



Microfluidization, time-effective and solvent free processing of nanoparticle containing thermosetting matrix resin suspensions for producing composites with enhanced thermal properties



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ARTICLE INFO

Article history:

Received 3 October 2016

Received in revised form 2 November 2016

Accepted 4 November 2016

Available online 10 November 2016

Keywords:

Multi walled carbon nanotubes (MWCNTs)

Microfluidization

Composite materials

DSC (Dynamic Scanning Calorimetry)

DMA (Thermomechanical Analysis)

ABSTRACT

Dispersion of low content (0.1 wt.%) of multi walled carbon nanotubes (MWCNTs) in an epoxy resin through microfluidization was brought under spotlight as a case study. For the sake of performance comparison, epoxy resin suspension with the same MWCNT content was also produced through sonication, and the energy consumed to disperse MWCNTs in the epoxy resin remained the same for the both processes. At the equal energy consumption level, preparing the resin suspension through microfluidization was determined to take approximately 4.5 min, which is about 9 times lower than it takes for the same suspension to be prepared through sonication. Following cure of the prepared suspensions, thermal and thermo-mechanical properties of the resulting composites were systematically investigated. As a result, glass transition temperature (T_g) of the neat epoxy and its composites produced by sonication and microfluidization were determined to be 78 ± 3 , 83 ± 5 and 88 ± 4 °C, respectively. Moreover, thermal expansion coefficient (CTE) of the epoxy resin was reduced by 7% when it was blended with MWCNTs through microfluidization, while no significant change was observed in the corresponding CTE value when through sonication. Thermomechanical testing findings implied about 5% and 10% improvement in storage and loss modulus values of the composites produced by microfluidization over by sonication. Transmission electron microscopy (TEM) examination showed that MWCNTs were more homogeneously and individually dispersed in the epoxy resin by retaining their initial aspect ratio through microfluidization. Scanning electron microscopy (SEM) examination revealed that composite fracture surface morphology significantly varied, depending to large extent on the type of the process used. Based on the results obtained, it was proposed that microfluidization might pave the way for cost-and-time effective solvent-free processing of nanoparticle modified thermosetting resin suspensions, especially including those that could be promisingly used as matrix constituent for manufacturing fabric reinforced composites with multi-functional properties.

