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Material properties

Influence of carbon nanotubes on the rheology and dynamic mechanical properties of polyamide-12 for laser sintering

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ABSTRACT

The rheological behaviour of polymer nanocomposites is very important for polymer processing and understanding the structure-properties relationship. In this paper, the rheological properties of a polyamide 12 (PA12) - carbon nanotube (CNT) nanocomposite for laser sintering were studied. Compared to neat PA12, the presence of CNTs resulted in higher storage modulus (G'), loss modulus (G'') and viscosity (η). With an increase in temperature, viscosity showed an unusual increase for both PA12 and the PA12-CNT nanocomposites, which is likely to result from incomplete melting of powder particles. Dynamic mechanical analysis was carried out to examine the effect the CNTs were having on the laser sintered parts. The laser sintered PA12-CNT nanocomposite had an increased elastic modulus compared to that of neat PA12. The CNT and polymer matrix interaction hindered the chain motions, which resulted in higher loss modulus and decreased the thermal expansion coefficient.

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1. Introduction

Additive Manufacturing (AM) is a group of technologies which are applied to produce end-use products directly from 3D model data. Due to the greater flexibility with respect to product design and manufacture compared to conventional manufacturing methods, AM has been attracting more and more attention from both academic and industrial communities in recent years. One of the most well-established additive manufacturing technologies, laser sintering (LS), is a powder-based process which uses a laser as a heat source to fuse polymer powder into three dimensional parts [1].

Several polymers, such as polyamide (PA), polypropylene (PP), polystyrene (PS), polycarbonate (PC) and poly(ether-

ether-ketone) (PEEK) have been supplied as laser sintering materials [2], with PA being the most common polymeric material used, mainly due to its ease of processing [3]. However, current laser sintering materials cannot completely meet the needs of all products [4]. To meet the requirements for different applications, polymer/filler composites have been investigated to enhance the properties of laser sintered parts. With a small quantity of nano-filler, polymer nanocomposites have shown a significant improvement over the base polymer's mechanical, thermal and electrical properties [5]. Various reinforcing nanofillers combined with base polymers have been investigated for laser sintering [6–8]. In the work carried out by Goodridge et al. [6], a 3 wt% PA12/carbon nanofibre composite was prepared by melt mixing and cryogenic milling. The dynamic mechanical properties of the nanocomposite were 22% higher than those of the base PA12 laser sintered parts. However, irregular powder morphology weakened the

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reinforcement effect from the nanofillers. In the research performed by Athreya et al. [7], PA12/carbon black (4 wt%) nanocomposite powder was prepared and laser sintered. The PA12/carbon black parts showed improved electrical conductivity but decreased flexural modulus compared to neat PA12 parts. The reduction in flexural modulus was due to agglomeration of the carbon black particles in the laser-sintered part. Another study showed that CNT agglomerates caused a reduction in elongation at break for PA12-CNT laser sintered parts [8]. It is, therefore, essential for laser sintering of polymer nanocomposites that both the morphology of the powder particles and the dispersion of the nanoparticles within the polymer matrix are optimised.

To address this, in our previous work [9], individual PA12 powder particles were coated with carbon nanotubes (CNTs) in order to produce well-dispersed PA12-CNT nanocomposite powder with spherical morphology and suitable particle size for processing by laser sintering. Results showed that the PA12-CNT laser sintered parts had enhanced flexural properties (13.0% higher) and impact properties (123.9% higher). This was achieved without sacrificing elongation at break, which was in contrast to previous studies in the literature which showed some decrease in strength or decrease in elongation at break [7,8].

One of the aims of the study reported in this paper was to study the rheological behaviour of the nanocomposites produced by our coating method; rheology is a very important factor in polymer processing which has rarely been investigated for laser sintering nanocomposites.

