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Carbon nanotubes prevent the coagulation at high shear rates of aqueous suspensions of equiaxed ceramic nanoparticles

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Abstract

Equiaxed ceramic nanoparticles and their mixtures are expected to exhibit shear-thinning behaviour when dispersed colloidally in aqueous media, whereas shear-thickening is the expectation for large aspect ratio phases such as, for example, carbon nanotubes (CNTs). Here, contrary experimental evidence is presented demonstrating the occurrence of severe coagulation at high shear rates in colloidally stable, semi-concentrated, aqueous suspensions of equiaxed SiC nanoparticles (major phase) mixed with equiaxed $Y_3Al_5O_{12}$ nanoparticles (liquid-phase sintering additive), and how CNT addition prevents this coagulation if sufficient sonication is applied. It is also shown that although shear-thinning is the natural behaviour of the ceramic suspension up to moderate shear rates, coagulation is eventually a phenomenon inherent to the aqueous colloidal processing of these suspensions, with the critical shear rate for coagulation increasing and the rheopexy decreasing the better is the initial dispersion state achieved with the sonication. It is also shown that the critical shear rate for coagulation depends on the exact condition of shear rate increase, and that the re-sheared suspensions coagulate more significantly and at lower shear rates than the fresh suspensions. The mechanisms by which this coagulation occurs and is impeded by the CNTs are discussed, together with broader implications of these phenomena for the environmentally friendly processing of nanostructured ceramics and ceramic composites.

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1. Introduction

The fields of nanostructured ceramics and ceramic matrix nanocomposites (CMNCs) reinforced with carbon nanotubes (CNTs) are today at the forefront of research in materials science and related disciplines. By virtue of their tiny grain size at the nanoscale, nanoceramics exhibit a set of properties that have no parallel in the world of conventional micrometre and submicrometre ceramics. Some of these properties are crucial in many structural applications (for example, increased hardness, resistance to wear, and strength, to name just a few).^{1–6} Unfortunately, the price to pay for this nanoscale grain refinement is extreme brittleness,^{1,5} the bane of nanoceramics in many structural and functional applications. It is not surprising therefore that toughening of nanoceramics is the subject of

intense research, as success in this direction would have broad technological implications. In this regard, CNTs, with their exceptional set of properties and the unprecedented hierarchical grain-boundary structures of their composites with ceramics which have the form of a three-dimensional network of two-dimensional mats made up of random one-dimensional CNT bundles segregated over the surface of the ceramic grains,⁷ represent the ideal second-phase prototype for the reinforcement of the otherwise brittle nanoceramics, so that CNT-reinforced CMNCs have also captured the attention of the ceramics community.^{8–10}

The fabrication of nanoceramics and ceramic nanocomposites is an approachable challenge today thanks to the development of rapid densification techniques such as sparkplasma sintering,¹¹ microwave sintering,¹² and the more recent flash sintering.¹³ A processing aspect that continues to be crucial is the correct dispersion of the nanoparticles prior to sintering because they tend to agglomerate due to van der Waals forces. This becomes particularly important in the case of nanocomposites with various ceramic phases because the

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different nanoparticles may undergo highly undesirable heteroaggregation, and critical in the case of CNT-reinforced CMNCs because, due to their high aspect ratio, the CNTs are prone to intense entangling, and it is extremely difficult, if not impossible, to de-agglomerate them mechanically (for example by conventional ball-milling) without damaging them.¹⁰ Unsurprisingly, colloidal processing has become the most popular method to tackle the general problem of the dispersion of nanoscale particles. In this regard, the conventional wisdom in the field is that the typical equiaxed nanoparticles can be better and more easily dispersed in water than the mixtures of these same nanoparticles with CNTs.

In this study, however, we provide evidence to the contrary, demonstrating the unexpected severe coagulation at high shear rates of suspensions of equiaxed ceramic nanoparticles and its prevention with CNT addition. This was discovered during the environmentally friendly preparation by aqueous colloidal processing of mixtures of nano-SiC plus nano- $Y_3Al_5O_{12}$ (henceforth, abbreviated simply as YAG) without and with CNTs. Besides reporting these two unprecedented findings, we shall also discuss the possible mechanisms by which these phenomena occur, and the implications for wet-shaping suspensions of ceramic nanoparticles without and with CNTs.

2. Experimental procedure

The starting materials were commercially available multiwall CNTs and nanopowders of β-SiC and YAG, all three supplied by Nanostructured and Amorphous Materials Inc. (USA). According to the manufacturer's specifications, the SiC and YAG nanopowders comprise equiaxed nanoparticles with average sizes of 45-55 nm and 40 nm, respectively, and the CNTs have lengths of $0.5-2 \,\mu\text{m}$ and diameters of $10-20 \,\text{nm}$, and have been functionalized with OH⁻ surface groups. As can be seen in Fig. 1, the independent characterization performed in the present work by transmission electron microscopy (TEM; Tecnai G2-20 Twin, Eindhoven, the Netherlands) essentially supports these morphological features. The specific surface areas determined by N2-adsorption isotherms (Monosorb Surface Area Analyser, MS-13, Quantachrome Corporation, Boynton Beach, USA) were 31, 27, and 161 m² g⁻¹ for the nano-SiC, nano-YAG, and CNTs, respectively.

Firstly, the colloidal stability of the nano-SiC, nano-YAG, and CNTs was individually investigated on dilute, single-phase suspensions prepared at a solid content of 10^{-1} g/l in a solution of deionized water with KCl 10^{-2} M as inert electrolyte mixed in the volume ratio 9:1 with ethanol, and subsequently equilibrated for 30 min. This study involved the systematic measurement of the zeta potential, first as a function of deflocculant content at natural pH. The pH was varied in the acidic–basic range by using 10^{-1} M HCl or KOH solutions, respectively, and was measured with a conventional pH-metre (Titrino DMS 716, Metrohm, Switzerland). The deflocculation was done using commercially available polyelectrolytes, in particular, a synthetic polyelectrolyte of unknown composition (PKV; Produkt KV5088, Zschimmer-Schwarz, Germany) for the nano-SiC and



Fig. 1. Representative TEM micrographs of the (A) SiC nanoparticles, (B) YAG nanoparticles, and (C) CNTs in the as-received powders. The images were acquired in bright-field mode at 200 kV accelerating potential. Planar defects are seen in some of the SiC nanoparticles, which is a common feature in β -SiC.

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