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1. Introduction

Thermosetting polymers have aroused great interest among the scientific community over the last three decades due to their relatively low cost and ease of processing. They are network-forming polymers and include epoxy, phenolic, unsaturated polyester, polyurethane, cyanate ester, bismaleimide, acrylate and many others. They have been in majority used as matrix materials for the manufacture of fiber reinforced composites. However, thermosetting polymers exhibit poor damage tolerance combined with low strength and stiffness relative to other engineering materials, such as metals [1–8]. Most thermosetting polymers therefore incorporate various types of particulate fillers to reduce cost, to modify thermal and mechanical properties, or to reduce shrinkage during cure. Nanotechnology has very recently made it possible to look at thermosetting polymers from a different perspective, when it comes to creating new application areas in composite manufacturing industry. With this respect, the use of nanoparticles of high strength and modulus as filler constituent for thermosetting polymers has been believed to pave the path to producing novel multi-functional composites of extraordinary versatility [3–7]. As a very recent example, carbon nanotubes (CNTs) have emerged as one of the most promising nanofiller candidates and have been therefore utilized for various types of polymers to manufacture electrically conductive composites with at least retained or improved thermal and mechanical properties [8,9]. In fact, a CNT is a tube-shaped material, made of carbon, having a diameter as thin as a few nanometers yet exhibiting a length as long as hundreds of microns. They are unique because the bonding between the atoms is very strong and they can have huge aspect ratios [4]. CNTs are usually categorized as either single-walled (SWCNT) which is just like a regular straw composed of only one wall, or multi-walled nanotubes (MWCNTs), a collection of nested tubes of continuously increasing diameters, held together by interatomic forces. Unlike the use of traditional micro-sized filler particles, such as carbon black (CB), that usually end up compromising among polymer mechanical properties, leading to high strength combined with low toughness, the use of CNTs is expected to provide a synchronized increase in polymer composite mechanical and thermal properties [10]. However, the studies in the literature reported up to date showed that CNT modified polymer composites demonstrated mechanical strength and thermal properties very far below the theoretical predictions [7–9]. Specifically speaking, it is required that the two major problems be solved prior to putting theoretical predictions into practical use. The former is weak interfacial bonding between CNTs and the surrounding polymer matrix, a struggle arising from very inert surfaces of CNTs, while the latter is a strong tendency of CNTs to form agglomerates because of their huge surface area and aspect ratio [3–8]. Those problems are not just only limited to the use of CNTs, but also to the use of most of the fiber-like fillers at nanoscale, too. To alleviate the first challenge, two different strategies are adopted, one of which is the covalent functionalization, while the other is the non-covalent functionalization. The covalent functionalization has been most commonly used to graft chemical functional groups that are compatible with the matrix resin onto CNT surfaces to promote the interfacial interactions in between [2,4]. Strong acids, such as nitric and sulfuric, or their mixture at different specified weight ratios are used to oxidize CNT surface followed by imparting the desired functional groups onto them to ease their processing when blended with polymers. Nevertheless, incorporation of functional groups into a conjugated π -electron system, as in the case of such graphite structures as CNTs, causes formation of sp^3 -carbons which carry the functional groups. Thus, the chemical modification interrupts the conjugation, hence inducing a shape distortion in the graphitic layer structure, which is detrimental to final mechanical, thermal, and electrical properties of CNTs [6]. That stands for the reason why application of any covalent functionalization reported in the literature up to date brought about an uncontrolled length reduction in CNTs along with significant surface flaws in their structure [10]. To overcome these drawbacks, the non-covalent functionalization of CNTs has been lately adopted [3]. It is believed to help better preserve the CNT conjugated double bonds than does the covalent functionalization. The non-covalent functionalization has been several times shown to result in more stable and concentrated dispersions of CNTs both in organic and in aqueous solvents than it would be possible with the covalent functionalization [2–6]. Once agglomerated CNTs are properly disentangled and the strong van der Waals forces among the adjacent bunches are gotten rid of with application of high shear, CNT dispersions could be achieved as a means of polar and π -stacking interaction of CNTs in company of a suitable solvent, with their structures mainly intact remaining [2,3,6]. Another non-covalent functionalization approach to favoring CNT dispersion in organic solvents is to cover the CNT surface with a thin layer of a dispersant phase, a molecule characterized by a high affinity towards CNT sidewalls, which is also to be predominantly soluble in the solvent of choice [3,4]. Both small molecules and high weight polymers have been utilized as dispersant phases for this purpose.

As for the second challenge, the huge surface area of CNTs leads to strong attractive forces to occur among the bunch of CNTs, which ends up with formation of agglomerated clusters. The extent of agglomeration is highly critical to the mechanical properties of resulting CNT modified polymer composites [7–10]. In the literature, a number of different techniques, including high speed mechanical stirring, magnetic stirring, reflux, shear mixing, 3-roll milling, or, most commonly, ultrasonication, either mild sonication in a water bath, or high-power sonication using a metal tip have been employed for proper dispersion of CNTs in thermosetting polymers [1–13]. As a result, high-power sonication has been cited as the most promising among all [5–8]. During high-power sonication, a pulsed ultrasound with certain amplitude is applied in an effort to divide large agglomerates into relatively small or individual CNTs. However, the magnitude of applied vibration energy is limited to distance from the sonicator tip. Sonication is therefore claimed to shorten the length of CNTs uncontrollably by applying a high local energy input nearby the sonicator tip, which might be detrimental to some extent to the thermal, electrical, and mechanical characteristics of CNTs [4,6,8]. In our early studies, 3-roll milling turned out to be more promising

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