

# Epoxy/Alumina Nanoparticle Composites. II. Influence of Silane Coupling Agent Treatment on Mechanical Performance and Wear Resistance

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**ABSTRACT:** Two silane coupling agents (SCAs) were used to treat alumina nanoparticles to improve nanoparticle dispersion into the polymer matrix. [3-(2-Aminoethyl-amino)-propyl]-trimethoxysilane [a reactive silane coupling agent (RSCA)] was able to form covalent bonds with both the reinforcing agent (alumina nanoparticles) and the epoxy matrix, whereas 3-(trimethoxysilyl) propyl methacrylate (a nonreactive SCA) could not covalently interact with the epoxy resin. The strengthening of the filler–matrix interactions by means of covalent bonds resulted in improved flexural strength, strain, and especially, wear resistance. The

wear rate and the weight loss of the RSCA-treated alumina nanoparticle composite were the best among the investigated composite samples and equal to these of the neat epoxy matrix. This improvement was due to better nanoparticle dispersion, a result of the SCA treatment of the alumina nanoparticles. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 101: 4410–4417, 2006

**Key words:** matrix; mechanical properties; nanoparticles; surfaces

## INTRODUCTION

The growing interest in nanotechnology and nanomaterials is based on the fact that when nanosized, materials exhibit new physical, chemical, optical, or electronic properties that a single molecule or the same material in bulk does not possess. A very peculiar property of nanoparticles is their large specific surface area, which defines a large matrix–filler interphase when the nanoparticles are incorporated into a matrix.

Several methods are presently mostly used to produce polymeric nanocomposites, including the sol–gel route,<sup>1,2</sup> *in situ* intercalative polymerization,<sup>3</sup> and *in situ* polymerization.<sup>4,5</sup> However, there have been few studies on nanocomposite preparation by direct mixing of nanoparticles and the polymer matrix, especially for thermoset matrices, so knowledge about this topic and what can or cannot be accomplished by it is still incomplete.

The main problem that has to be solved for the handling of nanoparticles is their agglomeration. Because of their high specific surface area, nanoparticles aggregate between themselves very easily. The nano-

particle aggregates are bigger than nanometer scale, are very strong, and behave as independent particles, so the specific properties that the nanosize implies are lost. When inorganic nanoparticles are directly incorporated into an epoxy resin matrix, two possibilities for overcoming nanoparticle agglomeration exist: (1) the application of strong shear forces during the mixing process and (2) the prevention of agglomeration by preliminary surface treatment of the nanoparticles, for example, with silane coupling agents (SCAs). In a previous study,<sup>6</sup> both methods were applied to prepare epoxy/alumina nanoparticle composites, and the dynamic mechanical behavior of these composites was studied. In this study, we went further by studying the mechanical properties (modulus, strength, deformation, and microhardness) and the wear resistance of these materials.

Generally, rigid particulate fillers increase the modulus and cause a dramatic decrease in the elongation at break of composites compared to the neat polymer matrix.<sup>7</sup> Also, these fillers often reduce the tensile strength of composites. Usually, fillers decrease the wear rate (*W*) of the polymer if there is strong adhesive filler–polymer bonding. Hard compounds, such as alumina (in micrometer size or larger), are not suitable as fillers for the preparation of wear-resistant materials because of their abrasive effect on the counterface. However, as Schwartz and Bahadur<sup>8</sup> assumed, hard nanosize particles might be suitable as fillers for

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materials for sliding applications because the angularity of the particles decreases with their decreasing size, so they would not be expected to abrade the counterface. In addition, nanoparticles have two more advantages as matrix fillers for sliding wear applications compared to bigger filler particles:<sup>8</sup> (1) they will be able to blend well with the worn particles, thus strengthening the transfer film, and (2) as the nanoparticles have a large surface area, this will significantly improve the adhesion between the filler and the polymer matrix and reduce the wear of the material. For common composites with fillers of micrometer size and bigger, such an improvement of the interfacial adhesion is known to enhance the mechanical performance of the composite materials and is normally achieved by the surface treatment of the fillers, for example, by coupling agents. The coupling agent is a chemical additive that provides a way to overcome the incompatibility of hydrophobicity and hydrophilicity between a polymer and a solid inorganic surface. If the coupling agent can chemically react with both the polymer matrix and the filler, it can further enhance adhesion between them.

Surface-treated fillers often give composites with increased tensile strengths. Surface treatment of the filler particles may also change the state of their agglomeration in addition to acting as a coupling agent. Also, coupling agents may change the wetting of the filler by the polymer so that there will be fewer voids and bubbles of entrapped air. Thus, treating the surface of the filler particles can improve various properties of the composite materials.<sup>7</sup> This fact defined our interest in treating alumina nanoparticles with SCAs as a way to overcome their agglomeration before we incorporated them into an epoxy matrix. The influence of the surface treatment on the mechanical performance of the final composites was thoroughly studied.

