

## FRACTURE TOUGHNESS OF NANOMODIFIED EPOXY RESINS AND GLASS REINFORCED LAMINATES

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### Abstract

*The present work reports some preliminary experimental results concerned with the benefits deriving from the matrix nanomodification of epoxy resins and composite laminates made by vacuum infusion of woven glass fabrics. The following properties are investigated: mode I fracture toughness and crack propagation resistance for neat and clay-modified epoxy, interlaminar shear strength, mode I delamination resistance for base and clay-modified epoxy laminates.*

### 1 Introduction

One of the most important challenges in material science is to develop new materials or to modify current ones to get improved specific characteristics.

Towards this direction, the perspective of getting substantial improvements of mechanical properties at low nanofiller volume fractions and costs has arisen significant interest in the use of nanoclay modified epoxy resins as matrices for composite laminates [1-8].

As a matter of facts, the addition of a suited nanofiller, sufficiently dispersed and compatible with the epoxy resin, could be a smart solution to improve the interlaminar fracture behavior of traditional composites which is a weak matrix dominated property.

This paper presents the preliminary results of the ongoing studies carried out by the authors on the effect of nanomodification and its industrial potential by discussing the experimental data obtained on neat and nanomodified epoxy, as well as on neat and nanomodified epoxy laminates (ternary laminates). After a brief description of the adopted materials and the manufacturing process, chosen for their industrial potential, we will present and discuss the experimental results obtained on nanomodified resins and laminates.

### 2 Materials

A DGEBA-based epoxy resin (EC157) from Elantas-Camattini was used as matrix polymer in this study. In addition, an amminic hardener (W131) has been used. The fraction of this component has been the one suggested by the manufacturer for the neat epoxy (3:1) for each investigated nanofiller content. The combination of these components is especially suitable for infusion processes due to its very low viscosity and long average pot life at 25°C (135min declared). The main mechanical properties of the adopted epoxy system, as specified by the supplier, are summarized in Table 1.

The nanoclay used in this work was the organo-modified polymeric layered silicate, Nanocor I.30, (Nanocor, USA). The organocation rendering the silicate compatible with polymer matrices is octadecylammonium. It is suitable to be used in amine-cured epoxy resin systems because of easily dispersion and improvement on modulus, T<sub>g</sub>, chemical resistance and other properties.

Finally a twill glass fiber fabric VV-350T with generic sizing supplied by G. Angeloni S.r.l. has been used as reinforcement for neat and nanomodified epoxy laminates.

**Table 1.** Properties of EC157/W131 epoxy systems by Elantas-Camattini

E (GPa)	3.2-3.5
$\sigma_R$ (MPa)	68-76
$\varepsilon_R$	6-8%

### 3 Nanocomposite and laminate manufacturing

The nanoclay dispersion into the epoxy resin was made by a shear mixing (SM) process carried out with a DISPERMAT TU shear blender from VMA-Getzmann. Once added the nanoclay into the resin, the dispersion process worked at an average rate of 2000 rpm for about 40 min. This allowed to reach a good distribution and dispersion of the nanofiller, promoting nanoclay intercalation/exfoliation and cluster breaking.

In order to achieve finer results, a sonication process was carried out by means of a HIELSCHER UP 200s SONICATOR. It was always operated at constant duty cycle (50%) and amplitude (0.7) and continued for 10 min.

At the end of the sonication, the hardener was added into the nanomodified epoxy system previously obtained and a new SM process was realized for 5 min at an average rate of about 1000 rpm, just to improve the resin-hardener mixing.

Due to some catalytic effects of the nanoclays with the epoxy resin used and possible problems resulting by resin overheating, during the entire process the resin was cooled with the aid of an external water bath.

Before the infusion process, in order to reduce the amount of trapped air produced during SM and sonication, a degassing process was realized using a low-vacuum pump that induced a very low pressure into the resin's pot and promoted bubbles explosion.

