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► **To cite this version:**

Maurizio Avella, Ramiro Dell'Erba, Ezio Martuscelli, Richard Partch. Thermosetting based composites reinforced with silicon carbide whiskers. *Journal of Polymer Materials*, 2000, 7, pp.443-456. hal-01998490

**HAL Id: hal-01998490**

**<https://hal.science/hal-01998490>**

Submitted on 5 Mar 2019

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This paper is a pre-print. The final paper is available on:

Journal of Polymer Materials “Thermosetting based composites reinforced with silicon carbide whiskers ”, M. Avella, R. dell’Erba and E. Martuscelli. Vol. 7, p.443-456 (2000).

# Thermosetting Based Composites Reinforced with Silicon Carbide Whiskers

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## ABSTRACT

*The curing process and the mechanical performances of two composite systems constituted by thermosetting matrices (a tetrafunctional epoxy and a phthalic polyester) reinforced by silicon carbide whiskers have been investigated. In order to improve the interactions at the matrix/reinforcement interface, different coupling agents were experimented to cover the whisker surface. In particular two series of coupling agents, having inorganic and organic nature, were used. Thermal and mechanical properties of prepared composites have been compared and discussed, together with the morphology of their fractured samples. The results obtained have shown that the final properties of the composites strongly depend on the coating agent used. Finally, to evaluate the competitiveness of the whiskers with respect to other reinforcements, the specific mechanical properties of composites have been compared with the homologues having glass fibers as the second phase.*

KEY WORDS: Composites, silicon carbide, epoxy, polyester, coupling agents, morphology, interface.

## INTRODUCTION

Silicon carbide whiskers (SiCw) are a very interesting filler for composites: in fact, owing to their small diameter, they have very few defects in crystalline structure resulting in values of mechanical parameters very close to the theoretical limits expected from the elasticity theory<sup>1</sup>. For the first time whiskers were used especially in ceramic-ceramic composites<sup>2-6</sup> and very few attempts exist, on our knowledge, to use them as reinforcement of brittle resins<sup>7</sup> or polymers<sup>8</sup>.

In composite materials a good adhesion, together with high homogeneity of dispersion of

the filler in the matrix, is important to improve their mechanical properties. As a matter of fact, whiskers clustering can act as defect regions reducing the final properties of the material. The surface interaction energy between whisker-whisker and whisker-matrix is responsible for cluster formation. To achieve a good mixing it is required that the interactions between whisker and matrix are favorable to avoid the clustering of the whiskers.

The purpose of this paper was to prepare and characterize some epoxies and polyester based composites, reinforced by SiCw. Particular attention to the interactions between the whiskers and the matrix at the interface was

addressed. This latter aim was obtained by varying the chemical nature of whisker surface by coating them with a layer of different substances, like titanium nitride (TiN), titanium dioxide (TiO<sub>2</sub>), poly(divinylbenzene), 21 wt % poly(divinylbenzene) and then 5 wt% grafted poly(methymethacrylate), 21 wt% poly (divinylbenzene) and then 11 wt % grafted poly (glycidylacrylate). In this way the influence of two families of coating materials (inorganic and organic) was investigated.

Thermal, mechanical tests, and morphological analysis were performed to obtain a correlation between the macroscopic behavior of the composites and the morphology of the samples.

## EXPERIMENTAL

### Materials

Silicon carbide whiskers having an average diameter of 3 micrometer and 50 micrometer length were supplied by Advanced Refractory Technologies Inc., Buffalo, New York (USA). Several coatings, as listed in Table 1, were used to cover the whisker surface and the thickness of the coating layer was approximately 1/5 of the average value of the filler diameter.

TABLE 1. Characteristics of Resins and Reinforcements used.

Materials	Code
Epoxy resin	ER
Polyester resin	UP
Neat silicon carbide whisker (SiCw)	SiCw1
SiCw coated by titanium nitride	SiCw2
SiCw coated by titanium dioxide	SiCw3
SiCw coated by poly (divinylbenzene)	SiCw4
SiCw coated by 21 wt% poly (divinylbenzene)	SiCw5
and then 5 wt% grafted poly(methymetacrylate)	
SiCw coated by 21 wt % poly (divinylbenzene)	SiCw6
and then 11 wt% grafted poly (glycidylacrylate)	
Glass fiber	GF

The standard procedure to coat the whiskers surface was the following (e.g. sample SiCw 6, see Table 1): 12.5 g of whiskers have been dispersed in a solution containing 440 mL mineral spirits, 0.5 mL 4-vinylpyridine, 5 mL divinylbenzene and 0.5 g benzoyl peroxide. Next the solution

was stirred at 100°C for 18 h. The reaction mixture was cooled to 50°C and 0.8 mL methyl methacrylate and 0.5 g benzoyl peroxide were added dropwise. The mixture was heated at 90°C for 4h and filtered using 0.2 μm polycarbonate membrane and the residue was dried at 50°C and 20 torr of pressure for 5 h.

Glass fibers CP713 supplied by Vitrofil S.p.A. (Vado Ligure, Italy) E glass coated by a γ-amine propyltriethoxysilane and have a typical radius of 5 micrometer. The fibers (GF) were chopped in short filaments (max. 1 mm long) by an electric grinding mill, while whiskers were used as received.

The epoxy resin (ER) is a tetrafunctional epoxy resin, trade name MY 720 CIBA-GEYGY, Switzerland, tetraglycidyl 1 - 4, 4' p-diaminodiphenylmethane, (TGDDM). The hardener was an aromatic diamine, 4, 4'-diaminodiphenylsulfone (DDS).

Lonza Inc. (Switzerland) supplied the polyester resin (UP), trade name 270SW12. In the UP formulation 0.1% by weight of hydroquinone was used as inhibitor to prevent premature curing.

### Specimens Preparation

*Epoxy based composites.* Epoxy resin was prepared at 115 °C in a round bottom flask under vigorous stirring for 15 min at a rate at 100 RPM. The hardener used was 2 wt %, 4, 4'-diaminodiphenylsulfone (DDS), to obtain a complete dissolution of the hardener in the resin. Afterwards, the SiCw (10 wt%) were added and mixed for 15 min followed by degaussing for 20 min.