## EXPERIMENTAL

### Materials

General-purpose epoxy resin DER 331, a reaction product of epichlorohydrin and bisphenol A (Dow Chemical, Ibbenbueren, Germany) was used as the matrix. The hardener, 2,2'-dimethyl-4,4' methylenebis-cyclohexylamin, was purchased from Vantico, Ltd. (Bergkamen, Germany). Alumina nanoparticles ( $\text{Al}_2\text{O}_3$ ), with a size of 40 nm and a specific surface area of 41  $\text{m}^2/\text{g}$ , were purchased from Nanophase Technologies Corp. (Romeoville, IL).

Two SCAs were used for the nanoparticle treatment, namely, [3-(2-aminoethylamino)-propyl]-trimethoxysilane [a reactive silane coupling agent (RSCA)] and 3-(trimethoxysilyl) propyl methacrylate [a nonreactive silane coupling agent (NRSCA)], purchased

from Gelest, Inc. (Karlsruhe, Germany). Both SCAs reacted via the silanol groups (a result of the easy hydrolysis of their trimethoxysilyl groups) with the hydroxyl groups on the alumina surface, which thus formed covalent bonds, but they differed in their ability to form covalent bonds with the epoxy matrix. The first SCA could chemically react via its amino groups with the epoxy groups of the matrix, and it was designated as a RSCA. The second one could not react chemically with the matrix, and it was designated as a NRSCA.

### Nanoparticle treatment with SCAs and composite preparation

The nanoparticle treatment by SCA together with the composite preparation is described in detail elsewhere.<sup>6</sup>

### Characterization of the composite properties

The static mechanical properties of the epoxy/alumina nanoparticle composites were tested in a three-point bending mode with a Zwick 1474 instrument (Zwick GmbH and Co., Ulm, Germany). The size of the tested specimens was 10 mm  $\times$  4 mm  $\times$  80 mm. The maximum load applied was 10 kN at a testing speed of 2 mm/min; the distance between the clamps was 64 mm.

The sliding wear resistance of the composites was tested with block-on-ring equipment. The size of the tested samples was 4 mm  $\times$  5 mm  $\times$  5 mm. The tests were performed at room temperature against a rotating cylinder counterpart made from 100Cr6 steel with a surface roughness of 0.14  $\mu\text{m}$ . An apparent contact surface pressure ( $p$ ) of 1 MPa and a rotation velocity of 1 m/s were used. The experiments were carried out as a function of the wearing time, with four different testing times (4, 8, 12, and 20 h) used. For every set of conditions, three samples were tested, and the results were averaged.

$W$  was related to the worn volume of the sample ( $\Delta V$ ), the sliding distance ( $L$ ), and the apparent contact surface area ( $A_o$ ) as follows:

$$W = \frac{\Delta V}{LA_o} \quad (1)$$

We obtained the specific wear rate ( $W_s$ ) by dividing  $W$  by  $p$ :

$$W_s = \frac{\Delta m}{L_p F_N} \quad (2)$$

$\Delta V$  is defined as

$$\Delta V = \frac{\Delta m}{\rho} \quad (3)$$

where  $\Delta m$  is the weight loss during wearing obtained from the initial weight of the sample,  $w$  initial is subtracted from its final weight and  $\rho$  is the density of the material.  $p$  is defined as

$$p = F_N A_o \quad (4)$$

where  $F_N$  is the normal force.  $W_s$  has dimensions of volume/energy, and it is a measure how much energy is needed to remove a certain volume of the material. Wear is not a characteristic of the material itself but a characteristic of the system material counterpart.

The microhardness of the epoxy/alumina nanoparticle composites was tested with a dynamic ultramicrohardness tester (DUH-202, Shimadzu, Duisburg, Germany). This instrument allowed instant computer recording of the indentation depth as a function of the applied load and its subsequent removal. Hence, the universal microhardness was determined from the force-indentation depth curve. The Vicker's microhardness of the samples was determined from the diagonals of the indentation mark. Three different loads were used to eliminate the load dependence of the Vicker's microhardness.

