Effect of nano and micro SiO₂ weight percent on interlaminar fracture

toughness of woven roving/ epoxy composites

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Abstract	Key words
Effect of nano and micro SiO_2 particles with different weight percent (2,4,6,8 and 10) % wt on the Interlaminar fracture toughness (G _{Ic}) of 16-plies of woven roving glass fiber /epoxy composites prepared by hand lay – up technique were investigated. The specimens were tested using DCB test (mode I). Area method was used to compute the interlaminar fracture toughness The results show that G _I , would increase with the	nano and micro SiO interlaminar fractur toughness, woven roving composites
$\frac{1}{2}$	

increasing in the filler content, the main failure in microcomposites and nanocomposites was delamination in the layers, the delamination reduced with increasing in the filler content.

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دراسة تأثير النسب الوزنية لجزيئات السليكا المايكروية والنانوية على متانة الكسر الطبقية لمتراكبات الايبوكسى المدعمة بالالياف نوع المنتظم حارث ابراهيم جعفر، زينب رحيم مسلم قسم الفيزياء ، كلية العلوم ، جامعة بغداد

الخلاصة

تم في هذا البحث در اسة تاثير اضافة نسب وزنية مختلفة من جزيئات السليكا المايكروية والنانوية على متانة الكسر الطبقية لمتراكبات الايبوكسى المدعمة بالالياف نوع المنتظم المحضرة بتقنية التشكيل اليدوي وبنسب 2 و 4 و 6 و 8 و 10 %. فحصت العينات باستخدام نموذج العتلة المزدوج (النمط الاول) واستخدمت طريقة المساحة لحساب متانة الكسر الطبقية. اظهرت النتائج أن متانة الكسر الطبقية تزداد بزيادة نسبة الجزيئات المضافة وإن السبب الرئيسي للفشل في المتراكبات المايكر وية والنانوية هو التوسعات الطبقية و التي لو حظ انها تقل بز يادة نسب الجز يئات المضافة.

Introduction

Modern composite materials use highstrength fibers in a resin matrix. The fibers and matrix are combined to form a single ply.

Composite laminates are formed by stacking plies of different orientations, but one of the most common failure modes of composite structures is delamination between plies. Delamination is a crack that forms between adjacent plies [1].

Woven composites are advanced materials that are commonly used in aerospace applications. Their use is interesting owing to their excellent durability over complex geometries, their effective manufacturing cost and their good damage tolerance properties [2].

Many literatures are devoted with the study the mechanical properties of woven composites, the aim of the work was to study the effect of micro and nano SiO₂ particles on the interlaminar fracture toughness of woven roving composites, which defined as the energy absorbed by the laminate divided by the newly formed delamination crack area, the delamination occurs from the combination of an existing crack tip that provides a stress concentration, interlaminar and Mode stresses. Ι interlaminar fracture toughness. There is a need to improve the interlaminar fracture toughness of laminate composites, this was done by inclusion micro particles to improve G_{Ic.} and Nanoparticle-modified polymer composites (also termed polymeric nanocomposites) have attracted great scientific and technological interest owing to their physico-mechanical, thermal and other properties [3].

Sun [4] shows that the addition of nano filler into Epoxy adhesive improve the debonding and shear strength .The presence of nano particles is in the improvement of delamination resistance of the joint.

Han and Cho [5], shows that the fracture toughness of nano silica filled Epoxy molding compound was found to improve in temperature range (from ambient to 230 °C), fractography suggest that the nano particles act as surface modifier of micro silica particles.

Zhang [6] found that the fracture toughness of SiO_2 /EP nanocomposites enhanced with increasing silica content up to 14% vol.

Tadaharu Adachi [7] study the effect of particle size and volume fraction on mode I fracture toughness of Epoxy composites filled with SiO_2 micro and nano particles, which is increased as vol. fraction increased and particle diameter decreased.

Tsai & Cheng [8] investigate the fracture toughness of glass fiber /Ep composites using different content of SiO_2 nanoparticle 10% wt, 20% wt, which is increased as compared with those without any silica particles.

Tsai & Cheng [9] investigate the interlaminar fracture toughness of glass fiber/Epoxy composites which consist of SiO₂ nanoparticles and rubber particles by using two types of rubber (CTBN) & (CSR), the inclusion of SiO₂ nanoparticle with CSR increase the fracture toughness up to 82%, when the epoxy matrices were modified with CTBN rubber particles, the fracture toughness was around 48%.

Kinloch et al [10] describe the fracture of nanosilica and rubber toughened Epoxy fiber composites, the results demonstrate that there is a transfer of toughness from the increases that have been observed in the bulk, rate effect with the addition of nanosilica have been explored, with increase in composite stiffness leading to delamination and energy absorption upon impact.

Hsieh [11] shows that the largest increase in toughness observed for hybrid material containg 9% wt& 15% wt of the rubber micro particles and silica nano particles.

Theoretical Part

1. Area method

Area method was one of the methods used to calculate the interlaminar fracture toughness (G_{Ic}) , from the load–displacement curve and crack length measurements, the interlaminar fracture toughness (G_{IC}) used in this study is based on the area method [12] given in Equation (1):

$$GIC = \frac{\Delta Aij}{w(aj - ai)} \tag{1}$$

where ΔA_{ij} is the area under loaddisplacement curve between crack lengths a_j and a_i and w is the specimen width. The DCB specimen is loaded linearly to P_1 where the crack begins to extend. During crack extension from a1 to a_2 , the load drops to P₂. If the specimen is then unloaded, the loss in strain energy due to crack extension is simply the area, ΔA_{12} , between the loading and unloading curves, as shown in Fig.1.