Successively, the blend was placed between two glass sheets and laterally contained in a 3.5-mm thick Teflon frame; the whole system was inserted in a stove and cured for 5 h at 190 °C.

*Polyester based composites.* The mixing procedure was similar to that used in epoxy based composites, but time and temperatures and the hardener used (dibenzylperoxide, 2 wt %) for the reaction were different. The mixing was effected at room temperature, owing to the low viscosity of UP resin, at a rate of 400 RPM for 15 min. The filler content was fixed at 10 wt % and the degassing was performed for 3 min only, to prevent ester loss. The curing process was different owing to the high residual strain and the exothermic reactions during cure; the specimens were immersed in a water bath, instead of a stove, and kept at 70°C for 7 h. Finally a post-cure of 2 h at 100°C was carried out by a stove.

### Techniques

The degradation process of the materials was investigated by thermogravimetric analysis (TGA). The TGA

of all samples was carried out with a Mettler M3/TA equipped with a TC10 processor; the experiments were performed in dynamic mode at a scan rate of 20°C/min. from 40°C up to 800°C in nitrogen.

The degree of curing of epoxy resin was measured by means of a Mettler TA-3000 differential scanning calorimetry (DSC), with a precision of 0.2°C in nitrogen. The residual heat of curing was measured to check the completeness of curing cycles of the resins.

The mechanical behavior of the composites was examined in different ways by dynamic, fracture, flexure, and heat deflection tests (HDT).

Dynamic-mechanical measurements were carried out by means of a dynamic thermal analyzer (DMTA MKIII Polymer Labs.) operating in a single cantilever bending mode (strain < 0.1%) at a frequency of 1 Hz. The samples, in the form of small bars (30 mm × 6mm × 6 mm), were examined in the temperature range -100°C to 320°C (0°C to 300 °C for UP based composites) with a heating rate of 1 °C/min.

The elastic modulus and the glass transition temperature ( $T_g$ ) were also measured.

An attempt to correlate the shift of  $T_g$  with an adhesion parameter of the fiber-matrix interface was performed. This can be done by defining the parameter  $A^{9,10}$ , by the following equation:

$$A = \frac{1}{1 - v_f} \cdot \frac{\tan \delta_c}{\tan \delta_m} - 1$$

being  $\tan \delta_c = \tan \delta_m + \tan \delta_i \cdot \tan \delta$  is the tangent of the phase difference between the stress and the strain in the composite ( $c$ ), the matrix ( $m$ ) and the interface ( $i$ ). The volume fraction of the filler is indicated by  $v_f$ . Therefore  $A$  can be positive or negative. Strong interactions between the filler and the matrix at the interface lead to reduce the macromolecular mobility close to the filler surface. This decreases  $\tan \delta_i$  and consequently  $A$ . Thus a low value of  $A$  indicates a higher degree of interaction between the phases, allowing a rough method to measure the adhesion for different kind to fillers in the same matrix.

Fracture tests were carried out with a Charpy Instrument Pendulum machine (Ceast Autographics Pendulum) at an impact speed of 1 m/sec. Samples with a notch depth to width ratio of 0.3 and a span length of 48 mm, 6.0 mm wide and 60 mm long were fractured at room temperature (ASTM D256). The relative curve of energy and load plotted against time or displacement were recorded. The impact data were analyzed according to the linear elastic fracture mechanics approach (LEFM)<sup>11</sup>. From this approach two parameters can be determined which accurately described the conditions for the onset of crack growth in the material. One is the critical stress intensity factor,  $K_{Ic}$ . The other parameter is the critical strain energy release,  $G_c$ , which represents the energy necessary to initiate the crack propagation. They are calculated according to Brown and Srawley<sup>11</sup> for  $K_{Ic}$  and to Plati and Williams<sup>13</sup> for  $G_c$ .

The flexural modulus of the resin based composites was measured with an Instron machine (model 1122).

HDT analysis was performed by a Perkin-Elmer thermomechanical analyzer (TMA) three points bending to determinate the deflection temperature of the samples under fixed load; the applied load was 0.15 MPa. The flexure of the sample vs temperature, at a scan rate of 20°C/min from 100°C to 350°C was recorded.

The fiber-matrix adhesion and morphological investigation were carried out with a Philips 501 SEM on fractured surfaces obtained by tensile and fracture tests; the samples, for SEM observation, were metallized by means of a Polaron sputtering apparatus with Au-Pd alloy, 18.10<sup>-9</sup> m thickness, approximately.

## RESULTS AND DISCUSSION

### Thermal Analysis

*Thermogravimetric analysis of epoxy based composites.* In Fig. 1a the thermogravimetric curves relative to the most meaningful epoxy resin (ER) based composites samples: ER (neat resin), ER/SiCw1, ER/SiCw2, ER/SiCw5, all 10 wt % are shown. The temperatures corresponding to the maximum rate of degradation of all ER based samples are reported in Table 2. A shift toward higher temperature is observed in the case of the composite materials: a slight increase (about 20°C) of the temperature at which 50% of the materials is degraded occurred in the composites. Two steps in the degradation process can be observed for all the materials.

TABLE 2. Temperature Corresponding to the Maximum Rate of Degradation for ER and UP based Composites Measured by TGA.

Sample Code	Max rate degradation temperature (°C)
ER	373
ER/SiCw1	375
ER/SiCw2	375
ER/SiCw3	375
ER/SiCw4	375
ER/SiCw5	383
ER/SiCw6	380
UP	400
UP/SiCw1	401
UP/SiCw6	401
UP/GF	399