Density evaluation was carried out with a Mettler density-measuring device (Giessen, Germany). The density of the specimens ( $D$ ) was evaluated with the following equation:

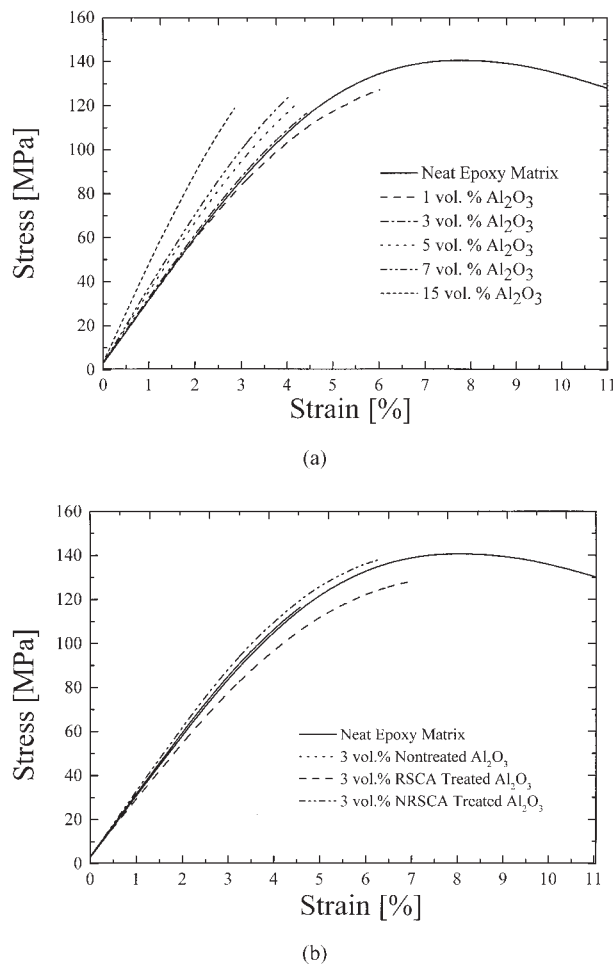
$$D = \frac{A(V_s + 0.001)}{(A - B)(A_s - B_s)} \quad (5)$$

where  $A$  is the sample weight in air,  $B$  is the sample weight in water;  $A_s$  and  $B_s$  are the weights of the standard in air and water, respectively;  $V_s$  is the standard volume (mL); and  $0.001 \text{ g/cm}^3$  is a correction coefficient. All weights here are in grams.

## RESULTS AND DISCUSSION

### Stress-strain behavior of the epoxy/alumina nanoparticle composites

The stress-strain curves for all epoxy/untreated alumina nanoparticle composites are shown in Figure 1(a). The curves were obtained after we averaged the curves obtained by the experimental tests of at least eight specimens. The slope of the curves, or the flexural modulus, increased as the alumina content increased. Also, the ultimate flexural strain drastically decreased compared to the neat epoxy resin, as was the case for common composites.<sup>7</sup> This was in accordance with the expectations that by direct mixing of the alumina nanoparticles into the epoxy resin, the



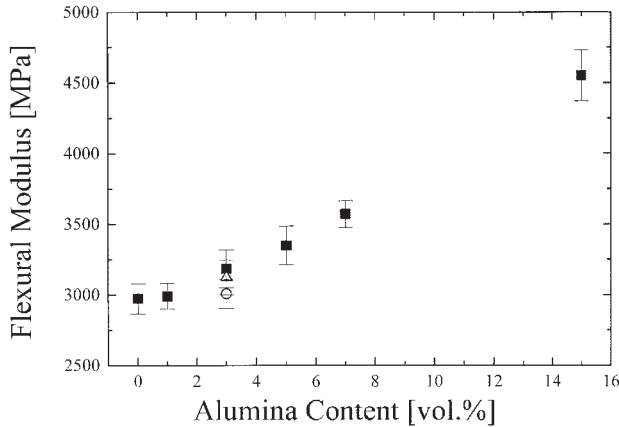
**Figure 1** Stress-strain curves for the neat epoxy resin, (a) epoxy/untreated and (b) epoxy/SCA-treated alumina nanoparticle composites.

nanoparticles existed, in fact, as very stable agglomerates. This means that most probably, a nanolevel dispersion could not be attained only by the application of strong shear forces during the mixing process. So, the final composites behaved as common composites filled with micrometer-size fillers.

The stress-strain curves for both composites containing SCA-treated alumina nanoparticles are shown in Figure 1(b). For comparison, the curves of the neat epoxy matrix and the composite containing the same amount (3 vol % alumina) but untreated nanoparticles are also presented. Both SCA-treated nanoparticle samples showed higher 3–4% ultimate flexural strain compared to the untreated nanoparticle sample.

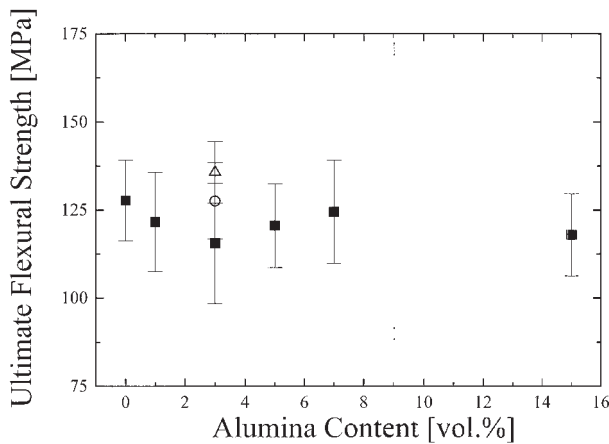
The dependence of the flexural modulus, ultimate flexural strength, and strain on the alumina nanoparticle volume content is more clearly shown in Figures 2–4. The corresponding values of these mechanical parameters are presented in Table I.

