The stacked lamellar texture on the fracture surfaces of fibre composites

RICHARD E. ROBERTSON, VIORICA E. MINDROIU Research Staff, Ford Motor Company, Dearborn, Michigan 48121, USA

A fracture surface texture, which has been variously termed as "lacerations", "hackles" or "serrations", is often observed on the matrix surface of fibre composites, most often in resin-rich regions. This texture, referred to here as a "stacked lamellar texture" to emphasize its plate-like nature, was studied in an E-glass/epoxy composite. Scanning electron fractographs of these materials suggest that the stacked lamellar texture arises from crack fingers due to a meniscus instability mechanism interacting with a reorienting stress field.

1. Introduction

A texture that is often seen on the fracture surfaces of the polymeric matrix of fibre composites consists of stacks of overlapping scale-like lamellae of the matrix material. An example of this texture is visible in Fig. 1. The dark horizontal bands in Fig. 1 resulted from the pullout of fibres and have roughly the width of the fibres. The lamellae tend to be oriented perpendicularly to the fibre axes, although they are usually bent over in a direction along them, and have a width roughly equal to the distance between adjacent fibres. This lamellar texture was specifically noted by Sinclair and Chamis [1] who referred to it as "lacerations", by Morris [2] who referred to it as "hackles", and by Awerbuch and Hahn [3] and Johannesson et al. [4] who referred to it as "serrations". Since none of these three terms convey the sense of a plate-like structure, we prefer "stacked lamellar texture". (It should be noted that the lamellar texture is unrelated to the "lamellae" that occur in crystalline polymers; the matrix resins in which the stacked lamellar texture has been seen are crosslinked and amorphous and are incapable of crystallizing.)

Various features of the stacked lamellar texture have been studied and several mechanisms have been proposed for their formation [1-4]. The texture is usually found in resin-rich regions where the separation between the fibres is a fibre diameter or more. Also, the texture has most often been seen when the composite was subjected to shear [1, 3, 4]. Awerbuch and Hahn [3], who studied uniaxial graphite/epoxy specimens that were pulled off-axis in tension, noted that the texture was most prominent as the angle between the tensile and fibre axes approached zero. However, Morris [2] reported the occurrence of the stacked lamellar texture in specimens pulled at 90° to the fibre axis.

Morris studied also the tendency for the lamellae to be bent over in a direction usually parallel with the fibre axis. He noted that the direction of slant or tilt of the lamellae is opposite on the two surfaces, meaning that lamellae would be parallel if the opposite surfaces were brought together again. Morris also found that the direction of tilt on a given surface was characteristic of the direction of crack growth; i.e. the direction of tilt was the same over the whole surface except where the direction of fracture happened to change.

For the formation of the stacked lamellar texture, Morris noted the similarity between this texture and the shear cracks often visible in resinrich areas, which from the side look like elongated Ss and are oriented normal to the direction of maximum resolved tensile stress. But Morris suggests that the stacked lamellar texture is induced by flexure after the primary cleavage has separated the material in two.

Johannesson et al. [4] studied angle-ply

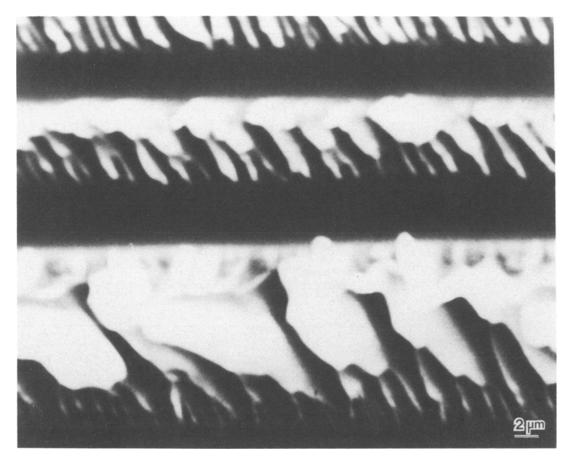


Figure 1 Stacked lamellar texture on the fracture surface of a cleaved E-glass/epoxy specimen.

specimens of graphite/epoxy failed in tension and noted some finer subdivision of the matrix near the fibre imprint as well as an occasional interleaving of the lamellae across fibre imprints. They conclude that the fibres and matrix debond before the lamellae are formed.