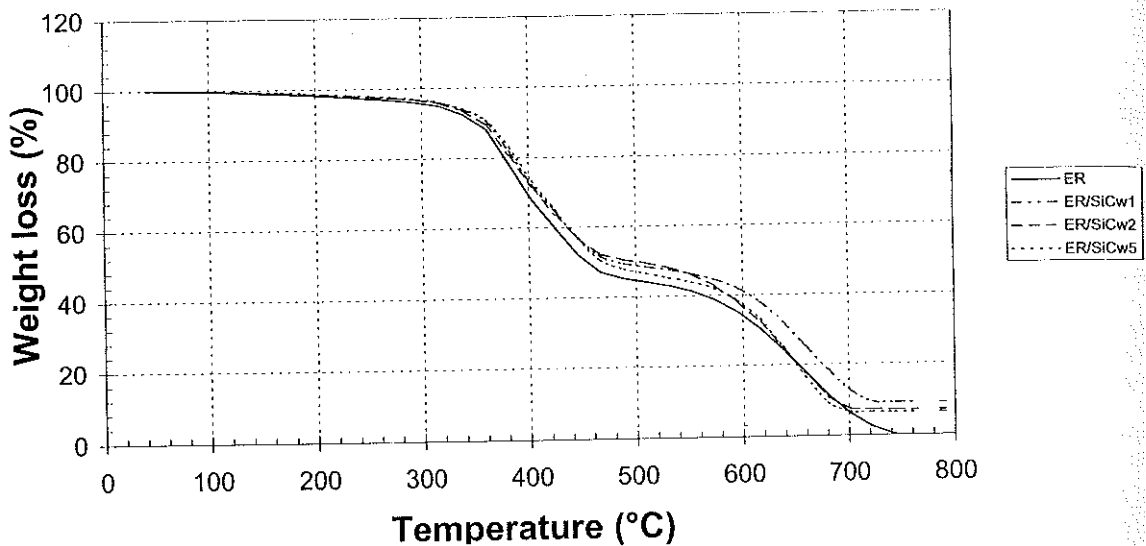


Fig. 1a. Thermogravimetric Curves of ER Composites Performed in Nitrogen at a Scan Rate of 20°C/min

This finding indicates that the interaction between the matrix and whiskers coating makes the composite more thermally stable with respect to the neat matrix. This effect was especially obtained in the case of ER/SiCw5 and ER/SiCw6 composites, while the other samples show little difference if compared with the resin reinforced by neat whiskers.

*Thermogravimetric analysis of polyester based composites.* In the case of UP based composite the shift of the onset temperature is much less pronounced (Fig. 1b). The temperatures corresponding to the maximum rate of degradation are shown in Table 2. No significant differences between these values can be outlined. The curves exactly overlap up to the temperature of 340°C, which is so high that the resin is completely degraded while the reinforcements are still thermally stable.

*DSC analysis.* The analysis by DSC was effected in order to study if the presence of whiskers could influence the completion of the curing reaction process. Cured and non cured samples were heated from 30°C to 300°C at a heating rate of 20°C/min. In Table 3 the residual heat calculated for some cured and uncured

samples, (samples ER, ER/SiCw1, ER/SiCw2, ER/SiCw5, ER/SiCw6, UP, UP/SiCw1, UP/SiCw6, UP/GF) are shown. It can be underlined that the residual heat of the cured samples is little (less than 10%) with respect to the uncured materials. No influence on the cure cycle due to the presence of different kinds of reinforcements can be detected.

TABLE 3. Residual Heat of Uncured and Cured ER and UP based Composites.

Sample Code	$\Delta H_{res}$ uncured (J/gr)	$\Delta H_{res}$ cured (J/gr)
ER	58	4
ER/SiCw1	50	2
ER/SiCw2	49	4
ER/SiCw5	47	5
ER/SiCw6	53	5
UP	65	6
UP/SiCw1	55	6
UP/SiCw6	53	4
UP/GF	50	3

### DMTA Analysis

DMTA analysis was performed on the ER and UP based composites, in the temperature range between 100°C and 320°C and between

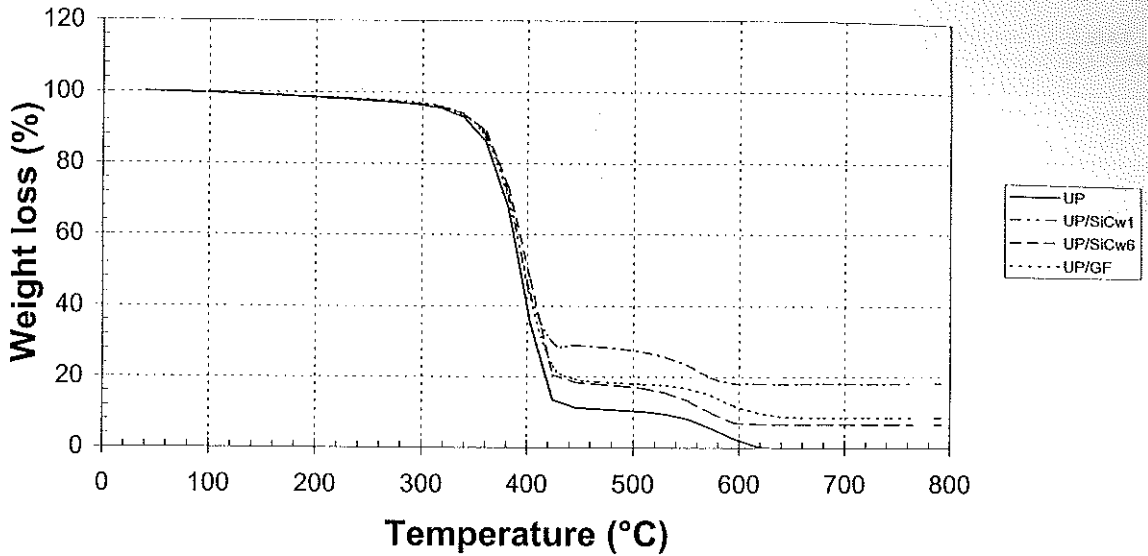


Fig. 1b. Thermogravimetric Curves of UP Composites Performed in Nitrogen at a Scan Rate of 20°C/min

100°C and 300°C respectively. From this analysis the flexural modulus, E, and the loss modulus,  $\tan\delta$ , vs temperature were carried out.