A linear increase of the flexural modulus was observed as the alumina content increased (Fig. 2). The

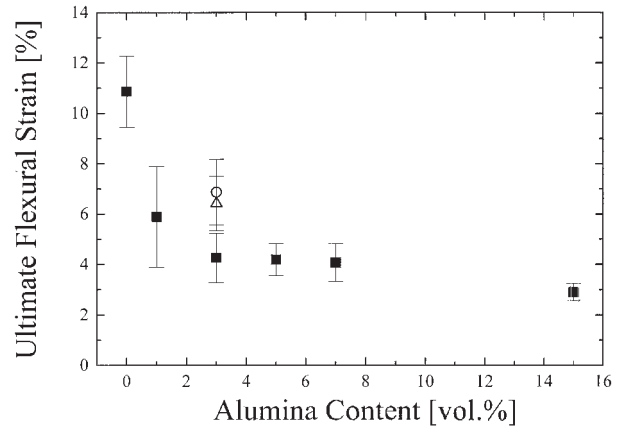


**Figure 2** Flexural modulus of the epoxy/alumina nanoparticle composites versus the alumina volume content for composites containing (■) untreated alumina nanoparticles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles.

sample with 1 vol % alumina had a modulus equal within experimental error to the modulus of the neat epoxy matrix. For 15 vol % alumina content, the modulus increased up to 4500 MPa. This could have been due to both (1) the nanoparticle reinforcing effect and (2) the higher alumina nanoparticle agglomeration as the filler content increased. The SCA treatment of the alumina nanoparticles resulted in a decrease in the flexural modulus compared to the corresponding untreated nanoparticle sample (Fig. 2). The lowest flexural modulus among all of the samples containing 3 vol % alumina was detected for the RSCA-treated alumina nanoparticle sample. A possible explanation of this observation could be the fact that the SCA treatment influenced the filler agglomeration state. It



**Figure 3** Ultimate flexural strength of the epoxy/alumina nanoparticle composites versus the alumina volume content for composites containing (■) untreated alumina nanoparticles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles.



**Figure 4** Ultimate flexural strain of the epoxy/alumina nanoparticle composites versus the alumina volume content for composites containing (■) untreated alumina nanoparticles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles.

is known that for high-density polyethylene filled with untreated and SCA-treated talc,<sup>9</sup> SCA changed the properties of the composite mainly through enhancing the filler dispersion and modification of the matrix morphology. Manna et al.<sup>10</sup> studied epoxidized rubber-clay mixtures, where a coupling agent that could chemically react with the matrix was used. The authors concluded that the enhancement in the physical properties in the presence of the coupling agent was due to better filler dispersion. Thus, the increase in the modulus due to the presence of the nanoparticle agglomerates for the SCA alumina-treated samples diminished because of better nanoparticle dispersion. As a result, both SCA-treated nanoparticle samples had lower flexural moduli (Table I).

The dependence of the ultimate flexural strength on the alumina content is shown in Figure 3. The scattering of the values of this parameter was quite large (large error bars), but a slight minimum at 3 vol % alumina content was observed. Schwartz and Bahadur<sup>8</sup> detected a similar minimum in a system similar to this: a poly(phenylene sulfide) (PPS) matrix filled with alumina nanoparticles with a diameter of 33 nm. These authors observed that the flexural strength of these composites passed through a minimum at 2 vol % alumina as the filler content increased.<sup>8</sup> They explained this minimum with a weakening introduced by the presence of a heterogeneous phase. With a further increase in the filler content, the strength increased due to mechanical strengthening from the hard particles. In Figure 3, the ultimate flexural strength of both SCA-treated samples is also shown. It was obvious that SCA treatment resulted in higher flexural stress values compared to those of the untreated nanoparticle sample (Table I). As already mentioned, very often, SCA treatment of the filler results