Using fibre composite specimens with intentionally large resin-rich regions, we have obtained fractographs that are able to shed further light on the lamellar texture and its formation.

2. Experimental procedure

2.1. Material

The material studied was obtained from a stoichiometric mixture of a trifunctional aromatic epoxy resin containing a tertiary amine (Ciba-Geigy 0500) and 4-methylhexahydrophthalic anhydride. The liquid epoxy resin-hardener mixture was applied to E-glass rovings wrapped around moulded polypropylene plates backed by glass plates. The polypropylene was used to allow easy release of the sheet after moulding. The E-glass roving was wrapped with roughly 5 mm separation between wraps to obtain fibre-free resin areas. Before curing, the sheets of fibres and resin were de-aerated in a vacuum chamber for about half an hour, during which much bubbling took place in the resin. A second polypropylene plate was then placed on top of the fibres and resin, and the resin was cured in an air-circulating oven for 30 min at 120° C followed by 30 min at 150° C. The sufficiency of this cure schedule was indicated by the absence of anhydride following cure, as measured by infra-red spectroscopy.

2.2. Specimen preparation

The fracture surfaces were obtained by cleavage. Specimens of the desired size and shape were cut from the prepared sheets with a diamond saw. In general, the specimens consisted of a clear band of matrix straddled by parallel bands of fibrereinforced matrix. The location of the fracture surface was started with another saw cut made in the clear matrix away from the fibres. The

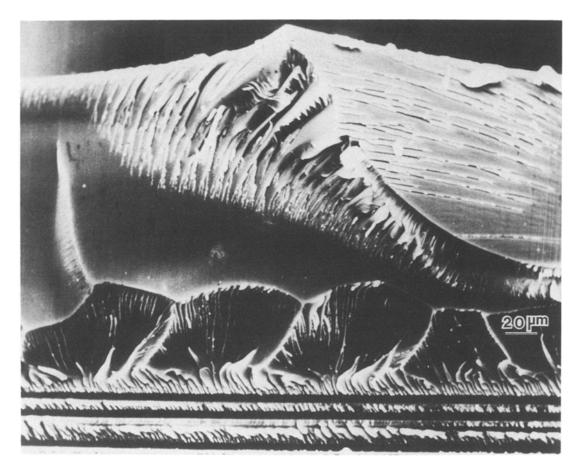


Figure 2 Overall view of a fracture surface of a cleaved E-glass/epoxy specimen showing the stacked lamellar texture near the bottom of the figure and again near the top.

cut in each specimen was sharpened by pressing a razor blade into it, and then the specimen was cleaved manually. Cleavage usually occurred in these materials with some difficulty; i.e. the materials seemed very tough. Though the fracture began in the resin, it usually veered off toward the fibres where it was restrained to follow them. After cleavage the specimens were mounted and sputtered with a thin layer of gold for use in the SEM.

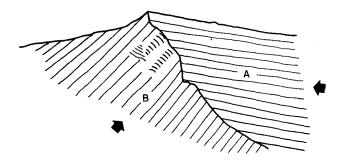
2.3. Examination procedure

The specimens were examined in a JSM-2 scanning electron microscope (SEM). As the best compromise between obtaining optimum resolution and the tendency for the specimens to charge, an accelerating voltage of 15 kV was used for all micrographs. To enhance the contrast, which was particularly important for the relatively shallow topography of the basic longitudinal texture to be discussed below, the specimen

normal had to be tilted away from the incident electron beam toward the detector by 40 to 45° .