*Epoxy based composites.* The modulus E vs temperature relative to ER based composites (samples ER, ER/SiCw1, ER/SiCw2, ER/SiCw5)

in Fig. 2 is shown. To avoid the overlapping of the plots we have reported the modulus, calculated at the temperature of 100°C and 300°C, in Table 4. From these data the following can be deduced:

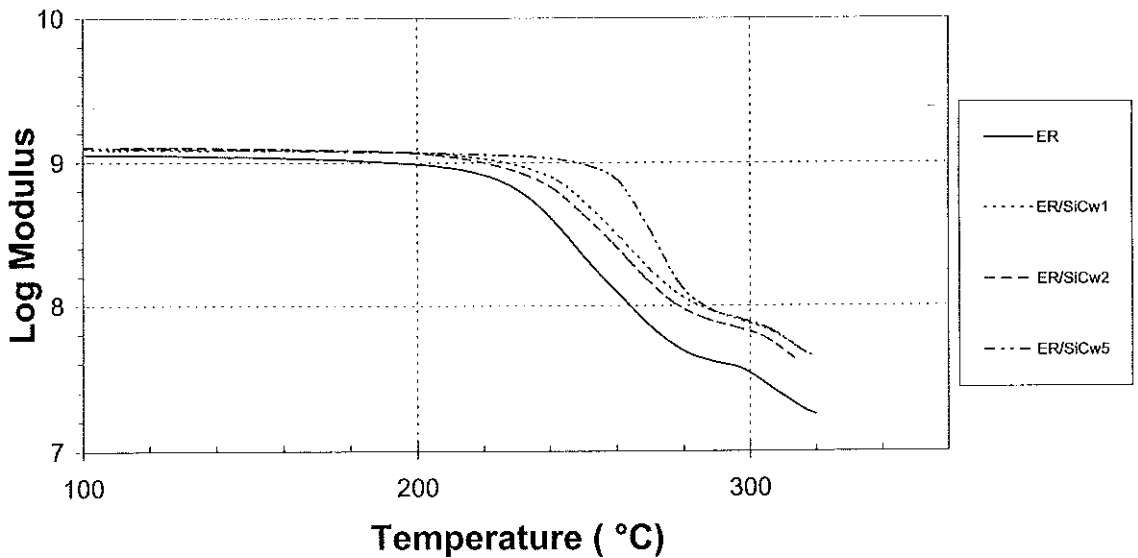


Fig. 2. DMTA Flexural Modulus, E, vs temperature for ER based Composites

TABLE 4. Storage Modulus, Glass Transition Temperature and A Parameter of ER based Composites

Sample Code	E(MPa) at 100°C	E(MPa) at 300°C	T <sub>g</sub> (°C)	A(T = 200°C)
ER	1132	25	260	0
ER/SiCw1	1243	50	266	-0.20
ER/SiCw2	1266	25	263	0.30
ER/SiCw3	1254	50	260	-0.20
ER/SiCw4	1325	68	264	0.18
ER/SiCw5	1369	76	273	-0.50
ER/SiCw6	1365	70	272	-0.45

1. The modulus E increases by the action of the reinforcements. The coated whiskers achieve better results with respect to the neat whiskers. At high temperature the composites filled by SiCw4, SiCw5, and SiCw6 (whiskers coated by organic coating) show a better performance with respect to the other composites and neat resin.

tion between the resin and the filler reduces the chain mobility in the interface volume and consequently enhances the T<sub>g</sub> of the composites. It can be outlined that the ER reinforced by SiCw5 and SiCw6, shows higher T<sub>g</sub> values indicating a better fiber-matrix adhesion probably due to the interaction of the coating formulations with epoxy functional groups.

These results are also supported by the examination of the interaction parameter A values, vs temperature shown in Fig. 3, for the samples ER/SiCw1, ER/SiCw2 and ER/SiCw5. In order to study the behavior of parameter A for all the samples, its value at 200°C for all the ER based composites is reported in Table 4. This temperature was selected in order to show the adhesion behavior at an intermediate temperature of the DMTA analysis. Also in this case, as can be seen from Table 4 and Fig. 3,

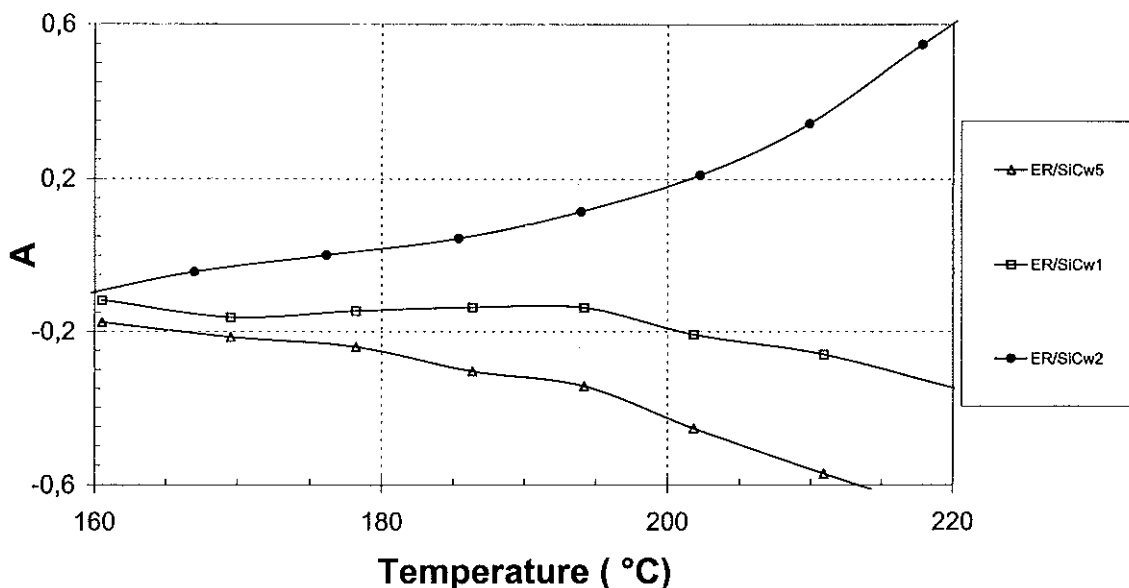


Fig. 3. Adhesion Parameter, A, vs Temperature for ER based Composites

2. The glass transition temperature, T<sub>g</sub>, is changed by the presence of the filler. A higher value of T<sub>g</sub> can be ascribed to a stronger adhesion between the whiskers and the matrix<sup>9</sup>. As a matter of fact, a good adhe-

the best result is obtained by the sample ER/SiCw5; its performance is very similar to the sample ER/SiCw6.