TABLE I  
Mechanical Properties of Epoxy/Alumina Nanoparticle Composites

Sample (vol % Al <sub>2</sub> O <sub>3</sub> )	Flexural modulus (MPa)	Ultimate flexural stress (MPa)	Ultimate flexural strain (%)	$\rho$ (g/cm <sup>3</sup> )
Neat epoxy resin	2972 ± 107	128 ± 11	10.9 ± 1.4	1.1419 ± 0.0017
1 vol % untreated	2989 ± 91	122 ± 14	6 ± 2	1.1667 ± 0.0003
3 vol % untreated	3183 ± 134	116 ± 17	4.3 ± 0.98	1.2186 ± 0.0012
5 vol % untreated	3348 ± 138	121 ± 12	4.2 ± 0.6	1.2699 ± 0.0010
7 vol % untreated	3571 ± 94	124 ± 15	4.1 ± 0.8	1.3211 ± 0.0010
15 vol % untreated	4550 ± 179	118 ± 12	2.9 ± 0.3	1.5288 ± 0.0017
3 vol % RSCA-treated	3007 ± 103	128 ± 11	6.9 ± 1.3	1.2181 ± 0.0006
3 vol % NRSCA-treated	3123 ± 124	136 ± 9	6.4 ± 1.1	1.220 ± 0.004

in an increase in the tensile strength of the composites.<sup>7</sup>

The ultimate flexural strain was the parameter most strongly influenced by the alumina content increase. For the 3 vol % untreated alumina nanoparticle sample, which as we have already seen, slightly increased the flexural modulus and slightly decreased the ultimate flexural strength, the ultimate flexural strain dropped almost three times compared to the neat epoxy matrix. For alumina contents higher than 3 vol %, the ultimate flexural strain continued to decrease but at a much slower rate. The SCA treatment of the nanoparticles resulted in an increased ultimate flexural strain compared to the untreated nanoparticle sample (Fig. 4). Both SCA-treated nanoparticle samples had almost equal ultimate flexural strains, which were nearly twice higher than the ultimate flexural strain of the untreated nanoparticle composite. This could have been due to the much better nanoparticle dispersion in these two composites as a result of the SCA treatment.<sup>9,10</sup>

### Microhardness of the epoxy/alumina nanoparticle composites

In Figure 5, the alumina content dependence of the universal microhardness for the epoxy/alumina nanoparticle composites is shown. The universal microhardness increased as the alumina content increased for both loads presented in Figure 5. Naturally, the microhardness values obtained for the lower load were higher compared to the values obtained for the higher load. Both SCA-treated nanoparticles samples had universal microhardness values slightly lower than the corresponding untreated nanoparticle sample for both loads (Table II). This was probably due to better filler dispersion and the decreased content of the nanoparticle agglomerates, both of which resulted from the RSCA treatment.

Vicker's microhardness of the epoxy/alumina nanoparticle composites also increased as the alumina content increased, but this dependence was not linear

as was the case with the universal microhardness (Fig. 6). The curve could be separated into two parts. In the first part, from 0 to 7 vol % alumina content, Vicker's microhardness increased and leveled off, keeping a value of 240 MPa. For higher alumina contents (from 7 to 15 vol %), the Vicker's microhardness increased more sharply. The nonlinearity of the first part of this dependence can be explained by the still-existing elasticity (defined by the high percentage of the polymer matrix) of the composite at low alumina contents. The higher the elasticity was, the higher the elastic recovery was, and the Vicker's microhardness values were a little bit heightened, which caused a deviation in the linear dependence already observed for the universal microhardness. The further increase in the Vicker's microhardness at 15 vol % alumina content can be explained both by the increased alumina content and the enhanced nanoparticle agglomeration into composite as the alumina content increased. The Vicker's

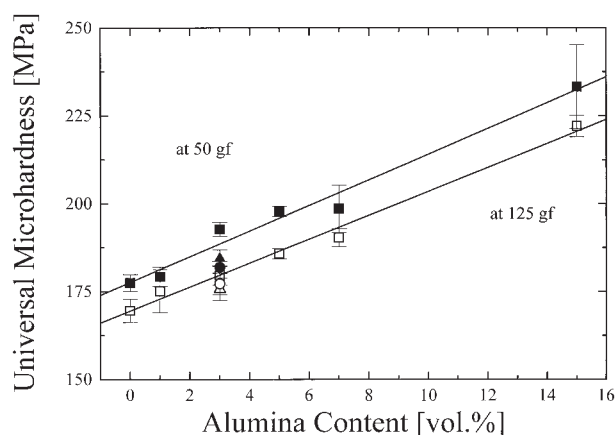


Figure 5 Universal microhardness of the epoxy/alumina nanoparticle composites versus the alumina volume content for two different loads: 50 gf for composites containing (■) untreated and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles and 125 gf for composites containing (■) untreated and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles.

TABLE II  
Microhardness of Epoxy/Alumina Nanoparticle Composites

Sample (vol % Al <sub>2</sub> O <sub>3</sub> )	Universal microhardness at 50 gf (MPa)	Universal microhardness at 125 gf (MPa)	Vicker's microhardness (MPa)
Neat epoxy resin	177 ± 2	169 ± 3	211 ± 8
1 vol % untreated	179 ± 3	175 ± 6	231 ± 4
3 vol % untreated	193 ± 2	180 ± 3	242 ± 3
5 vol % untreated	198 ± 2	186 ± 2	241 ± 1
7 vol % untreated	199 ± 7	190 ± 3	244 ± 2
15 vol % untreated	233 ± 12	222 ± 3	285 ± 2
3 vol % RSCA-treated	182 ± 2	177 ± 3	226 ± 2
3 vol % NRSCA-treated	185 ± 2	176 ± 3	244 ± 9

microhardness of the NRSCA-treated nanoparticle sample was equal to the value obtained for the untreated nanoparticle sample. In contrast, the RSCA-treated nanoparticle sample was softer. This softening coincided with the results for the universal microhardness and also was explained again by the better filler dispersion and the decreased content of the nanoparticle agglomerates, both of which resulted from the RSCA treatment.

