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ScienceDirect

Procedia Structural Integrity 5 (2017) 647–652

Structural Integrity Procedia

www.elsevier.com/locate/procedia

2nd International Conference on Structural Integrity, ICSI 2017, 4-7 September 2017, Funchal, Madeira, Portugal

Mechanical properties of epoxy nanocomposites reinforced with functionalized silica nanoparticles

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Abstract

Functionalization of silica nanoparticles is needed in order to improve the mechanical performance of epoxy nanocomposites. Previous experience showed that direct dispersion of commercially available silica nanofillers does not improve significantly the mechanical properties of resulting nanocomposites. Fumed silica nanoparticles from Sigma Aldrich (175-225 nm BET) were functionalized carrying exactly 0.28 mmol of phenylazide per 1g of dry particles. During drying they aggregate and their dispersion in epoxy becomes challenging. We use sonication to break agglomerates and obtain adequate dispersion. Different methods of fabrication are presented along with comments on the resulting tensile composite behavior.

Peer-review under responsibility of the Scientific Committee of ICSI 2017

Keywords: silica nanoparticles; functionalization; sonication; tensile mechanical properties.

1. Introduction

Polymer nanocomposites emerged as important structural materials, competing with neat polymers and classical

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2452-3216
Peer-review under responsibility of the Scientific Committee of ICSI 2017
10.1016/j.prostr.2017.07.034

composites. These materials 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 by Zhou et al. (2008); more than 100 recipes can be found in the literature. Essentially, the main problem is to disperse uniformly the nanofillers, as mentioned by Gkikas (2012) and Montazeri (2014). 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 by Cosmoiu et al. (2015a, 2015b). Improvements of the manufacturing process were established in order to produce uniformly distributed fillers in the epoxy matrix. On the other hand (Cui et al. (2013)), it is important to use functionalized nanoparticles that are more compatible with the matrix and hence easier to mix.

It is generally desired that these nanocomposites have superior strength, ductility and toughness. In Zandiatashbar et al. (2012) and Zhang et al. (2007), graphene platelets (GPL) and multi-wall carbon nanotubes (MWNT) epoxy composites with various weight fractions (0 to 0.5 wt%) were prepared, and were tested under monotonic, cyclic (fatigue) and creep conditions. It was observed that the addition of GPL and MWNT has a marginal effect on the stress-strain curve at all strain rates investigated. However, GPL reduces the creep rate at elevated temperatures, especially in the transient creep regime. Both MWNT and GPL led to a reduction of crack growth rate under fatigue conditions. The fracture toughness of such nanocomposites can be also improved, as shown by Ayatollahi et al. (2012) and Picu et al. (2013). As mentioned above and broadly discussed in the literature, the selection of the filling fraction, filler type and filler functionalization has a significant influence on the mechanical properties of the composite.

In this work we study the influence of silica nanopowders on the mechanical static properties of epoxy-based nanocomposites.

2. Method 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. During sonication, the temperature was maintained at 60 °C by using a circulating cold water system surrounding the container in which the mixture was sonicated; a temperature gauge was placed inside the mixture for control purposes.
- 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.
- The thermal cycle was carried on as prescribed by the producer of the epoxy resin.

3. Materials used for specimen 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, degassing and pouring in the mould and was hence preferred.