The results outlined from the behavior of parameter A reflect the behavior obtained from



the TGA analysis; so it can be noted that reinforcements SiCw6 and SiCw5 are able to achieve fiber-matrix adhesion better than uncoated whiskers. These results correspond to better mechanical properties and higher thermal stability. For other composite samples the coated whiskers show marginal improvement over the uncoated whiskers. From the above results it can be concluded that:

(a) An organic coating leads to a better mechanical properties with respect to the uncoated whiskers.

Table 5. Storage Modulus, Glass Transition Temperature and A Parameter of UP based Composites.

Sample Code	E (MPa) at 0°C	E (MPa) at 100°C	E (MPa) at 200°C	T <sub>g</sub> (°C)	A(T = 100°C)
UP	877	454	50	163	0
UP/SiCw1	1361	606	173	162	0.02
UP/SiCw6	984	577	121	162	0.08
UP/GF	893	589	174	164	-0.02

1. A strong enhancement of the modulus, with respect of the heat resin, is observed for

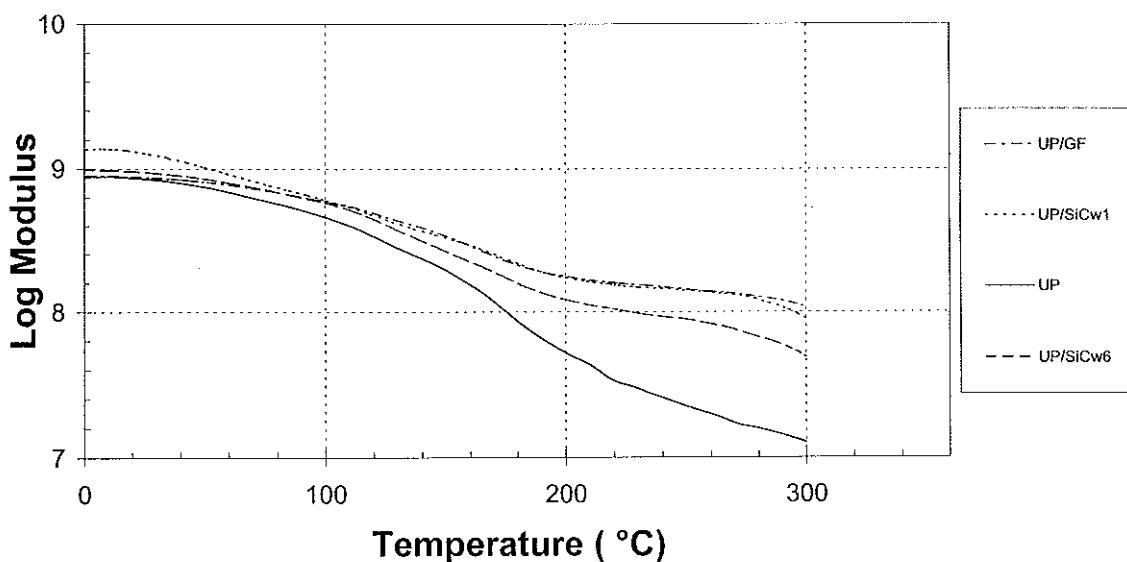


Fig. 4. DMTA Flexural Modulus, E, vs Temperature for UP based Composites

(b) An inorganic coating leads to a similar mechanical properties to those of the uncoated whiskers.

*Polyester based composites.* In Fig. 4, the flexural modulus, E, is plotted vs temperature for the UP based composites. In Table 5 the modulus at the temperatures of 0°C, 100°C, and 200°C for all the materials is shown. The following conclusions may be obtained:

the sample UP/SiCw1 in all the temperature ranges. The improvement of the modulus at 0°C was measured about the 50% (see Table 5 and Fig. 4). For temperature higher than 100°C the behavior of the sample UP/SiCw1 is similar to the sample UP/GF.

2. The sample UP/SiCw6 shows lower enhancement of the modulus, E, with respect to the sample UP/SiCw1, probably owing to

the fiber-matrix interactions indicating that, in this case, this coating is not the best for polyester resin, while it seemed to work for the epoxy matrix.

At high temperature the trend of samples UP/SiCw1 and UP/GF is very similar as can be seen from Fig. 4. At temperatures higher than 150°C, close to the  $T_g$ , the modulus drops much more for the neat UP with respect to the composites.

The adhesion parameter,  $A$  is shown in Fig. 5 to understand the relation between the fiber-matrix adhesion and the modulus for UP composites. Its value at 100°C in Table 5 is shown as in the case of ER based composites.

It should be underlined that a comparison between GF reinforced composites and those reinforced with SiC whiskers is difficult owing to the different nature and size of the fillers. However, indicatively, the trend of  $A$  shows the best result for UP/GF followed by UP/SiCw1 and UP/SiCw6.

### Mechanical Test and Morphological Analysis

In Table 6 the values of the flexural modulus,  $E$ , the parameters relative to the impact tests ( $K_c$  and  $G_c$ ) and the deflection temperature ( $T_d$ ) for the ER and PU based composites are reported.

From these data the following emerges:

1. An improvement of the flexural modulus,  $E$ , and of the fracture parameters ( $K_c$  and  $G_c$ ) is observed for the samples ER/SiCw1 (uncoated whiskers), ER/SiCw5 and ER/SiCw6 (organic coated whiskers). The enhancement of  $K_c$  was found more than 50%, while  $G_c$  is enhanced more than 100% in the case of SiCw1 and SiCw6 fillers.
2. The other coatings seem to improve only slightly the final properties of ER based composites.
3. The presence of SiCw does not produce strong influences in the  $T_d$  in all ER based composites.

