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EFFECT OF THERMAL SHOCK ON FLEXURAL MODULUS OF THERMALLY AND CRYOGENICALLY CONDITIONED KEVLAR/EPOXY COMPOSITES

B. C. RAY

Department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela- 769008, INDIA

bcray@nitrkl.ac.in

ABSTRACT

The utilisation of polymeric matrix composite materials in safety critical structures necessitates their full characterisation especially where changing temperature environment is a concern. An attempt has been made here to assess the effects of thermal shocks on flexural modulus of thermally and cryogenically conditioned Kevlar/epoxy composites. Thermal and cryogenic conditionings were concurrently followed by downthermal shock (positive to negative temperature excursion) and upthermal shock (negative to positive temperature excursion) treatments respectively on the composite laminates. Residual thermal stresses developed by temperature gradient should be given critical attention in many application areas. The 3-point short beam shear (SBS) test was conducted on the conditioned specimens to evaluate modulus. The test results may indicate the relative level of bond strength in a composite system where only the bonding level is a variable. Post-curing strengthening effect of thermal conditioning and mechanical keying factor of cryogenic conditioning are investigated here by scanning electron micrographs. The high radial expansion coefficient of Kevlar fibre causes weakening of interfacial adhesion under the influence of temperature gradient. However, the weak interface may readily allow crack deflection along the interface and improves energy-absorbing capacity.

1. INTRODUCTION

The mechanical properties of composite materials are strongly dependent on the character and architecture of fibre, the extent of resin cure and the nature of the fibre/polymer interface. The fibre/matrix interfacial behaviour can be explained by the mechanical principle with the assumptions made at the level of fibre/matrix adhesion and/or by using the surface chemistry approach [1]. The interfacial bond strength is weaker in Kevlar/epoxy composites compared to carbon/epoxy composites [2]. Kevlar fibres have high specific strength, great cohesiveness and a tendency to form fibrils. They absorb higher energy than brittle fibres and are widely used in aircraft, aerospace and ballistic applications. Unfortunately, the high radial expansion coefficient of Kevlar fibre causes an unfavourable tensile stress at the interface [3]. The weak interfacial adhesion between Kevlar fibre surface and polymer matrix is of major influence on the response of the composite to stress after environmental exposure. The interfacial bonding is further influenced by exposure to complex and active environments [4-6]. The weak interfacial adhesion of Kevlar/polymer composites makes them more susceptible to environmental degradation. But a weak interface may promote extensive debonding. That can lead to a significant increase in impact energy. The fibre/matrix deadhesion mechanism is dependent on the interfacial bond strength between fibre and matrix. Composites with low interfacial bond strength may exhibit large areas of interfacial debonding. It may intensify delamination to speed-up laminate failure. The present investigation aims to study the effect of thermal shock on flexural modulus of thermally or cryogenically conditioned Kevlar 49/epoxy laminates. Kevlar 49 has been specifically engineered for polymer reinforcement and is intended more for the aerospace industries. These aerospace materials are subjected to severe thermal stresses that may be produced by aerodynamic heating or by localized intense fire. The present literature is unfortunately not so extensive and so inconclusive in this area. Thermal shock may induce intense thermal stresses in the components. The concentration of thermal stresses around defects may result in premature failure. The damage analysis for aerospace structure which are exposed to intense thermal shock requires technique for determining thermal stress intensity factor and critical studies of fracture behaviour [7]. It may reasonably be assumed that the interfacial chemistry is the net result of a number of contributions to the fibre/matrix adhesion. These contributions possibly include chemical bonding, secondary forces of attractions, residual thermal compression forces due to differential shrinkage and also mechanical interlocking between the fibre and the polymer matrix [8]. The test used here was a 3-point flexural test on a specimen with a very small span length, which generally initiate failure by interlaminar shear. This assumption is valid where only the bonding level is a variable. The fibre/matrix interfacial debonding may be reflected in the 3-point bend test. The main objective of this study is to evaluate the prior effects of thermal or cryogenic conditioning in counteracting the detrimental effect of thermal shock in Kevlar/epoxy system. Cyclic exposure is an important factor when considering the service condition of a plastic material. The temperature in Reno, NV, has a daily mean variation of 20°C temperature and can range annually from –30°C to 40°C temperature. Flights are also frequently experiencing a wide fluctuation of temperatures from ground level to high altitude temperature during their service cycles (take-off, airborne and landing steps). The high gradient of thermal shock is selected here to get the accelerated aging behaviour. The phenomenal effect of thermal shock on mechanical performance in a very short time period may be a matter of concern here in the present study. The accelerated testing results are most often extrapolated back to the normal service temperature to predict the long-term performance of materials.