1 hr of degassing process was enough to obtain a clear and translucent nanomodified resin. CT specimens were finally manufactured by directly pouring the nanomodified resin into the silicone moulds, whose geometry and size were in agreement with the ASTM 5045-99 guidelines [9].

Laminates were obtained, instead, by vacuum infusing degassed neat or nanomodified resin into a vacuum bag, where 16 layers of twill glass fabric were laid up, the overall final thickness being about 4 mm.

A 15  $\mu\text{m}$  thick aluminum film has been used to create a pre-crack on the DCB specimens, whose dimensions were in agreement with the ASTM D 5528-01 guidelines [10].

Demoulding has been finally performed after complete curing, followed by a post curing as suggested by the resin manufacturer.

### 4 Experimental results and discussion

The experimental program included the following tests:

- mode I tests on neat and nanomodified epoxy resin (CT specimens);
- mode I tests on neat and nanomodified epoxy laminates (DCB specimens);
- interlaminar shear tests on neat and nanomodified epoxy laminates.

The experimental results for the neat and nanomodified epoxy and for the epoxy laminates are presented and discussed in the following sections.

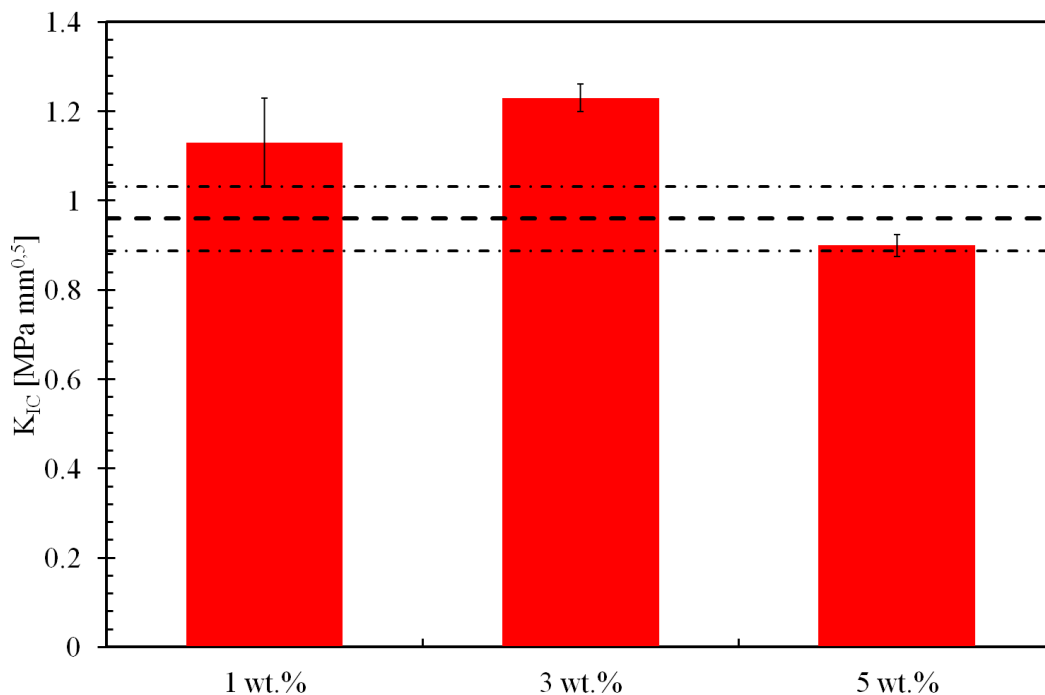
All tests have been carried out by using a MTS 858 servo-hydraulic machine, equipped with a 1.5/15 kN load cell.

#### 4.1 Nanomodified epoxy

Mode I static tests on CT specimens have been carried out. Tests procedures, specimens size and geometry were in agreement with the ASTM D 5045-99 suggestions [9]. The pre-crack were performed by manual tapping, giving rise to an average crack length of about 3 mm.