TABLE 6. Values of Mechanical Parameters of the ER and UP Composites: Flexural Modulus,  $E$ , Critical Stress Intensity Factor,  $K_c$ , Critical Energy Release,  $G_c$ , Deflection Temperature,  $T_d$

Sample Code	$E$ (GPa)	$K_c$ (MN/m <sup>3/2</sup> )	$G_c$ (KJ/m <sup>2</sup> )	$T_d$ (°C)
ER	3.1	0.8	0.20	330
ER/SiCw1	3.7	1.2	0.44	330
ER/SiCw2	3.1	0.8	0.20	330
ER/SiCw3	3.4	1.0	0.36	330
ER/SiCw4	3.6	0.9	0.29	330
ER/SiCw5	4.0	1.2	0.38	335
ER/SiCw6	3.8	1.2	0.41	335
UP	2.3	0.52	0.17	330
UP/SiCw1	3.0	0.75	0.33	330
UP/SiCw6	2.9	0.78	0.35	325
UP/GF	3.0	1.55	1.3	350

In the UP based samples the presence of the whiskers influences positively the final properties of the composites. The enhancement of the modulus is about 25% for all the composites. The improvement of  $K_c$  is about 40% and 100% for  $G_c$  for the samples UP/SiCw1 and UP/SiCw6. Unfortunately, the presence of the whiskers do not achieve the performances of GF reinforced UP(UP/GF sample), probably owing to the different size of the glass fibers. Also in the case of UP based composites the deflection temperature is not strongly influenced by the whiskers; only in the UP/GF sample the presence of glass fibers causes a little increase of  $T_d$ .

### SEM Analysis

The morphological aspects of the mechanical behavior of the composites were investigated by SEM micrographs.

*Epoxy based composites.* In Fig. 6a-6d the micrographs of ER neat resin and composites are shown. All the pictures are taken near the notch tip in the region of crack initiation by the Charpy method fracture.

The typical behavior of brittle fractured surface, without any plastic deformation area, is seen in the micrograph of neat ER (Fig. 6a). In Fig. 6b the composite ER/SiCw2 (inorganic

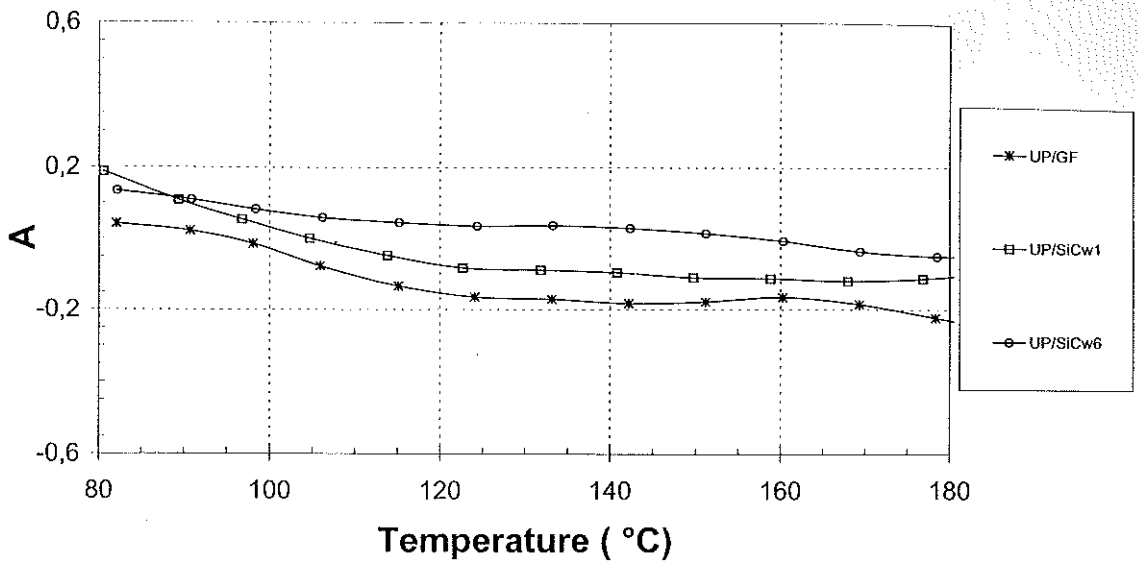


Fig. 5. Adhesion Parameter, A, vs Temperature for UP based Composites

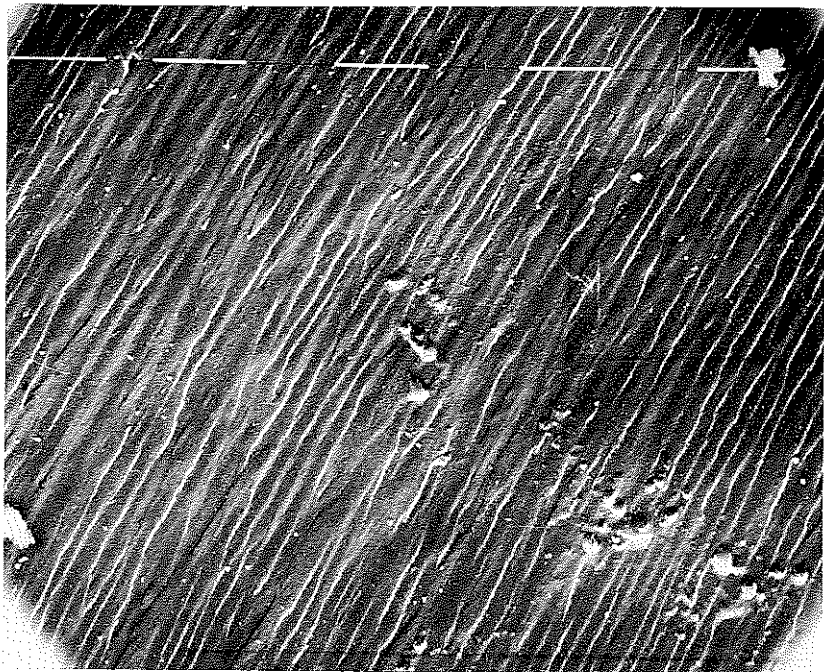


Fig. 6. SEM Micrographs; (a) Neat ER Resin 1250X;

