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Review

Structural assessment of nanocomposites

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ABSTRACT

This paper provides an overview on structural assessment of nanocomposite materials. First of all, a brief description of advanced structure characterization methods such as scanning electron microscopy, X-ray diffraction, transmission electron microscopy, atomic force microscopy, and scanning tunneling microscopy is presented. Secondly, applications of these methods for analysis of structures and compositions of typical nanocomposites are introduced. The nanocomposites are formed by different nanoscale processing technologies. Electrochemically polymerized polyaniline (PANi) nanocomposites, thermomechanically processed metal matrix nanocomposites, nanocast ceramic matrix composites are typical examples discussed in this paper. Case studies on several functional nanocomposites for energy storage/conversion, catalysis and sensing applications are mentioned. After that, assessment of the interface structures of nanocomposite materials using surface characterization techniques and mechanical damage models is discussed. Finally, concluding remarks are provided.

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1. Introduction

A composite material has the synergistic properties of its matrix and reinforcement. The structure of a composite can be controlled in manufacturing processes. It determines the performances of the composite material. Composite materials can be classified by matrix types. There are three major types according to the nature of the matrices, i.e. metal matrix composites, ceramic matric composites and polymer matrix composites. Composite materials are also classified as continuously reinforced or discontinuously reinforced depending on the aspect ratios of the reinforcements. Still, composite materials can be classified by the shape of reinforcements. If the reinforcement in a composite material is zero dimensional, i.e. in particle form, the material is called particle reinforced composite. Obviously, particle reinforced composites belong to discontinuous ones. One of the examples is the particle dispersion reinforced high strength steels. If the reinforcement in a composite material is in long fiber form, the material is called one dimensionally reinforced composite. Ultrahigh molecular weight polyethylene (UHMWPE) fiber reinforced epoxies fall into this category. The reinforcement in a composite could be in planar form. In such a case, the material is a 2-D composite. For example, the hybrid composite containing alternatively stacked aluminum foil and fiber reinforced layer is a two dimensionally reinforced composite material. Three dimensional reinforcements are typically produced by interweaving continuous fibers. For example, woven carbon fibers form 3-D frames. The frames can be impregnated with phenolic resin. Then, calcination in inert atmosphere generate three dimensionally reinforced carbon–carbon composite materials. 2-D and 3-D composites are continuously reinforced materials.

Nanocomposite materials consist of nanoscale reinforcements. The structures of nanocomposites determine the properties and performances of the materials. In this paper, structural assessment of nanoscale phase reinforced composite materials is reviewed. Various structural characterization tools such as scanning electron microscopy, X-ray diffraction, transmission electron microscopy, atomic force microscopy, and scanning tunneling microscopy will be briefly introduced. The structures of typical nanocomposites will be shown. Finally, we will extend our discussions on assessment of structural integrity of nanocomposites using an energy dissipation model and a nonlinear mechanical damage model.

2. Nanoscale structure characterization techniques

Since the size of phases in nanocomposites is at nanometer level, powerful characterization tools are needed for observing each phase and assessing the structures of the composite materials. A nanometer is one billionth of a meter, which is so small that high resolution microscopes have to be used. The following subsections provide a brief description of various morphological analysis methods. For more details, it is encourage to read the related books. First, the atomic force microscopy is introduced. Then the scanning probe technique is discussed and the work mechanism of scanning tunneling microscopy is given. After that, electron microscopic techniques including scanning electron microscopy and transmission electron microscopy are presented. X-ray diffraction, energy dispersive spectrum and focus ion beam techniques are also briefly mentioned in the last part of this section.

2.1. Atomic force microscopy

An atomic force microscope (AFM) uses a tiny and sharp tip to tap or touch the surface of the specimen. Atomic force microscopy (AFM) is classified as a kind of scanning probe microscopy (SPM). The resolution of AFM is at the sub-nanometer scale or angstrom level. The magnification in an atomic force microscope is the ratio of the actual size of a feature to the size of the feature when viewed on a displaying device. There are different work modes. AFM may run under either contact mode or non-contact mode (tapping mode). Under contact mode, the scanning tip is attached to the end of a cantilever across the specimen surface while monitoring the change in cantilever deflection with a split photodiode detector. The tip may contact the specimen surface through an absorbed fluid layer on the surface. A feedback loop maintains a constant deflection. According to Hook's law, the magnitude of the atomic force can be calculated, which is at the level of nano-Newton or micro-Newton. The vertical distance the scanner moves at each pixel is stored to form the topographic image of the specimen's surface. Typically, the contact mode is used for imaging hard and shallow surface, the structure with periodicity, or the specimen in liquid environment.

Under a non-contact mode or tapping mode, the cantilever is oscillating near or at the resonance frequency. The oscillating amplitude is in the range from several tens to one hundred nanometers. The tip lightly taps on the surface of the specimen when the scanner moves. A feedback loop maintains a constant oscillating amplitude. The vertical position of the tip is measured at each pixel of scan to generate the topographic image. Comparing the contact mode and the non-contact mode, there is difference in the resolution of the image. The contact mode allows to generate much higher resolution images. However, the tapping mode maintains a constant tip-specimen reaction. The tip has less chance to be struck or broken. Besides, in the contact mode, the tip scratches the surface of the specimen. This may cause the deformation of the surface of those soft materials. The images obtained could have some extent of distortion.

AFM may also run in the so-called phase mode. The work mechanism is based on the fact that measuring the phase shift of the cantilever beam holding the AFM tip is carried out in stead of detecting its resonance frequency change. The phase mode is unique in the fact that it can generate material composition information. Even though the surface of a specimen is flat, if the material consists of different phases or functional groups, the surface mapping results can reflect the phase/composition information. For example, the vibration phase shift of the AFM tip generated by $-CH_3$, and $-COOH$ can produce clear AFM images of certain polymers with significant contrast revealing the locations of these functional groups.

2.2. Scanning tunneling microscopy

A scanning tunneling microscope (STM) works under the following mechanism. A very fine tungsten tip made through electrochemical etching is positioned within a couple of nanometers