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A Method for Dispersion Degree Characterization Using Electro Conductive Mode of Atomic Force Microscopy

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Abstract

Nano-fillers in nanocomposites may grant the matrix material new exceptional properties or improve the existing ones. Usually the effect depends on how frequent the filler is distributed in the matrix. If the filler is well dispersed, then we need a smaller amount of nanoparticles to achieve the same effect. Therefore, it is important to be able to quantitatively characterize a composite, to determine the most useful concentration of nano-filler. Here we propose a new method for polyisoprene – carbon black (Pi-CB) nano-composite characterization with the electro-conducting mode of atomic force microscopy (EC-AFM). In this method electro-conductive channels (ECC) are measured with EC-AFM, and a three dimensional data set is obtained. Maija.Bundule@viaa.gov.lv

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Keywords: Nanocomposite; Dispersion degree; Atomic force microscope (AFM); Carbon black (CB); Polyisoprene (Pi)

1. Introduction

Nanocomposites are popular, because the nano-filler adds new or significantly improves the existing properties of the matrix material. Usually the dispersion degree shows how beneficial the effect of added nano-filler will be. The

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desired effect of nanocomposites usually corresponds to a specific concentration region. As it is desirable to achieve the most beneficial effect using the least amount of nano-filler, it is important to be able to quantitatively characterize the dispersion degree of the nano-filler. Ideally, there is a correlation between a parameter that derives from the dispersion degree and a parameter which describes the desired nanocomposite property.

The characterization of the degree of dispersion includes two aspects – first is to experimentally measure a property of the nanocomposite, that, secondly, is mathematically processed to acquire a quantitative parameter which describes the degree of dispersion. The experimental measurement can be done using visual techniques, macroscopic physical properties determination, spectroscopic techniques or radiation scattering.

The first part in general is realized by microscopy techniques – optical[1], scanning electron[2], transmission electron[3] or atomic force microscopy techniques[4]. Usually an image is acquired which is further mathematically processed. This is the most direct way, because an image shows directly how the filler particles are distributed in the matrix. The disadvantages of it are that mostly only a part of the whole sample is seen and therefore characterized, which sometimes may not completely represent the whole material. Another disadvantage is that only the surface or a thin film of the sample is observed and the volume is not taken into account.

Some macroscopic physical properties of a material are capable to give away the degree of filler dispersion. The important aspect is the change in the properties, for example rheology[5], barrier properties[6] or viscosity[7]. Usually the dispersion degree very significantly impacts the macroscopic physical properties therefore that can be used to quantitatively describe the dispersion degree. There are also spectroscopic techniques – X-ray photoelectron spectroscopy, fluorescence, dielectric spectroscopy – and scattering techniques – X-ray, visible light and neutron scattering – for the characterization of dispersion degree. The spectroscopy techniques measure the amount of particle interaction with the matrix and therefore determine how well dispersed is the nano-filler, but the scattering techniques basically observe the scattered radiation. When the radiation flux interacts with the composite, the particles scatter the light in certain angles which depend on the particles size. These techniques take into account the whole sample, but the disadvantage is that they need prior calibration, to determine what value attributes to which degree of dispersion. The second part – mathematical processing – is very dependent on the specific nanocomposite material. There are methods for fiber[8], sheet[9] or particle type of filler[10].

In this paper we propose a way for specifically Pi-CB nanocomposite characterization using atomic force microscopy electro conductive mode (EC-AFM). Usually AFM would only determine the surface structure of the composite, but by using the electro conductive mode, we see the channel system what is made of the electro conductive nano-filler particles. Therefore, we can indirectly characterize the sample in volume. The mathematical method was chosen from a recognized publication[10] and further adapted to our purpose.

2. Experimental measurements

2.1. Sample preparation

The composite material is made from dispersing carbon nanoparticles in polyisoprene matrix. The uncured rubber contents are as shown in Table 1. Nano-filler is electro conductive carbon black (CB; Printex XE-2) with average particle size 30 nm.

Table 1. Contents of uncured rubber.

Component	Mass parts
Natural polyisoprene rubber	100
Zink oxide	5
Sulphur	3.5
Stearic acid	1
N-cikloheksil-2-benzothiazole-sulfenamide	0.8

The samples are prepared by a solution method. Firstly, uncured rubber is dissolved in chloroform. In another flask CB is dispersed in chloroform by ultrasound probe (Hielscher UP200S) for 5 minutes with the specific