In the laser sintering process, polymers melt and flow. Understanding the motion of the polymer composites liquid is very important for analysis of the processing operation and optimisation, as well as investigating the structure-property relationship of the nanocomposites. From studies carried out on conventional polymer nanocomposites, rheological properties have been shown to influence the fabrication and processing (residual stress, void content, etc.) of such materials [10,11]. Jin and co-workers [12] found that the amount of nanoclay loading had a significant influence on the rheology of a polyol/nanoclay mixture when processing polyurethane (PU) nanocomposites. It was found that to achieve good dispersion of the nanoclay with critical viscosity, 3 wt% was the optimum nanoclay loading. This finding provided useful information for the preparation of PU/nanoclay composites in their study, which could also be a helpful guideline for polymer nanocomposite preparation for laser sintering. Several studies have shown that the composite phase morphology and the interfacial interactions between polymer chains and nanofiller also have a direct correlation with the rheological properties of nanocomposites [13–15]. Lim and Park [14] reported these effects on the rheological behaviour of different polymer/nanoclay composites by oscillatory frequency and shearing rheological tests. The results indicated the existence of strong interactions between polymer matrix and nanoclay, and the formation of some particle network structure and randomly oriented silicate layers. These rheological studies with conventional polymer nanocomposites could also be carried out on polymer nanocomposites for laser sintering to offer a

fundamental understanding of the processability of these materials, as well as the structure-properties relationship in the nanocomposites.

In this work, a comprehensive rheological investigation of a polymer nanocomposite for laser sintering was carried out to examine the influence of the nanofiller on the rheology properties of the polymer matrix. Due to their excellent mechanical, thermal and electrical properties, carbon nanotubes are considered to be ideal reinforcing nanofillers for polymers [16] and were, therefore, chosen as the nanofiller. PA12 was chosen as the polymer matrix in this study due to its established use for laser sintering. In addition to rheological experiments, to examine the changes in mechanical properties of laser sintered parts within a temperature range, dynamic mechanical analysis was applied. The effect of the CNTs on the modulus and damping of the laser sintered PA12 samples was analysed.

2. Experimental

2.1. Materials

The polyamide 12 (PA12) powder used in this study was obtained from EOS GmbH (trade name 'PA2200'), and is a commonly used laser sintering polyamide material. Multi-walled carbon nanotubes, supplied by NanoAmor Materials Inc., had an average diameter of 10 nm and length of 1.5 μm (according to the manufacturer's datasheet). PA12-CNT nanocomposites were prepared by coating the CNTs on the surface of the PA12 particles using a patented method [17]. With this method, the final PA12-CNT nanocomposites powders have been shown to exhibit near-spherical morphology [9]. The nanocomposites were produced with two different CNT contents, 0.1 wt% (referred to herewith as PA12-CNT0.1) and 0.2 wt% (referred to herewith as PA12-CNT0.2).

2.2. Melt rheology

Dynamic oscillatory shear tests were conducted on a RADII instrument (Rheometrics[®], Inc) to measure the melt rheology. Tests were performed using parallel plate geometry with plate diameter of 25 mm. Neat PA12 (referred to herewith as PA12), PA12-CNT0.1 and PA12-CNT0.2 powders, which were dried in a vacuum oven at 80 °C for 12 hours, were formed into sheets by hot pressing at 210 °C. Sample discs with a diameter of 25 mm and thickness of 1 mm were prepared.

In a dynamic oscillatory shear test, a time dependent strain is applied during the test, and the resulting shear stress is:

$$\sigma(t) = \gamma_0 [G' \sin(\omega t) + G'' \cos(\omega t)]$$

where γ_0 is the strain amplitude, ω is the test frequency, t is the time, G' is the in-phase, elastic or storage modulus and G'' is the out-of-phase, viscous, or loss modulus, which is a measure of the energy dissipated per cycle. To maintain a linear response, a strain amplitude of 10% was used to obtain reasonable signal intensities at elevated temperature or low frequency. The testing temperature range was

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