional prepreg as shown in Fig. 2 b. The amount of the fibers is carefully measured and equal amounts are placed in each wave for the uniformity of the laminate. The two partially cured flat laminas are now placed on the two sides and the entire assembly, Fig. 2 c, is bagged and cured as per the temperature cycle shown in Fig. 3.

To test if the bonding between the plies in the two cure processes is good, several flat specimen were prepared by the same two cure cycles. All the laminas in these samples were flat. Five ply thick 150 mmx150 mm unidirectional laminas were prepared. These test specimens were partially cured at 65^0, 80^0 and 93^0 C (150^0, 175^0, 200^0 F). These partially cured laminates were then put together with a strip of teflon tape between them running along the edge on one side, as shown in Fig. 4, to simulate a crack in the finished sample. The layup now could be termed as $[0_5]_s$. They were then bagged by the usual process and co–cured at 190^0 C (375^0 F), with the same temperature cycle as shown in Fig.3. The panel was then cut into specimen of 38 mmX250 mm and door hinges were installed on the side which has the simulated crack. The

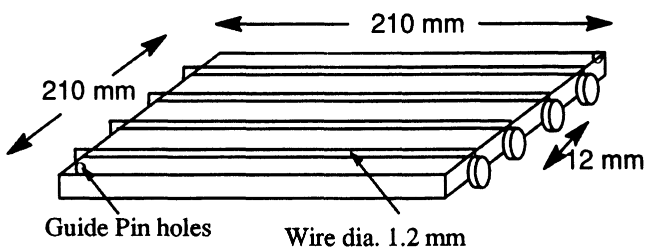

Fig. 1 Template to fabricate a wavy lamina.

details of the final sample, which is also called the 'double cantilevered beam (DCB) sample are shown in Fig. 4. Three samples were prepared where the double cure cycle was not followed and the sample was cured through the regular cure cycle without interruption to prepare the reference samples.

RESULTS AND DISCUSSION

As mentioned above, the first set of tests was performed to determine if the double cure process in the manufacture of these composites was appropriate or not. The question to be answered was: How does the fracture toughness of the specimen which are double cured compare to the reference specimen ? To answer this the specimens were subjected to double cantilever Mode I fracture testing.

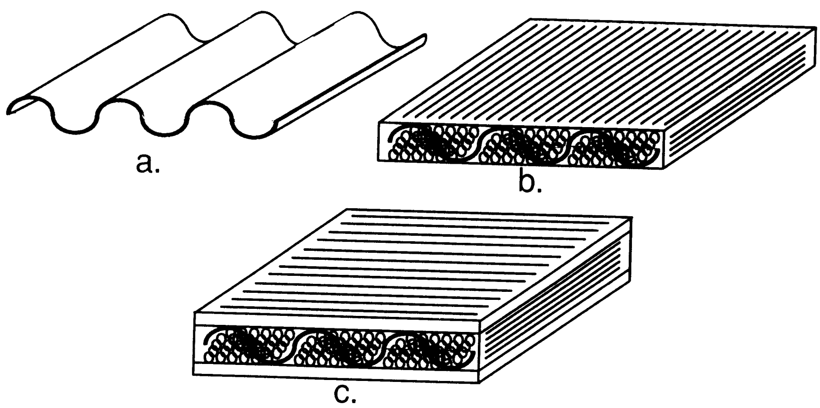

Fig. 2 Various stages in the fabrication of the wavy laminate.

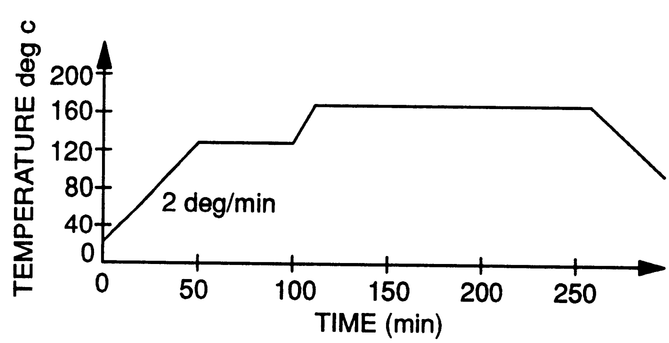

Fig. 3 Cure cycle for the graphite/epoxy composite.

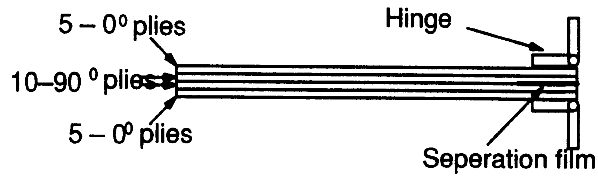

Fig. 4 A double cantilever sample with separation film to initiate crack.