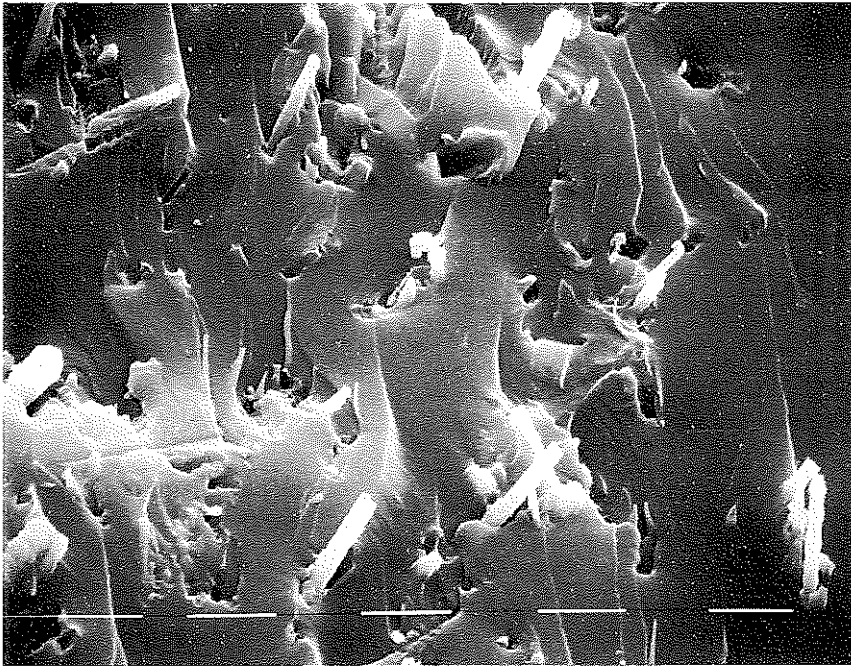


Fig. 6 (b). ER/SiCw1 Composite 1250 X;

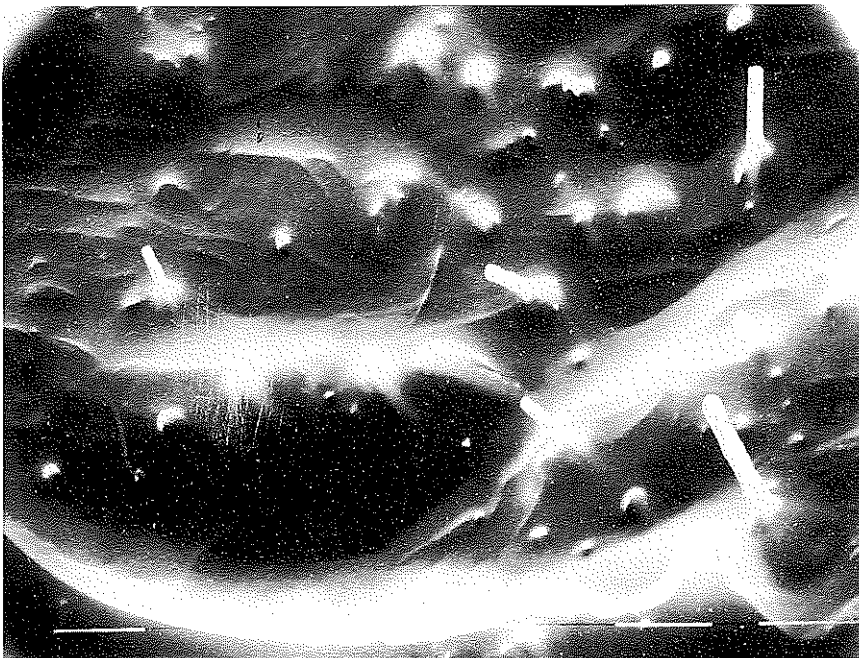


Fig. 6 (c). ER/SiCw6 Composite 1250X;

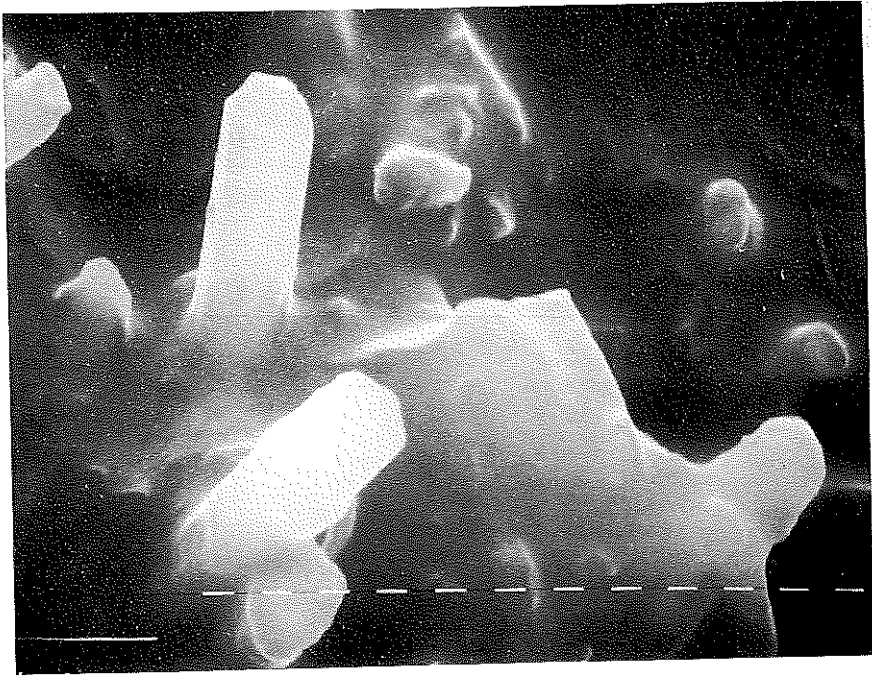


Fig. 6 (d). ER/SiCw6 Composite, 5000X;