2. EXPERIMENTAL DETAILS

Kevlar aramid 49 fibre of woven cloth and epoxy (Gougeon West System, 105 resin and 205 hardener) were used in the present experiments. The short beam shear (SBS) specimens were treated in a thermal shock environment with a 160°C temperature gradient. One batch of samples was thermally conditioned at a 80°C temperature for 5, 10, and 20 min times before they were immediately exposed to –80°C temperature for 5 min. The –80°C temperature was maintained in a saturated mixture of solid carbon dioxide in acetone. The other batch of samples were first cryogenically conditioned at –80°C temperature for the same time periods and then they were exposed to 80°C temperature for 5 min for the each stage of cryogenic conditioning. The three-point SBS tests were conducted almost immediately after the thermal shock treatment to avoid and/or minimize the possibility of relaxation phenomena in the composites at room temperature. The test was carried out as per ASTM (D 2344-84) standard.

The modulus values were calculated as follows:

$$\text{modulus} = pL^3/4bdt^3$$

where p is maximum load, b width of specimen, d deflection, t thickness of specimen and L is span length of specimen.

3. RESULTS AND DISCUSSION

The effect of thermal conditioning time on modulus of the downthermally shocked composites is shown in Fig. 1. Here, the specimens were first conditioned at a 80°C temperature for different times. Thereafter, thermal shock was followed from the different conditioning times. There is a large drop in modulus values for the 5 min conditioning time. Thereafter, the trend of an improvement is observed in the figure. The variation of modulus here is the net result of the post-curing strengthening phenomena and the debonding effect of thermal shock. The thermal conditioning is likely to change the chemistry at the fibre/matrix interface either by forming an interpenetrating network [2] and/or by further cross-linking in the epoxy matrix. These phenomena may impart better adhesion at the interface. The improved interfacial adhesion is likely to be counteracted by the adverse debonding effect of the following thermal shock. This weakening of the interface during thermal shock treatment is due the different coefficients of thermal expansion between epoxy matrix and Kevlar

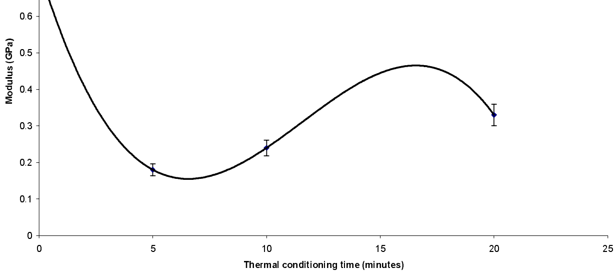

Fig.1: Effect of thermal shock on modulus of thermally conditioned Kevlar/epoxy composites.

Fig.2: SEM micrograph showing fibrilar morphology on the delaminated surfaces.