The fracture toughness of the samples was obtained by the area method. Here the critical energy release rate is determined from a loading–unloading sequence. For further details of testing and data reduction, the interested reader is referred to Carlsson and Pipes [4]. An average value of G_{IC} was obtained for each sample. The results based on five tests of each type of sample, for the DCB mode I fracture tests, are shown in Table I. It is observed that when the intermediate cure was 65^0 C, the composite laminas could be shaped, but the polymerization process, if any, did not effect the eventual fracture toughness of the composite. The slight increase in G_{Ic} can be attributed to the test variability. On the other hand as the intermediate cure temperature was increased, the effect on the fracture toughness became evident. At these temperatures the epoxy was semi–polymerized and during subsequent cure cycle the bonding was not as good. Based on these results the first step cure was done at 65^0 C for all the samples.

The wavy composite samples were now subjected to compression testing and non–destructive testing. The testing is a combination of the two and so the testing sequence is as follows. To obtain a benchmark state, these specimen are subjected to ultrasonic NDE. The purpose of these tests was to see if the wavyness in composites can be observed by the ultrasonic methods. A theoretical analysis was performed and the reflection coefficient was calculated. The results for the theoretical analysis are shown in Fig. 5. It is observed that the reflection coefficient is large at the nodal points and the flat regions in the curve are regions where the transmitted waves cease to exist due to the large angle between the two interfaces. For ultrasonic testing we have used the immersion mode where the specimen is fully immersed in water. We have used a 5 MHz, 0.5 "(12.7 mm) diameter, 2"(50.8 mm) focal length transducer. The transducer is set in a pulse–echo mode and the signal reflected from the back surface of the specimen is collected and its magnitude is plotted. This is the so called, C–Scan mode of interrogation. The step size in the scan is 0.025"(0.635 mm). A typical scan of the undamaged specimen is shown in Fig. 6.

It is observed that well defined bright and dark lines are obtained. The spacing of these lines is equal to half the wavelength of the wave in the composite.

Table I Critical Energy release rate for various graphite/epoxy samples.

SPECIMEN	G_{IC}
Single CureCycle	751.3
Double Cure (65^0 C)	767.0
Double Cure (80^0 C)	688.2
Double Cure (93^0 C)	548.1

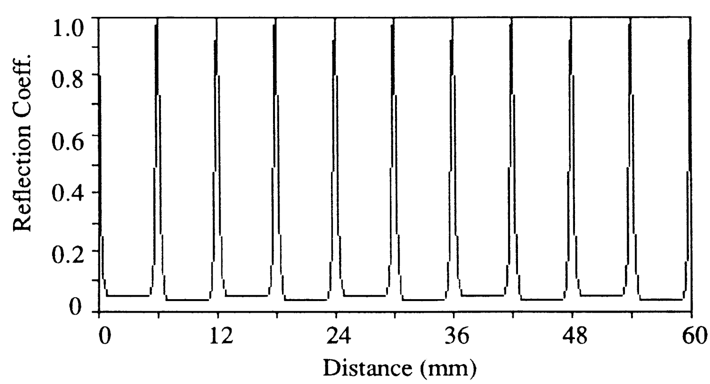

Fig. 5 The theoretical reflection coefficient of a wavy sample.

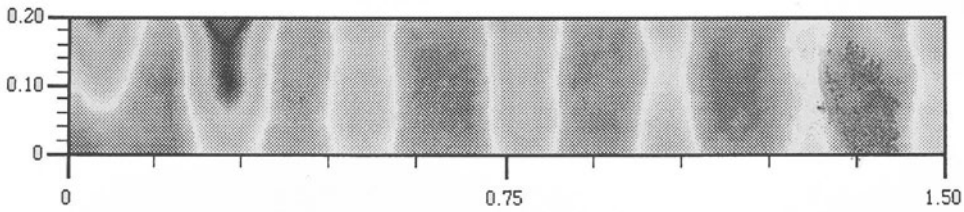

Fig. 6 C–Scan of an undamaged Graphite/Epoxy wavy sample.

Next, the wavy samples are subjected to the compression testing, Fig. 7, in a Servo–hydraulic MTS machine and the loads were applied such that the specimen does not show internal damage. Results shows that the laminate initially behaves as a non–linear elastic material with increasing stiffness. When the load becomes sufficiently high where the fibers buckle, the curve becomes highly non–linear. During the unloading cycle, the curve was non–linear and then follows the loading curve with very little difference in the slope of the load displacement curve.

Specimen were then loaded to a point where they underwent substantial damage. Now, we have observed these specimen under a microscope and a cross–section of the composite before and after the damage are as shown in Fig. 8. Finally, the Ultrasonic C–scan of the damaged specimen is shown in Fig. 9. It is observed that during loading the damage has taken place mainly in the form of delamination between the wavy and flat laminas. As a result of this delamination the scan does not show the regular light and dark lines. The reason for this is that the newly created surfaces are now acting as the wave scatterers and very little wave reaches the transducer.