#### W of the epoxy/alumina nanoparticle composites

In Figure 7,  $W$  of the epoxy/alumina nanoparticle composites is presented as a function of the alumina content. The lower the alumina content was, the larger the experimental error seemed to be. Perhaps this was related to the unstable wear behavior of the samples where the epoxy matrix dominated. Schwartz and Bahadur<sup>8</sup> observed a minimum  $W$  (at 2 vol % alumina) for alumina-nanoparticle-filled PPS. This minimum coincided with the minimum in the alumina content strength dependence for the same material.<sup>8</sup> For higher alumina volume fractions, the steady state  $W$

increased: the higher the filler content was, the higher  $W$  was.<sup>8</sup>

In our case, a definite minimum in  $W$  alumina content dependence was difficult to be seen. Rather,  $W$  oscillated, and its amplitude wore off as the alumina content increased. Both SCA-treated alumina nanoparticle composites exhibited stable  $W$  values (small error bars). Furthermore, the RSCA-treated alumina nanoparticle composite had a wear value equal to  $W$  of the neat epoxy resin but more steady than it. This steadiness in the wear behavior could have been due to the fact that RSCA-treated nanoparticles were very well dispersed and covalently bonded to the epoxy resin, which thus made the wearing off of the material more difficult.

#### W versus L

$W$  values for all of the composites containing untreated alumina nanoparticles are presented in Figure 8(a) as a function of  $L$ . It appeared that the lowest  $W$

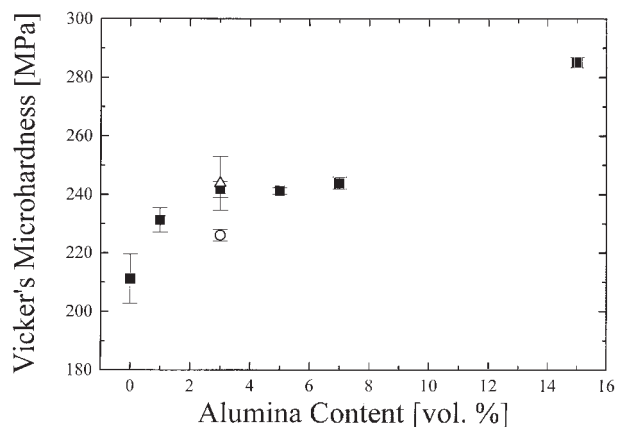


Figure 6 Vicker's microhardness of the epoxy/alumina nanoparticle composites versus the alumina volume content for composites containing (■) untreated alumina nanoparticles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles.

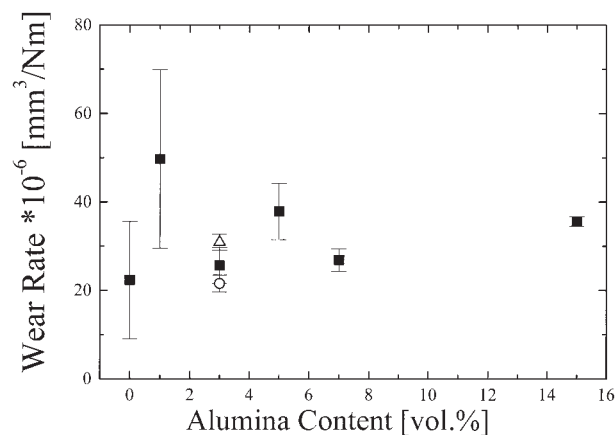
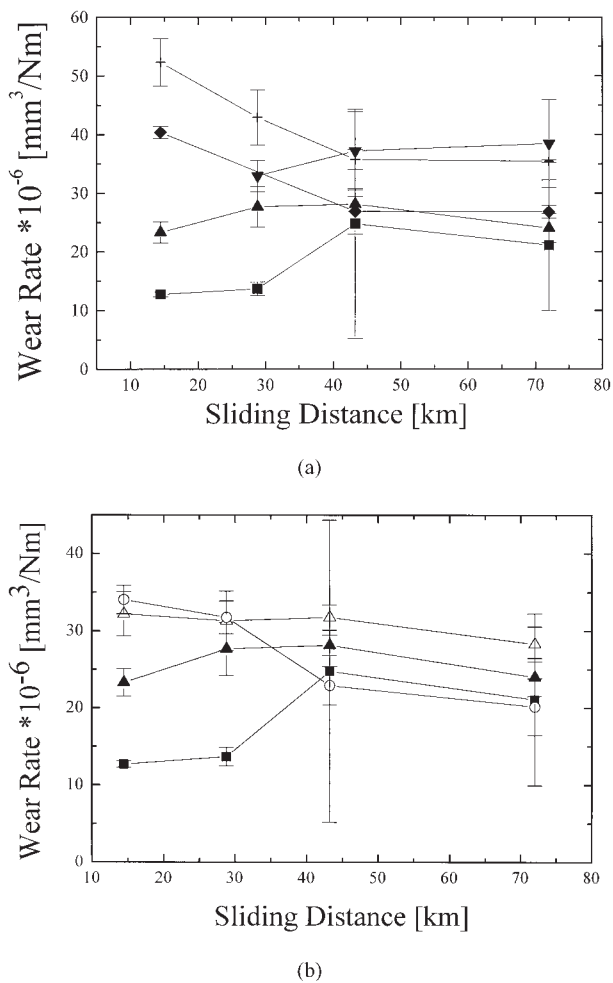