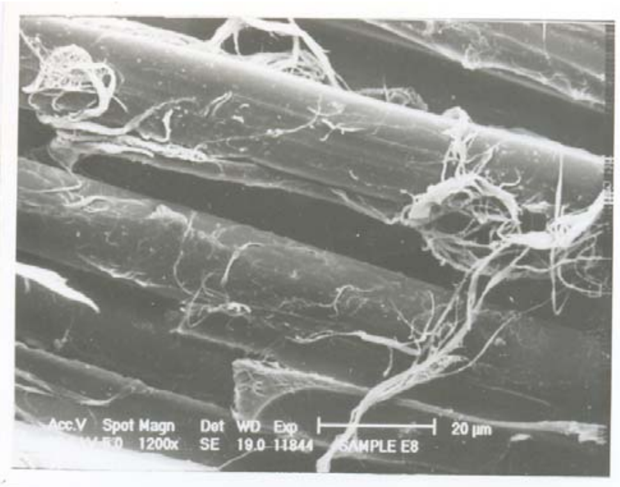

Fig.3: SEM micrograph revealing fibrils and micro-fibrils of Kevlar fibres.

reinforcement. The detrimental effect of thermal shock predominates at 5 min thermal conditioning because of the short post-curing time. That results in only small degree of further polymerization in the epoxy matrix. The weak interface readily allows crack deflection. The composite can sustain a large deflection here by permitting the absorption of more energy. Thermal conditioning for longer time duration contributes better fibre/matrix adhesion. The modulus values are improving with the conditioning time. A reasonable explanation is that crazing may possibly occur in a lightly cross-linked thermoset but not in well cured resins such as epoxy [2]. The highly cross-linked network does not permit gross plastic deformation, thereby limiting the energy-absorbing capability to a large extent. The scanning photomicrograph (Fig. 2) shows the extensive fibrillation of Kevlar fibres in the unconditioned Kevlar/epoxy laminates. Again the tendency to form fibrils is observed in the photomicrograph (Fig. 3) for the specimens those were conditioned for 20 minutes at 80° C temperature and then concurrently followed by downthermal shock treatment.

The variation of modulus of upthermally shocked Kevlar/epoxy composite is plotted against the cryogenic conditioning time in Fig. 4. Here, the SBS specimens were first cryogenically conditioned at a –80°C temperature for different durations Then the thermal shock was given to the specimen by immediately exposing them to 80°C temperature form each stage of cryogenic treatment. There is a noticeable improvement in modulus for the 5 minutes conditioning time. The synergistic effect in modulus

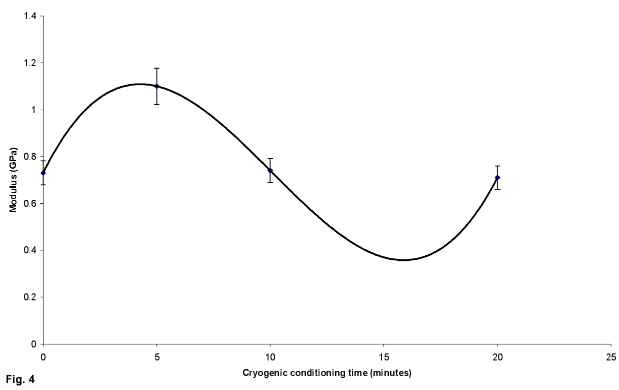

Fig.4: Effect of thermal shock on modulus of cryogenically conditioned Kevlar/epoxy composites.

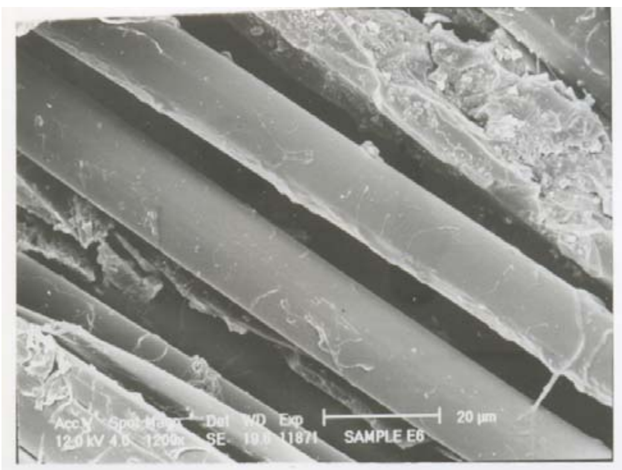

Fig.5: SEM photomicrograph showing multiple matrix cracking and intra-ply delamination on the delaminated surface of the conditioned specimen.