CONCLUSIONS

The Graphite/Epoxy prepregs can be used for the manufacture of complicated wave like plies in composites. The two step process does not alter the fracture toughness of the composite

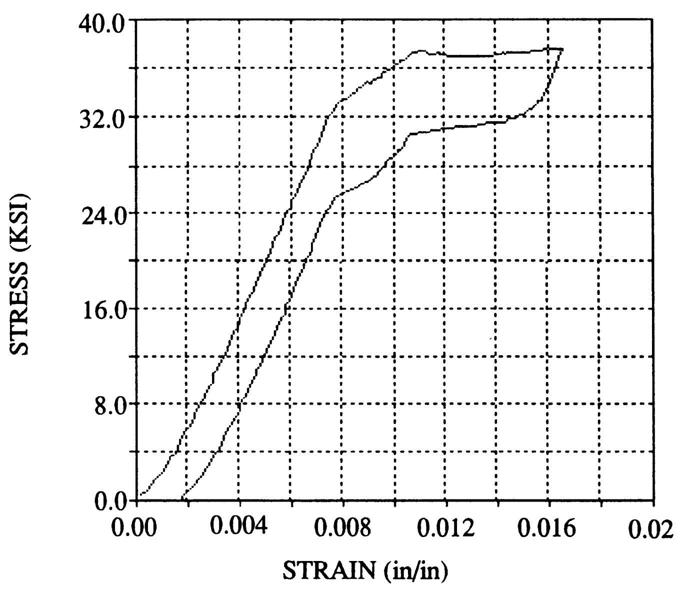

Fig. 7 Stress–Strain curve for the wavy composite under compression. Note loads are not enough for any internal damage.

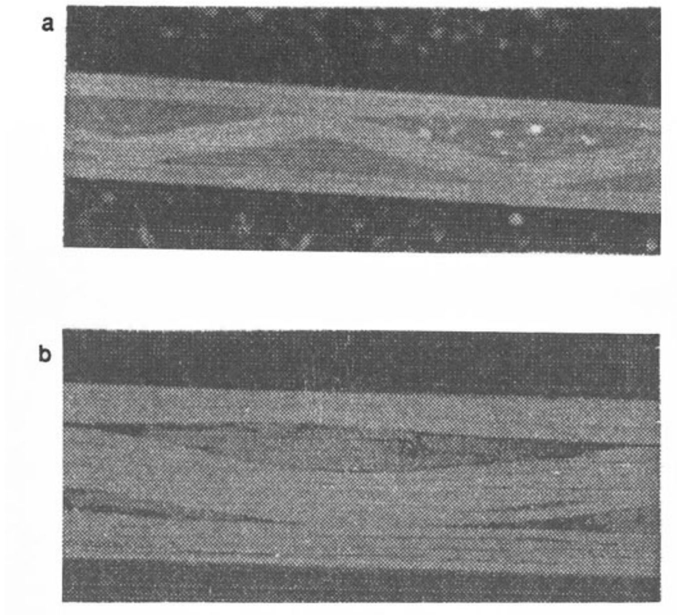

Fig. 9 Micrograph of wavy specimen (a) before and (b) after damage.

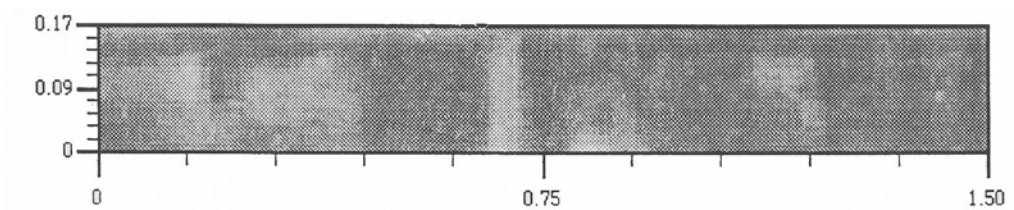

Fig. 10 C–Scan of the wavy sample after damage.

and good samples can be made. The wavy pattern in the composite can be theoretically predicted and are easily detected by ultrasonic c–scan which has alternate dark and light lines. The compression testing shows the material behaves in a non–linear elastic manner. Under excessive loading the damage occurs as debond at the interface of the wavy laminas.

REFERENCES

1. Garala, H.J., "Experimental evaluation of Graphite–Epoxy composite cylinders subjected to hydrostatic compressive loading," Proceedings of the 1987 SEM Spring Conference on Experimental Mechanics, SEM, Bethel, CT, p 948 (1987).
2. Hayer, M.W., Maahs,L.C., and Fuchs, H.P., "The influence of layer waviness on the stress state in hydrostatically loaded cylinders," J. of Reinforced Plastics and Composites, 7(11), p 601 (1988).
3. Telegadas, H.K., Hyers, M.W., "The influence of Layer Waviness on the stress state in hy drostatically loaded cylinders: Further results," J. of Reinforced Plastics and Composites, 9(5), p 503 (1990).
4. Leif A. Carlsson, and R. Byron Pipes, *Experimental Characterization of Advanced Com posite Materials*, (Prentice Hall, Inc, N.J., 1987).