Figure 7 Wear of the epoxy/alumina nanoparticle composites versus the alumina volume content for composites containing (■) untreated alumina nanoparticles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles. (All samples were tested against a counterpart made from 100Cr6 steel with a surface roughness of 0.14  $\mu\text{m}$ , at a  $p$  of 1 MPa and a rotation velocity of 1 m/s for 20 h.)



**Figure 8**  $W$  versus  $L$  for (■) the neat epoxy resin and for composites containing (a) untreated alumina nanoparticles [(▲) 3, (▼) 5, (◆) 7, and (+) 15 vol %] and (b) (▲) 3 vol % untreated particles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles. (The other testing parameters are the same as in Figure 7).

for all of tested  $L$ 's was for the neat epoxy matrix. Its  $W$  slightly increased with  $L$ . Up to a 3 vol % alumina nanoparticle content, the composites showed similar behavior: their  $W$ 's increased with increasing wear time. Above this alumina content,  $W$  decreased and leveled off with increasing  $L$ . This difference, most probably, was due to a changed wear mechanism as the alumina nanoparticle content increased. All samples, for which wear behavior is presented in Figure 8(a), achieved a steady  $W$  value after 12 h of wearing time, that is, after a  $L$  value of 40 km.

It is clearly seen in Figure 8(a) that with increasing alumina nanoparticle content, the steady-state  $W$  increased, which coincided with the observed results by Schwartz and Bahadur<sup>8</sup> for the alumina-nanoparticle-filled PPS system. However, these authors observed only an increase in wear as  $L$  increased; their  $L$  and alumina volume fraction values were almost in the same range as ours.

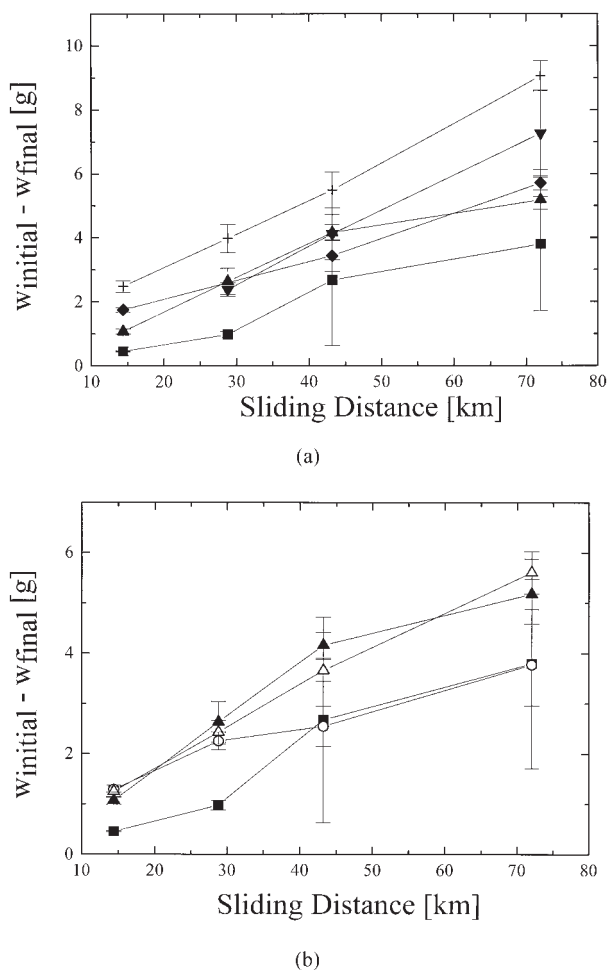
In Figure 8(b), the dependences of the  $W$  values of both samples containing SCA-treated nanoparticles on  $L$  are presented. For comparison, the  $W$  values for the neat epoxy resin and the composite containing 3 vol % untreated alumina nanoparticles are also shown. Although the sample with NRSCA-treated alumina nanoparticles showed a  $W$  that was almost independent of  $L$ , the RSCA-treated nanoparticle sample showed a decreasing  $W$  with  $L$ . This behavior was similar to the dependence observed for samples with a high content of untreated alumina nanoparticles [Fig. 8(a)]. This decrease in  $W$  with  $L$  was most probably related to the creation of a strong transfer film, which did not allow the sample to be worn after a  $L$  of 40 km. After 40 km, the sample with RSCA-treated alumina nanoparticles was the only composite with a  $W$  value equal to that of the neat epoxy matrix; that is, it had the highest wear resistance among the composites studied.