Four tests per percentage in weight of nanoclay conditions were carried out. The results are summarized in Figure 1. It is evident that, as expected, the fracture toughness of the clay-modified epoxy depends on the filler weight fraction. Generally speaking, an increase of the nanofiller weight fraction results into an improvement of the fracture toughness of the nanomodified system, up to a maximum value beyond which the experimental data show a leveling or even a worsening.

In particular, the experimental data shown in Figure 1 exhibit a significant improvement in terms of  $K_{IC}$  with respect to the neat epoxy resin for 1 wt.% I30 nanocomposite (+18%) and for 3 wt.% I30 nanocomposite (+29%). The same does not hold valid for 5 wt.% I30 nanomodified epoxy, for which, instead, a  $K_{IC}$  value slightly lower than that of the matrix has been obtained (-5.5%).

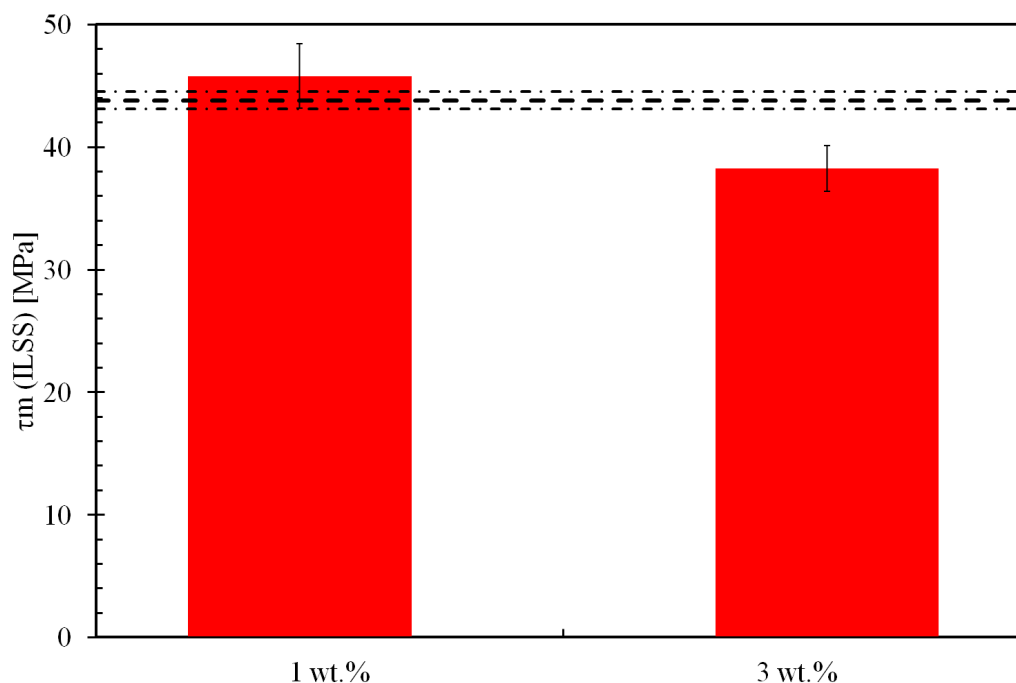


**Figure 1 .** Fracture toughness versus nanoclay content (4 tests per condition). The scatter band of the values for the neat resin is given by the dashed lines.

#### 4.2 Clay modified epoxy laminates

For ternary laminates, matrix toughness improvement itself is the most interesting and promising result, the interlaminar fracture behaviour of traditional composites being a weak matrix dominated property. In the previous section it has been proved that I30 nanomodified epoxy resin exhibits higher fracture properties with respect to the neat resin.

In order to evaluate the laminate interlaminar properties, interlaminar shear (ILSS) tests and mode I interlaminar fracture toughness quasi-static tests (DCB specimens) have been carried out.



