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FABRICATION OF NANOCOMPOSITES WITH SILICA NANOPARTICLES

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1. Introduction

Nanocomposites exhibit a combination of exceptional properties which usually cannot be achieved in standard composites. Some of the most studied systems are nanocomposite thermosets, which are filled with various forms of nano-carbon (carbon nanotubes, graphene), silica and alumina nanopowders, and other nanofillers. The influence of the technological methods used to produce nanocomposites was extensively discussed, only as an example [1]; more than 100 recipes can be found in the literature. Essentially, the main problem is to disperse uniformly the nanofillers, as mentioned [2] and [3]. Some considerations on the fabrication technology of nanocomposites filled with multi-wall carbon nanotubes (MWNT), alumina (Al_2O_3) and silica (SiO_2) nanoparticles were presented elsewhere, [4, 5]. Improvements of the manufacturing process were established in order to produce uniformly distributed fillers in the epoxy matrix. On the other hand it is important to use functionalized nanoparticles that are more compatible with the matrix and easier to mix.

2. Technologies of fabrication

For dispersing the fillers in the epoxy resin special equipment is needed. A shear mixer Thinky ARE-250 (Japan) with maximum rotation speed of 2000 rpm was used for mechanical mixing. A high energy sonicator, Sonics VCX-750 (US), having a generator with 750 W output, a 20 kHz convertor and a temperature controller, was used to fragment the conglomerated nanoparticles. A programmable vacuum oven Memmert VO 400 (UK) was used for curing. The final mixture of resin, nanofillers and hardener was poured in a silicon mould. For each batch 14 specimens were produced.

Several manufacturing procedures were explored such to improve the dispersion and avoid the formation of air bubbles in the resin. The sample preparation steps that led to the best results are as follows: mixing the resin with the nanoparticles with the shear mixer for 10 minutes at a speed of 1500 rpm; the resulting solution R+NP (R = resin, NP = nanoparticles) is sonicated for 2 hours. In Method M1, R+NP was put under a vacuum of 30 mbar for 2 hours at room temperature for degassing. In Method M2 this step was omitted. The hardener H (H = hardener) was added to R+NP after this step. The R+NP+H solution was mixed by hand for about 2 minutes, and poured in the silicon mold.

3. Materials used for fabrication

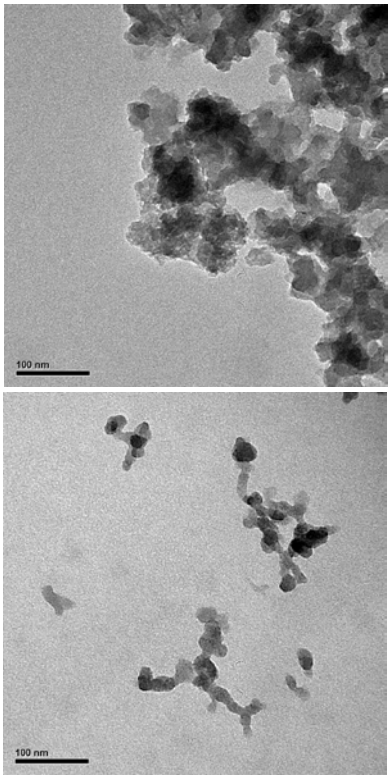
Various types of resin were used. These include Neukadur EP 986 produced by Altropol Kunststoff GmbH, Germany, which was used together with the hardener Neukadur HN 242 (with a pot time of 25 minutes). Another system considered was the same epoxy with Neukadur HN 246 as hardener (with a pot time of 240 minutes); the second curing agent gives more time for mixing with the resin.

Furthermore, two epoxy systems produced by BTO Epoxy and named System 2 (S2) and System 5 (S5) were considered. S2 uses a resin notated IR 77.31 and a slow hardener IH 77.15 (with a pot life of 110 minutes at 25 °C). S5 is under development and has a low viscosity of 100-300 mPas at 25 °C and a pot life of 80 minutes at 25 °C.

Both unfunctionalized and functionalized silica nanopowders were used. The unfunctionalized silica was produced by Sigma Aldrich and was supposed to have particles of 5-15 nm diameter and purity 99.5 wt% with some traces of metal.

The functionalized nanopowder was obtained by coupling of azidophenylsilanes to nanosilica with a specific surface area of 175-225 m^2 (determined using the Brunauer–Emmett–Teller (BET) theory). This is a fumed silica, with a fractal structure, consisting of particles of approximately 20-80 nm which are agglomerated and intergrown to form bigger aggregates.

Fig. 1 TEM images of the unfunctionalized (but clustered) nanoparticles (a) and the functionalized fumed silica nanopowder (b) are presented.



(a)

(b)

Fig. 1. Sigma Aldrich nanopowder: (a) clustered unfunctionalized silica of nominal particle size 5-15 nm; (b) functionalized fumed silica of 20-80 nm.

4. Mechanical testing of nanocomposites

Uniaxial traction testing of the specimens was performed using a Zwick/Roell testing machine, model Z010, with a maximum force of 10 kN. For each batch specimens were tested to determine the modulus of elasticity, the ultimate tensile strength, and the elongation at failure. Tests were carried on nanocomposites with unfunctionalized and functionalized silica nanopowder. The testing speed was 1.5 mm/min which corresponds to an initial strain rate of approximately 10^{-3} s^{-1}.

Weight percentage wt% of silica powder was 0.1, 0.3, 0.5, 1.0, and 3.0 in different batches. The mechanical properties for samples prepared using method M1, for which the R+NP mixture was kept under a vacuum of 30 mbar for 2 hours at room temperature for degassing, and for samples prepared using method M2 (degassing was omitted as it was found that in some cases it generates additional gas bubbles in the specimens) were obtained.

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References

[1] Zhou, Y., Pervin, F., Lewis, L., Jeelani, S., Fabrication and characterization of carbon/epoxy composites mixed with multi-walled carbon nanotubes, Mater. Sci. Eng. A, 475, 2008, pp. 157-165.

[2] Gkikas, G., Barkoula, N.-M., Paipetis, A.S., Effect of dispersion conditions on the thermo-mechanical and toughness properties of multi walled carbon nanotubes-reinforced epoxy, Compos. Part B-Eng., 43, 2012, pp. 2697-2705.

[3] Montazeri, A., Chitsazzadeh, M., Effect of sonication parameters on the mechanical properties of multi-walled carbon nanotube/epoxy composites, Mater. Design, 56, 2014, pp. 500-508.

[4] Cosmoiu, I., Apostol, D.A., Picu, C.R., Constantinescu, D.M., Sandu, M., Manufacturing and testing of nanocomposites with carbon nanotubes and nanoparticles, UPB Sci. Bull., 77, 2015a, pp. 107–119.

[5] Cosmoiu, I., Apostol, D.A. Constantinescu, D.M., Picu, C.R., Sandu, M., 2015b. Advances on the Manufacturing Process of Nanocomposites with MWNT and Nanopowders, Appl. Mech. Mater., 760, 2015b, pp. 281-286.