3. Results

Fig. 2 shows an overall view of the fracture surface of a cleaved E-glass/epoxy specimen. The stacked lamellar texture associated with fibre pullout is visible at the bottom of this micrograph. The stacked lamellar texture of greater interest, however, is near the top. A schematic diagram of the upper region of Fig. 2 is shown in Fig. 3. The stacked lamellar texture is seen to be near the intersection of two major cracks that were moving in different directions. The nominal fracture direction in this specimen was the same as that on surface A in Fig. 3. The crack that formed surface B represents a secondary fracture that probably was initiated by the pullout of fibres at the bottom of Fig. 2. Yet the two cracks probably arrived at the intersecting ridge at roughly the same time. An examination of the



ends of some of the welts on surface A, the welts being the fibre-like entities that constitute most of the obvious texture on both fracture planes A and B [5], indicates that the crack on surface B may have reached the intersecting ridge in places ahead of that on surface A. These welt ends were not distorted nor were the welts detached from the steps to which they were associated [5], both of which would have occurred if crack A had preceded crack B in these areas.

The stacked lamellar texture in the upper part of Fig. 2 and the ends of the welts on surface Figure 3 Schematic diagram of the upper part of Fig. 2 showing the stacked lamellar texture and the directions of the cracks that formed the two major surfaces, A and B.

A are better seen in Fig. 4. Also visible in Fig. 4 is the basic longitudinal texture associated with the crack direction on surface B, diagonally from lower left to upper right [5]. The two visible regions of the basic longitudinal texture are just below the two regions of stacked lamellar texture. It is the basic longitudinal texture that we suggest has been the origin of the stacked lamellar texture.

4. Discussion

Because of the clear view of the fracture surface immediately adjacent to the stacked lammellar



Figure 4 The stacked lamellar texture in the upper part of Fig. 2 and the basic longitudinal texture associated with the fracture of surface B. texture, the formation of this texture in Fig. 4 is easier to discern than that associated with the fibres, although the two probably arise in much the same way. The basic longitudinal texture that is suggested to be the precursor of the stacked lamellar texture in Fig. 4 has been suggested to arise from a meniscus instability at the crack tip [5]. A meniscus instability was first proposed for fluids by Taylor [6] and its possible occurrence and nature in materials like matrix resins was discussed by Fields and Ashby [7] and by Argon and Salama [8]. A meniscus instability can result in any waviness of the crack front developing into a series of fingers protruding into the bulk ahead of the nominal crack front. Although the fingers often remain together in the same plane (except for the slight deviation that allows their progress through the resin to be visible as the basic longitudinal texture), they can lose coherence with one another in the presence of material or stress heterogeneities or gradients [5]. This can cause the fingers to move on to different planes.

In relatively homogeneous materials the paths of the crack fingers are governed largely by how the direction of the maximum resolved tensile stress changes from point to point. If the direction of the maximum resolved tensile stress rotates about an axis in the plane of the crack that is *perpendicular* to the instantaneous crack direction while the crack is moving, then the directions of all the crack fingers will change together. If, on the other hand, the tensile stress direction rotates about an axis parallel with the instantaneous crack direction, then the fingers continue to move in their former direction but the planes of the fingers rotate, putting the fingers on to different but parallel planes. This is indicated in Fig. 5 where a pair of fingers is viewed from a point ahead of the crack. Considering that the periodicity of the fingers tends to remain largely constant for a given material and temperature (the periodicity of the fingers was approximately 350 nm in the epoxy studied, which was cleaved at room temperature), the separation between the planes of the crack fingers can be imagined from Fig. 5 to increase as the rotation of the crack planes increases up to 90°. (It is uncertain what happens if the stress rotates back to its original orientation. Because the fingers arise from an instability, the tips of the fingers are likely to move somewhat randomly, moving laterally as well as forward. Hence, the coherence between

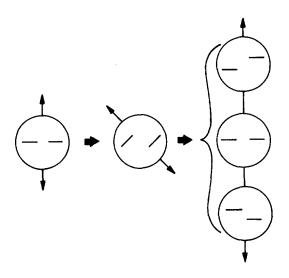


Figure 5 Schematic diagram showing the effect on the crack-finger orientation when the maximum resolved tensile stress rotates about an axis parallel with the instantaneous crack direction while the crack is in motion.

the fingers is likely to be lost, leaving the fingers on different planes even after the maximum resolved tensile stress has rotated back to its original orientation.)