**Figure 2 .** Comparison of interlaminar shear properties for neat and nanommodified epoxy laminates (the scatter band for neat epoxy laminates is given by the dashed lines).

ILSS tests have been carried out, according to ASTM D2344 [11]. The specimen thickness was 4 mm and the span was 10 mm. The crosshead speed was set at 1 mm/min and five specimens were tested for each material configuration. During the tests all the specimens failed by delamination in multiple shear condition. The experimental results are summarized in Figure 2, where a comparison between interlaminar shear strength of the neat, 1 wt.% and 3 wt.% I30 nanommodified epoxy laminates is shown. It is evident that, while 1 wt.% I30 nanommodified epoxy laminates exhibit an interlaminar strength slightly higher than that of the unmodified laminates, nanommodification with 3 wt.% I30 results in a significant worsening of the interlaminar shear strength.

DCB quasi-static tests have been also carried out, according to ASTM D5528 [10]. Four double cantilever beam (DCB) specimens for each material configuration have been tested. The crosshead speed was set at 0.5 mm/min. The mode I strain energy release rate values were computed using the compliance calibration method (CCM). The initiation values for  $G_I$  were determined according to three methods, as suggested by ASTM regulations: deviation from linearity (NL), visual observation and 5 % Offset/Maximum Load (5%).

The experimental results are shown in Figure 3, the initiation values being determined according to 5 % Offset/Maximum Load.

The obtained results on DCB tests allow to draw the following conclusions:

- an improvement in terms of initiation values has been achieved by nanommodification. In particular, 1 wt.% and 3 wt.% nanommodified laminates exhibit increments of about 10% and 31%, respectively, with respect to the unmodified laminates (Figure 4);
- the same conclusion cannot be drawn as far as propagation values are concerned. In this case nanommodified laminates exhibit a slightly worse behavior than the neat epoxy laminates.