#### Weight loss versus $L$

The  $\Delta m$  values of all samples investigated in this study are presented in Figure 9. The values were averaged for all tested specimens from one composite. For the untreated nanoparticle composites [Fig. 9(a)], an increase in weight loss was observed with increasing  $L$  for all samples. Naturally, the weight loss during wearing increased as the alumina content increased. The neat epoxy matrix had the lowest weight loss for the whole  $L$ . These results were expected, as the densities of the composites increased with increasing alumina content (Table I).

In Figure 9(b), the weight loss dependence on  $L$  for both SCA-treated alumina nanoparticle composites are presented. For the sake of comparison, the results for the neat epoxy resin and the 3 vol % untreated alumina nanoparticle composite are also shown. The composite containing 3 vol % NRSCA-treated alumina nanoparticles had almost the same weight loss as  $L$  increased as the sample containing the same amount of untreated nanoparticles. This fact coincided with those already observed in ref. 6 that NRSCA did not change very much the matrix–filler interaction and that the properties of the respective composite were very close to the properties of the composite containing the same amount of untreated alumina nanoparticles.

Up to a  $L$  of 40 km, the RSCA alumina-treated sample had weight losses lower than the untreated alumina composite and higher than the neat epoxy resin. After this critical  $L$ , it had weight losses equal to the neat epoxy resin, which stayed constant as  $L$  increased. This must have been due to the formation of a strong transfer film, which did not allow further wear of this composite sample. Obviously, the RSCA treatment of alumina nanoparticles influenced the



**Figure 9** Weight loss versus  $L$  for (■) the neat epoxy resin and for composites containing (a) untreated alumina nanoparticles [(▲) 3, (▼) 5, (◆) 7, and (+) 15 vol %] and (b) (▲) 3 vol % untreated particles and (○) RSCA-treated and (△) NRSCA-treated alumina nanoparticles. (The other testing parameters are the same as in Figure 7).

nanoparticles agglomeration and, hence, their dispersion in the epoxy matrix, which in turn, strongly influenced the strength of the transfer film formed between both sliding surfaces: the sample and the counterface.

## CONCLUSIONS

From this study, we drew the following conclusions:

1. The flexural modulus and microhardness (universal and Vicker's) of the untreated alumina nanoparticle/epoxy composites increased with alumina content, whereas the ultimate flexural strength passed through a minimum at 3 vol % alumina content. The ultimate flexural strain drastically decreased as the alumina content increased.

2. The SCA treatment of the alumina nanoparticles resulted in an improvement in the ultimate flexural strength and strain of the final composites. This could have been due both to improved nanoparticle dispersion into the epoxy matrix and to strengthened filler–matrix interactions. Nevertheless, the result was better mechanical performance of the epoxy/alumina nanoparticle composites.
3.  $W$  of the epoxy/untreated alumina nanoparticle slightly increased; however, it became more steady as the alumina content increased.
4. The increase in the alumina content changed the wear mechanism, as up to 5 vol %,  $W$  increased with  $L$ , whereas with a further increase in the alumina content, it decreased. Beyond a  $L$  value of 40 km, all composites containing untreated alumina nanoparticles showed a constant  $W$ .
5. RSCA treatment of the alumina nanoparticles resulted in an improvement in the wear resistance compared to all of the composites examined in this study. Furthermore, this treatment changed the wear mechanism as  $W$  dependence on  $L$  for this sample had the same profile as those of composites with much higher alumina contents. In contrast, NRSCA treatment of the alumina nanoparticles did not improve the wear behavior of the respective composite, and this sample behaved almost like the sample where the alumina had not been preliminary treated.
6. Treating alumina nanoparticles with RSCA allowed us to improve the nanoparticle dispersion in the composite and, hence, to improve to some extent its mechanical and wear performance.

SCA treatment was part of a procedure for polymer grafting onto alumina nanoparticles as a way to obtain epoxy/alumina nanocomposites. This part of our investigations is described elsewhere.<sup>11</sup>

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