For a general change in the direction of the maximum resolved tensile stress, rotating about an axis that is neither perpendicular nor parallel with the instantaneous crack direction, the moving crack fingers will both change direction and separate on to different planes. The fracture surfaces therefore can become very complex with the crack bifurcating (actually "multifurcating") while changing direction.

The stacked lamellar texture in Fig. 4 is suggested to result from the maximum resolved tensile stress suddenly rotating from being roughly perpendicular to the plane containing the basic longitudinal texture to being roughly perpendicular to the lamellae as the crack front moves into that region. The path of the crack fingers is that suggested in Fig. 6. The fingers, starting together on the same plane, turn and diverge on to different planes as the maximum resolved tensile stress rotates. The cause of the change of direction of the maximum resolved tensile stress was probably the confluence of cracks A and B (see Fig. 3). Although there is evidence that crack B reached the intersecting ridge before crack A in some places, namely where the basic longitudinal texture on B extends up to the ridge, it is likely that crack A reached the intersecting ridge ahead of crack B in other places,

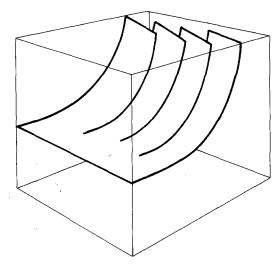


Figure 6 Schematic diagram showing the formation of the stacked lamellae by the fingers of the crack front following a reorientation of the tensile stress.

especially where the basic longitudinal texture on B was sweeping around toward a direction parallel with the intersecting ridge, just adjacent to the stacked lamellar texture (see Fig. 4). The arrival of crack A at the ridge would not only change the magnitude of the stress on crack B but could change the direction, especially if, because of the fracture, the loading on this part of the specimen were changing.

The origin of the stacked lamellar texture between the fibre imprints, as in Fig. 1, is suggested to be analogous. A primary fracture, occurring with the stress in an initial orientation and developing protruding fingers characteristic of the meniscus instability, is suggested to spread just as the stress is reorienting. The details of how the stacked lamellar texture forms may not be the same in all instances. As noted, the texture has occurred under both shear and cleavage nominal stress states. But a typical occurrence may begin, as suggested by Johannesson *et al.* [4], with the fibres pulling away from the matrix. Then, with the support of the fibres lost, the stress may reorient, at least locally, and produce the lamellae in the intervening matrix.

Although the stacked lamellar texture has almost always been seen in fibre composites, it is expected to occur more generally. According to the proposed mechanism the texture should arise whenever a growing crack front consists of finger-like protuberances and the stress field acting on the crack changes direction during growth. The frequent occurrence of the texture on the fracture surfaces of fibre composites is thought to be due, then, to the complex and changeable stress field associated with the fibres.

Acknowledgement

We wish to acknowledge the helpful discussions during the course of this work with our colleague, Dr P. Beardmore.

References

- J. H. SINCLAIR and C. C. CHAMIS, NASA-TP-1081 (1977); Sci. Tech. Aerosp. Rep. 16 Abstract No. N78-13138 (1978).
- G. E. MORRIS, "Nondestructive Evaluation and Flaw Criticality for Composite Materials", ASTM STP 696, edited by R. B. Pipes (American Society for Testing and Materials, Philadelphia, 1979) p. 274.
- 3. J. AWERBUCH and H. T. HAHN, in "Fatigue of Fibrous Composite Materials", ASTM STP 723 (American Society for Testing and Materials, Philadelphia, 1981) p. 243.
- 4. T. JOHANNESSON, P. SJOBLOM and R. SELDEN, J. Mater. Sci. 19 (1984) 1171.
- 5. R. E. ROBERTSON, V. E. MINDROIU and M. F. CHEUNG, Composite Sci. Tech. 22 (1985) 197.
- 6. G. I. TAYLOR, Proc. Roy. Soc. A201 (1950) 192.
- 7. R. J. FIELDS and M. F. ASHBY, *Phil. Mag.* 33 (1976) 33.
- A. S. ARGON and M. SALAMA, *Mater. Sci. Eng.* 23 (1976) 219.

Received 16 August and accepted 13 September 1984