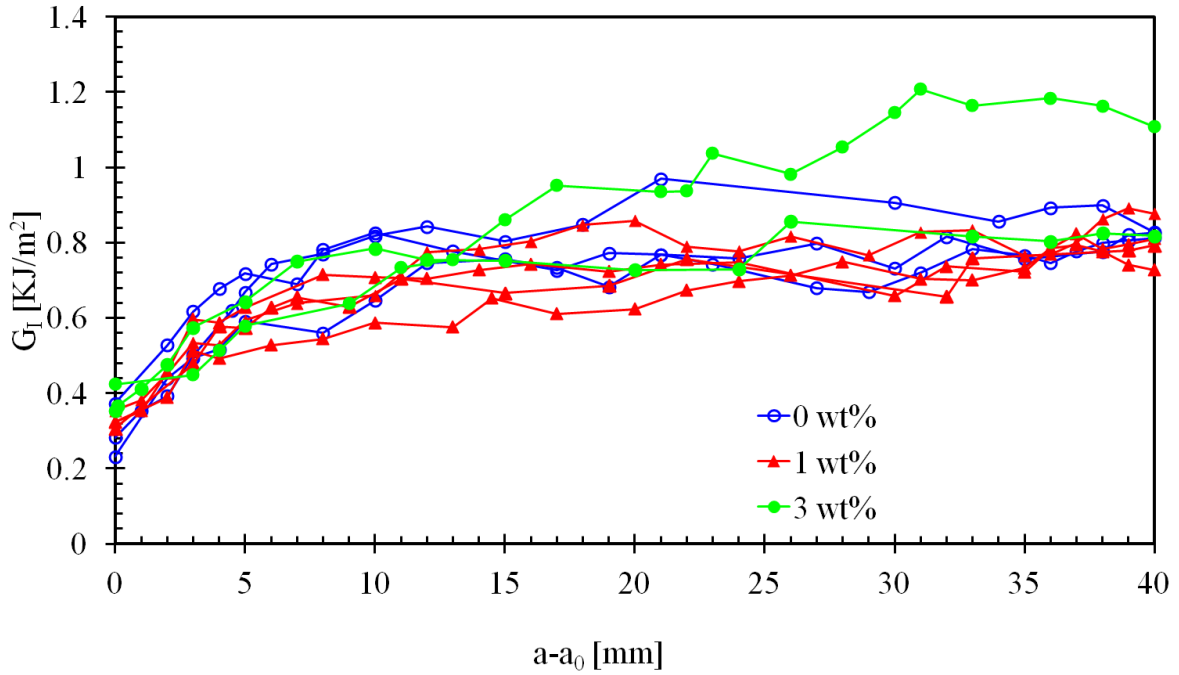


Figure 3. R-curves for neat and nanomodified epoxy laminate DCB specimens.

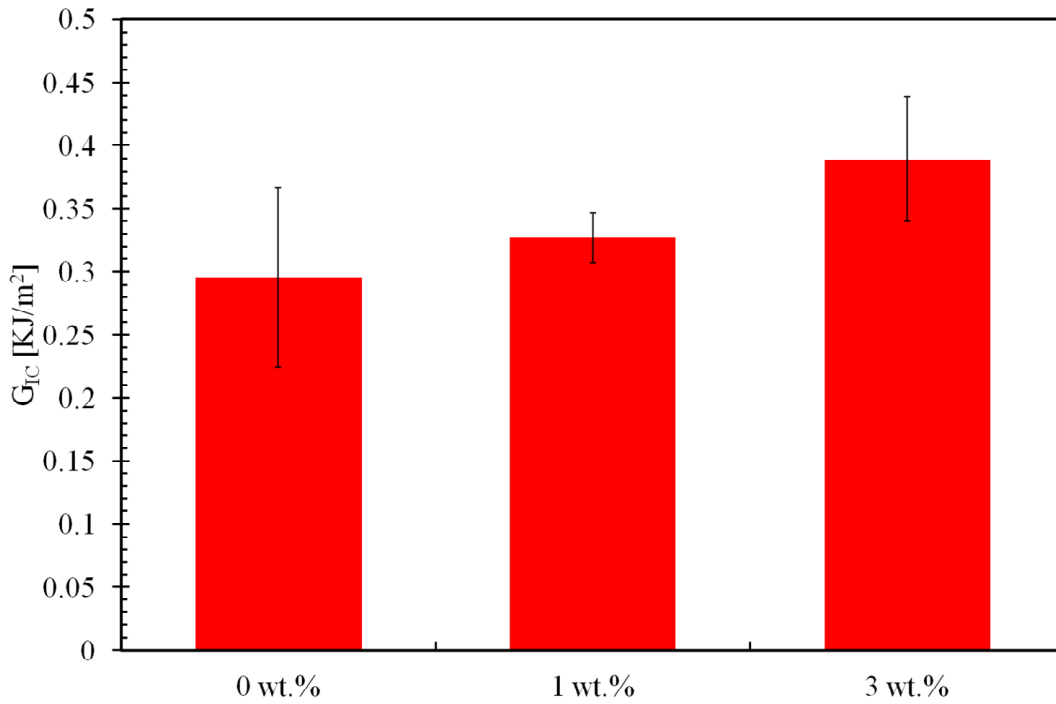


Figure 4. Initiation fracture toughness for neat and nanomodified epoxy laminate DCB specimens.

## 6. Conclusions

In the present work some preliminary experimental results concerned with the benefits deriving from the matrix nanomodification of epoxy resins and composite laminates made by vacuum infusion of woven glass fabrics have been reported. The following properties have been investigated: mode I fracture toughness and crack propagation resistance for neat and clay-modified epoxy, interlaminar shear strength, mode I delamination resistance for base and clay-modified epoxy laminates.

Available results indicate significant improvements in the fracture toughness and crack propagation threshold of clay modified epoxy.

On the other hand, the behaviour of clay-modified laminates is almost comparable to that of the neat epoxy laminates.

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