is probably due to the limited energy-absorbing capability. The point could be related to the strain-free state of the laminates. The residual compressive stress due to cryogenic conditioning is being nullified by the tensile stress during this kind of negative to positive temperature change thermal shock. The nature of the interfacial adhesion is strongly affected by the presence of residual stresses. These stresses are sometimes also relaxed by viscoelastic flow or creep in the polymer matrix [9]. The longer period of cryogenic conditioning attributes a greater amount of shrinkage compressive stress in the laminate. The specimen, although supporting lesser load, can sustain a larger deflection allowing the absorption of higher energy due to the presence of greater amount of cryogenic compressive stress. This stress causes a better adhesion by the mechanical interlocking principle at the interface. The stronger interface may not permit a large deflection during fracture. The multiple matrix crackings are evident in the scanning photomicrograph (Fig. 5) in the thermally shocked Kevlar/epoxy composites for the 15 min cryogenic conditioning. The matrix cracks can readily absorb energy during crack propagation. The same specimen also demonstrates inter-ply and intra-ply delamination (Fig. 6) for the same conditioned one.

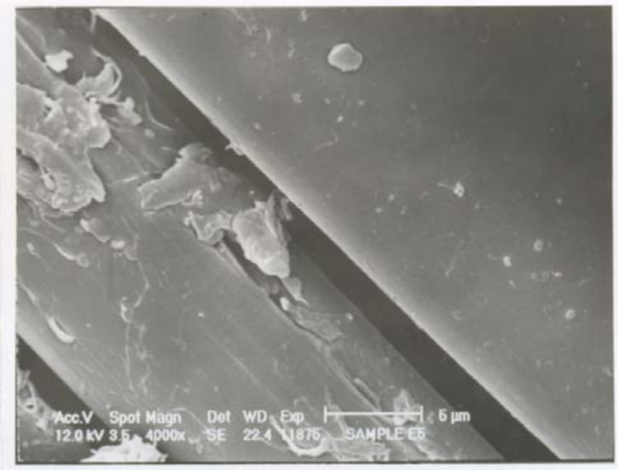

Fig.6: SEM photomicrograph showing inter-ply delamination and blocky outcrop of epoxy matrix.

A weaker interfacial bond may in general lead to intra-ply splitting and inter-ply delamination, when the splitting and/or delamination are the dominating failure modes [2]. However, when tensile or compression failure modes become prevalent, then a weaker interfacial bond may result in a poor flexural strength of the composite. The most important parameter that greatly influences the mechanical performance of composite materials is the fibre-matrix interfacial bond. The nature of this interface bond is not only significant for the strength and stiffness of the composite, but it also controls mechanisms of damage character and propagation [10, 11]. An interfacial reaction causes various morphological changes to matrix microstructure in proximity to the fibre surface. The interactions at interface are very important and complicated phenomena [12]. The mismatch of thermal expansion coefficient between fibre and polymer matrix, and the difference in Poisson's ratio may result in a triaxial stress state in the matrix. The resulting hydrostatic tensile stress severely reduces strain to failure of epoxy rein [13]. The chemical and structural changes in the epoxy matrix by environmental ageing influence the performance of a fibre-reinforced composite [14]. One possible explanation of interfacial failure due to environmental exposure is the existence of a chemical gradient in the structure of epoxy matrix from the fibre surface to the polymer bulk [15].

4. CONCLUSIONS

The incomplete understanding and a lack of effective control of the interface limits the accurate assessment of resulting properties. The effect of thermal shock exhibits a variation in failure modes between thermally and cryogenically aged samples. The thermal conditioning prior to the thermal shock treatment induces improved interfacial adhesion probably by the surface chemistry phenomena. The improvement of fibre/matrix interface strength to a small order for longer aging time is observed in the cryogenically conditioned specimens. This small increment here is possibly contributed by mechanical keying factor only. The debonding effect of thermal shock with a prior history of thermal and cryogenic conditionings of the Kevlar reinforcement thermosetting epoxy matrix is qualitatively assessed in the present study.

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