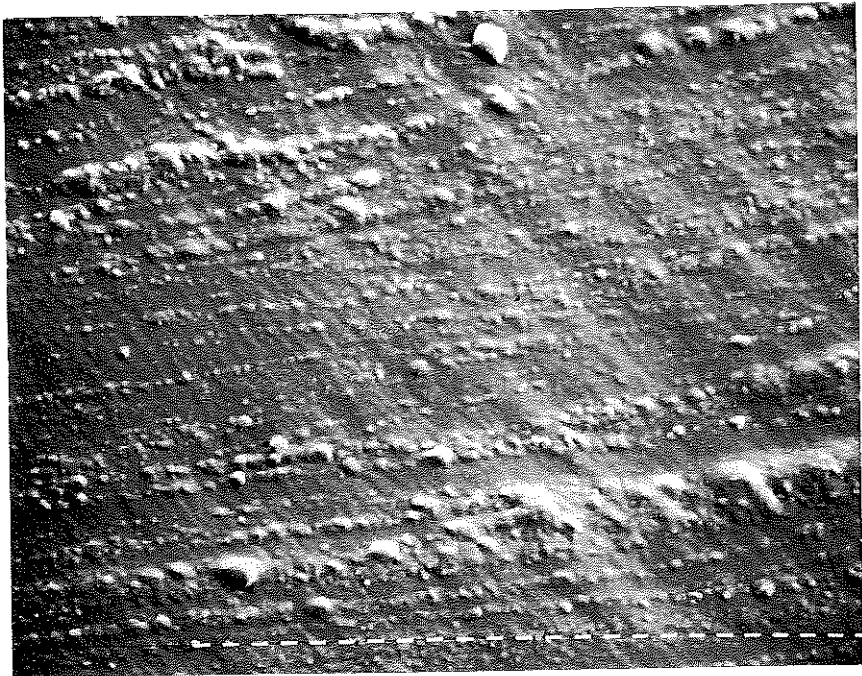


Fig. 7 (a). SEM Micrographs; (a) Neat UP Resin 2500 X;

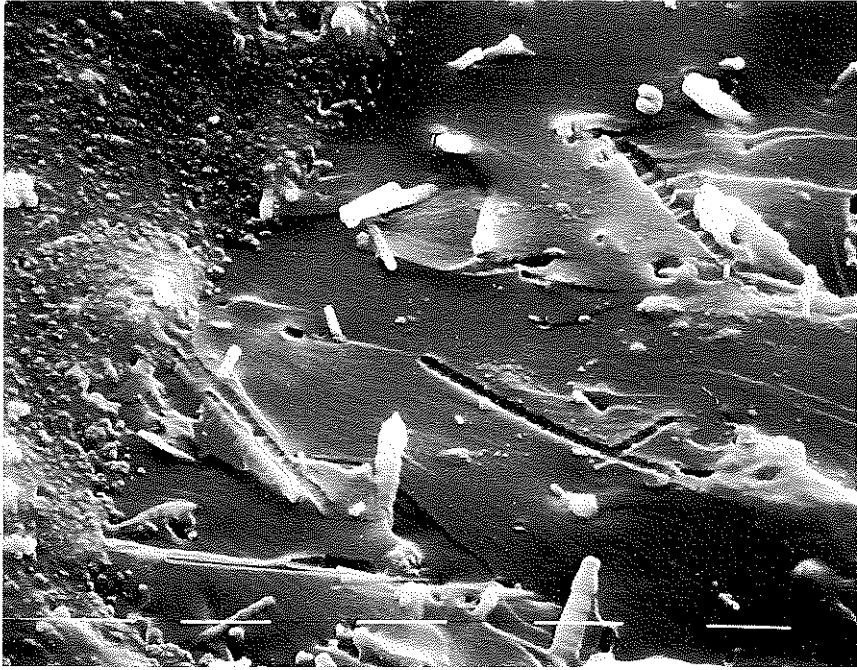


Fig. 7 (b). UP/SiCw1 Composite 1250X;

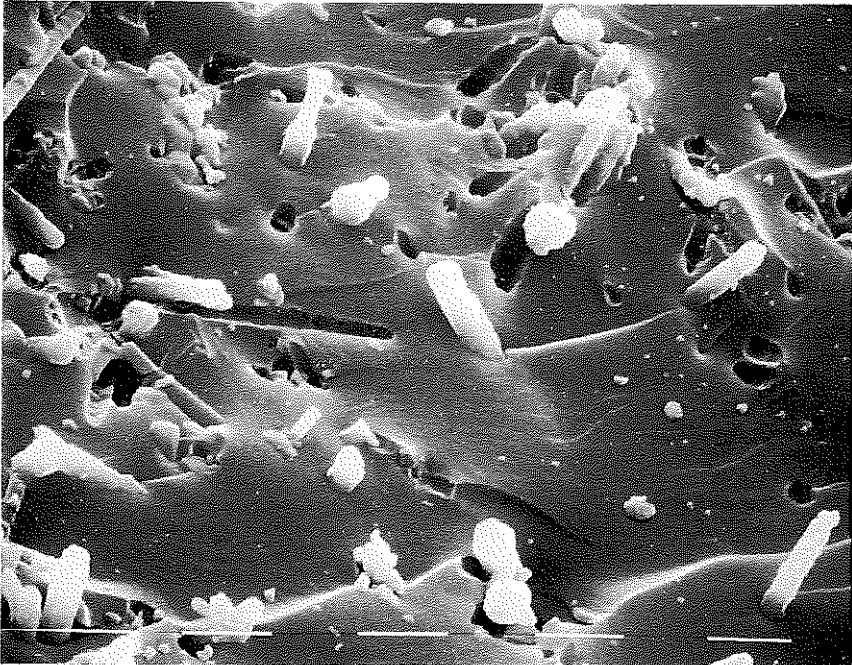


Fig. 7 (c). UP/SiCw6 Composite;



Fig 7 (d). UP/GF Composite 1250X;

coating) is shown; the whiskers appear poorly embedded in the matrix and no whitening owing to the stress suffered from the matrix can be seen in their neighbors. Many whiskers were pulled out from the matrix indicating a poor fiber-matrix adhesion. An enhancement of adhesion of the matrix with the filler may be seen in Fig. 6c (sample ER/SiCw6), and in the magnified picture (Fig. 6d). Presence of cluster aggregation is not observed.

*Polyester based composites.* The fractured surface of neat UP is shown in Fig. 7a; where a brittle fracture is noticed. In Fig. 7b the sample UP/SiCw1 appears without whisker clustering phenomena. However, many holes are visible where the whiskers were pulled out from the matrix. Particularly many holes, without whitening stress of the matrix, demonstrate that the filler was pulled out clean from the matrix.

From Figs. 7b and 7c there is no significant difference between the fractured surface of sample UP/SiCw1 and sample UP/SiCw6. On the contrary, in Fig. 7d, (sample UP/GF) many

cracking areas near the pulled out glass fibers can be noticed, demonstrating the stronger stress transmitted to the fiber from the matrix with respect to the other composites. However, it must be underlined that the different sizes of the GF filler lead to different stress distribution on the fiber surface.

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