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Influence of nanoparticle surface treatment on particle dispersion and interfacial adhesion in low-density polyethylene/aluminium oxide nanocomposites

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ABSTRACT

The effect of silsesquioxane coating of aluminium oxide nanoparticles on their dispersion and on the interfacial strength between nanoparticles and polymer matrix in low-density polyethylene composites was studied. The surface chemistry of the nanoparticles was tailored from hydroxyl groups to alkyl groups with different lengths by reacting methyltrimethoxysilane (C1), octyltriethoxysilane (C8) or octadecyltrimethoxysilane (C18) with aluminium oxide nanoparticles. The core-shell structure of the coated nanoparticles was assessed by transmission electron microscopy, infrared spectroscopy and thermogravimetry. The inter-particle distance of the nanocomposite based on C8-coated nanoparticles showed only a small deviation from the ideal value, indicating a very good particle dispersion in the polymer. The interfacial adhesion between nanoparticles and matrix was determined by stretching nanocomposite specimens in a tensile testing machine to strains well beyond the yield point. A drop in the stress-strain curve indicated the onset of cavitation and necking in the nanocomposites. Samples stretched to different strain levels were studied by scanning electron microscopy and the cavitation was found to be confined to particle interfaces. The composite based on C18-coated nanoparticles showed the highest strain at cavitation/necking suggesting a high interfacial adhesion between nanoparticles and polymer.

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1. Introduction

The metal conductor in extruded high-voltage direct current (HVDC) cables is surrounded by a layered structure consisting of semi-conductive layers (usually two layers placed on each side of an insulating layer; consisting of a polyolefin filled with carbon black), a thick highly insulating layer consisting of crosslinked polyethylene, a layer of water-absorbing material, an outer thin metal barrier layer that makes the system tight and an outer jacket [1]. Extruded HVDC cables are currently commercially used

for voltages up to 320 kV [1,2]. Recently, however, ABB developed a 525 kV DC cable system with a power rating range up to 2.6 GW intended for both submarine and underground applications [3]. This system is based on extruded HVDC cables with a structure as described above. However, in order to meet the forecast global energy/power demands, a voltage of 1 MV for the cables has to be reached by 2030 [4]. This requires a significant decrease in the electrical conductivity of the material used in the insulating layer. At least two different strategies are currently being used to increase the insulating performance of polyethylene. One strategy is to further refine the present material by the elimination of μm -sized impurities and by reducing the concentration of mobile ionic and polar

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species, which originate from the synthesis, processing and vulcanization [2]. Another strategy is to incorporate dispersed metal oxide nanoparticles (e.g. Al_2O_3 , MgO and ZnO) at low concentrations (typically a few mass per cent) in the polymer. These nanoparticles act as traps for the charges and ionic/polar species. This nanocomposite strategy appears promising and early experiments indicate a ten-fold decrease in the electrical conductivity in high electrical fields [5].

Different methods have been used to prepare polymer nanocomposites, including melt mixing [6], solution mixing [7] and *in-situ* polymerisation [8]. The incompatibility between nanoparticles and polymer matrix is usually a challenge and it may lead to particle agglomeration and void formation in the composites, which in some cases have detrimental effects on the performance of the materials [9]. Significant efforts have been devoted to the development of methods to modify the particle surfaces by either physical or chemical methods and to increase the compatibility between the inorganic particles and the polymer matrix. This includes encapsulating the particles in surfactant [10,11], and grafting polymer chains [7,12–15] and silanes [16–21] onto the particles. The use of surfactants would incorporate mobile ionic or polar species in the nanocomposite, and this is not a feasible solution for HVDC cable insulations since ionic/polar species increase the electrical conductivity of the insulation. Polymer-grafted nanoparticles have been prepared by a surface-initiated polymerisation method such as atom transfer radical polymerisation [7,14], by reversible addition fragmentation transfer polymerisation [9,13] and by grafting polymers onto nanoparticles [15]. These methods usually require several reaction steps and the use of large quantities of organic solvents, and this may limit their industrial applicability. The advantage of using silane chemistry is that the condensation chemistry is based on water or alcohols, that a wide range of different silanes is commercially available and that the process can be readily up-scaled [22].

The particle dispersion in the composites is one of the most important factors affecting the practical compatibility between the particles and the polymer. The presence of large agglomerates in the insulating material may for several reasons be detrimental for a HVDC cable [2]. Scanning and transmission electron microscopy are commonly used to assess the particle dispersion, but the material volume examined by these methods is usually small which means that a larger number of micrographs are required and that the images need to be interpreted by a reliable method for identification of the nanoparticles and analysed by a statistically adequate method. Several quantitative methods applicable to micrographs have been used to approach this problem [23,24], but few of them have been widely used due a lack of both generality and simplicity.

A sufficient interfacial adhesion between the particle and the polymer is another important requirement for nanocomposites in general and for the insulating material in HVDC cables in particular. Cables are subjected to mechanical strains/stresses during installation and this

may lead to cavitation in composites with a poor interfacial adhesion between the nanoparticles and the polymer, which can be detrimental for the performance of the insulation [2]. It has been reported that the interfacial adhesion between particle and polymer has an impact on the macroscopic mechanical properties such as modulus and yield stress [25,26]. However, it is not easy to isolate the effect of the mechanical properties on the interfacial adhesion, because other factors such as the dispersion of the particles and the crystalline morphology of the polymer are also influential [27]. Other methods that are dedicated to fibre composites, such as acoustic emission [28], single-fibre testing [29] or simulation [30] have been used. Methods have been developed to evaluate the interfacial properties of particles in composites, but they are mostly limited to polymer composites based on μm -sized non-fibrous particles [31,32] or particles with a special geometry (usually fibres) [29,33]. Efforts have also been made to evaluate the interfacial adhesion between the polymer and the nanoparticles in nanocomposites. The interfacial adhesion has been related to the mechanical properties of the nanocomposites. Bikiaris et al. [34] and Rong et al. [35] used the yield stresses of the nanocomposites and of the pristine polymer to assess the interfacial adhesion in polypropylene/silicon dioxide nanocomposites. Roumeli et al. [36] assessed the interfacial adhesion in crosslinked high-density polyethylene/diamond nanocomposites from the increase in the Young's modulus of the nanocomposites. Spectroscopic techniques have also been used. Li et al. [37] reported that Raman spectroscopy could be used to evaluate the interfacial stress transfer between particle and polymer matrix in poly(vinyl alcohol)/graphene oxide nanocomposites. Ciprari et al. [38] found that the intensities of several infrared bands were related to the amount of interphase between particle and polymer in PMMA/aluminium oxide and PMMA/iron oxide nanocomposites. The interfacial interactions in composites have a potential impact on the polymer chain dynamics at the interface, as revealed by the glass transition temperature and the intensity of subglass processes. Rong et al. [35] assessed the interfacial interactions in polypropylene/silicon dioxide nanocomposites from the intensity of the mechanical β relaxation process. Natarajan et al. [39] related the shift in the glass transition temperature to the interfacial energy between particle and polymer. The extensive straining of nanocomposite samples followed by high-resolution scanning electron microscopy seems however to be a novel method in this context.

In this paper, a series of silanes with terminal alkyl groups of different lengths (methyl, octyl and octadecyl groups) were used to modify the surface of aluminium oxide nanoparticles. The coated nanoparticles were compounded with a low-density polyethylene, and scanning electron microscopy was used to assess the dispersion of nanoparticles in the polymer. By gradually straining the nanocomposites and examining the strained composites in a scanning electron microscope, the interfacial adhesion between nanoparticles and matrix was assessed. At a certain strain level, different depending on the interfacial

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