Figure (1) Area method

Experimental Part Material used

- 1- Woven roving glass fiber
- 2- Epoxy resin and hardener
- 3- Micro SiO₂ particles (100um)
- 4- Nano SiO₂ (12nm)

1. Epoxy resin preparation

Epoxy resin type (Quick mast 105) was provided by DCP Company /Jordan, used with its hardener in ratio (1:3), the epoxy mixed with hardener in a container then the mixture was used to prepare composites.

2. Preparation of Micro-composites

1- SiO_2 microparticle of particle size (100µm) were used with (2,4,6,8 and 10) wt%., by adding with a mixture of Epoxy resin with hardener as mentioned above.

2-To prepare micro- composites, the mixture of micro SiO_2 particles with Epoxy resin and hardener were distributed on woven roving glass fiber using brush, rolling brush was used to entrapped the air bubbles from specimen.

3- The same procedure was repeated until 16 plies of glass fiber were attained.

4- The specimens were left to cure for 24 hours at room temperature.

3. Preparation of Nano-composites

The mixing process consisted of three steps. Firstly, the fumed nano SiO_2 particles were stirrer mixed with the epoxy resin, with different weight ratios (2,4,6,8 and 10) wt%., the mixing was done at 60 °C with a magnetic stirrer for 120 min. The mixture left to cool then hardener was added to the mixture.

Using paraffin wax, sheet of glass fiber was stick on glass plate, then the mixture was distributed using brush, rolling brush was used to entrap the air bubbles from specimen.

4. Preparation of DCB specimens

Hand lay –up technique was used to prepare 16-plies of woven roving glass fiber /epoxy resin, to prepare the DCB specimens with loading system type (end block) which was very simple and easy to attach to the beams of the specimen. An Aluminum foil of thickness (0.02 um) was inserted in the midplane denoted by a, with an insert length of 50 mm, during the lay-up process for creating the pre-crack.

To monitor the position of crack front, the side of specimens was painted white and marked at 10mm intervals. A pair of metallic hinges was glued to the loading end of the specimens in order to enable the load to be applied. The dimension of specimen according to ASTM (5528-01) was 250 mm long, 20 mm wide and 10 mm in thickness, Fig.2.

Instron testing machine (1122) was used with cross head speed 0.5mm/min, the set up was shown in Fig.3. Load and displacement data were chart recorded; the crack length and crack propagation was measured visually using the traveling microscope.



Fig.2: Specification of DCB specimens[10]



Fig.3: Instron testing machine with DCB test

Results and Discussion

In this section, load - displacement curve for woven roving mat glass fiber was investigated, Fig.3, in sector 1, the curve shows three regions, linear region which occur at the beginning of crack propagation followed by non linear region due to the failure mechanism supported by crack propagation and delamination. Fast and unstable crack propagation was observed in woven roving composites.

At linear region, the load was proportional to the small displacement, that mean that when the arm of the cantilever move on the direction of load, there are no bending effects occur along the arms in this stage. All the load which applied on the arms would be converted to accumulated energy at the end of primary crack, so that when the strain energy reach the value larger than the surface energy, the load will be decreased and the crack propagate. During the unloading, linear behavior was observed until the load become zero, this curve does not reach the starting point, (i.e. original point for loading curve), this means that there was a little deformation in the arm of specimens, leading to little residual displacement.



Fig.4: Load – Displacement curve in woven roving glass fiber /Epoxy composites

Under loading there was a transfer of plastic deformation from up and down layer into the mid plane crack without delamination as in Fig.4, it depend on the adhesion force between matrix and fibers. The unstable crack propagation can be characterized by saw tooth shaped load profile, as seen in sector 1. In sector 2, slow crack propagation was observed, while fast crack propagation observed in sector 3. For composites without additives, G_{Ic} was (548J/m²), interlaminar fracture toughness, G_{Ic} was effected by the particle size filler, the addition of micro SiO₂, Table1 to woven roving glass fiber /epoxy composites increased the G_{IC} and was less than nanocomposites. because the energy released in the crack depend on the inter particle distance between the matrix and filler, as the filler was increased, the more particles act as a barrier for crack

propagation, in this case the crack required higher energy to propagate though the specimen.

 Table 1: Interlaminar fracture toughness of micro & nanocomposites

miero a minocomposites		
Weight	Gc (J/m^2) for	Gc (J/m^2) for
Percentage	microcomposites	nanocomposites
of SiO ₂		
filler		
content		
0	548	548
2	1312	1907.2
4	1492	1704
6	2433	1167
8	1805	1516
10	2027	2127

The addition of nano SiO_2 particles to woven roving glass fiber / epoxy composites on the values of Interlaminar fracture toughness was shown in Table 1, the G_{Ic} values was higher, depend on different weight ratio (2,4,6,8 and 10) wt% .this was because of good interlocking between the matrix and filler and because nanoparticle behave as an obstacle for crack propagation , so that the energy released was found in the deformation of arms , this was shown in the residual displacement in the specimens .

Delamination occurs in both of micro and nanocomposites, this behavior reduced the stiffness of DCB arm and reduce the tendency of crack to propagate in the mid – plane layer. The delamination was reduced as the filler content was increased.

Conclusions

1- Interlaminar fracture toughness was higher in nanocomposites compared to microcomposites at low wt% percentage value of SiO₂ particles.

2- Delamination occurs in both of micro and nanocomposites and was reduced as the filler content was increased.

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