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Material Behaviour

Nanoindentation behaviour of layered silicate reinforced unsaturated polyester nanocomposites

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Abstract

The effect of various loading levels of nanoclay reinforcement on the nanomechanical properties of layered silicate nanoclay reinforced unsaturated polyester (UPE) nanocomposites were investigated by a nano-indentation test method. The clay was dispersed into a UPE matrix via blending using a mechanical stirrer. Structural studies were carried out using a wide angle X-ray diffraction (WAXD) method and correlated with the nanoindentation results. This shows that nanoindentation behaviour is strongly influenced by clay reinforcement and the degree of clay dispersion in the polymer matrix. The experimental results show that there is a strong correlation between nanomechanical properties and inter layer *d*-spacing of clay particles in the nanocomposite system. Incorporation of 1%, 3% and 5% by weight nanoclay into the polyester resin results in an improvement in hardness of 29%, 24% and 14%, respectively. The elastic modulus increased from 5393 MPa for unreinforced polyester to 6646 MPa (23% increase) with the introduction of 5% by weight nanoclay.

Keywords: Nanoindentation; Nanocomposite; Intercalation; Hardness; Nanoclay; Reduced modulus

1. Introduction

The use of clay/polymer nanocomposites technology has been demonstrated to be a useful way of producing materials with a wide range of engineering properties [1]. These improvements are achieved by the expansion of clay layers and the dispersion of the separated individual clay layers into the polymer [2].

Nanoindentation is a promising way of measuring the mechanical properties of materials at smaller length and load scales than allowed by other testing methods, thus allowing individual constituents to be examined [3]. Nano and micromechanical testing involves the use of rigid indenters, typically with diamond or diamond-coated tips [4]. Nanoindentation is also known as depth sensing indentation and involves obtaining quantitative force versus displacement data and determining the elastic modulus, E, and hardness values, H of materials even beyond their elastic limit [5]. Knowledge of such mechanical properties at the nano level can be important for certain materials selection and design criteria and applications.

In this study nanoindentation tests were carried out on nanoclay reinforced unsaturated polyester nanocomposite specimens manufactured by a hand lay up process. Varying clay loading levels were

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used in an attempt to analyse the effect of reinforcement on nanomechanical properties. A nanoindentation test method was employed to determine load–displacement, reduced modulus, elastic modulus and elastic plastic depth for evaluating nanohardness. Results from these nanoindentation tests, in the form of hardness and elastic modulii, are quantified, discussed and also compared to E-glass fibre reinforced unsaturated polyester composites.

2. Experimental details

2.1. Apparatus

All the nanoindentation tests in this study were performed using commercially available apparatus, namely the Nano Test^{TM} (Micro Materials, UK). A Berkovich (three sided pyramidal) diamond indenter tip manufactured by Micro Materials was used throughout [6] and a schematic diagram of the nanotest system is shown in Fig. 1.

Nine symmetrical indentations (in the form of a 3×3 matrix, 30 μm apart) as shown in Fig. 2 were made on each specimen. The coupons were cut from the composite laminates with approximate dimensions of $18\,mm \times 18\,mm \times 3\,mm$. The specimens were mounted onto the nanoindentation fixture using a suitable adhesive. All tests were conducted at an approximate temperature of 27 °C. Typical experimental indentation parameters used for all measurements were as follows:

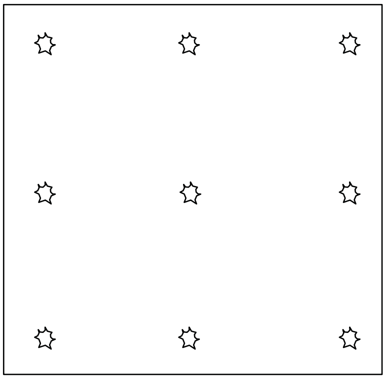

Fig. 2. Symmetrical indentations (30 μm apart) (not to scale).

Initial load: 0.1 mN.
The maximum load for all indents:15 mN.
Loading and unloading rate (strain rate): 2.00 mN/s.
Dwell time or holding time at maximum load: 5 s.

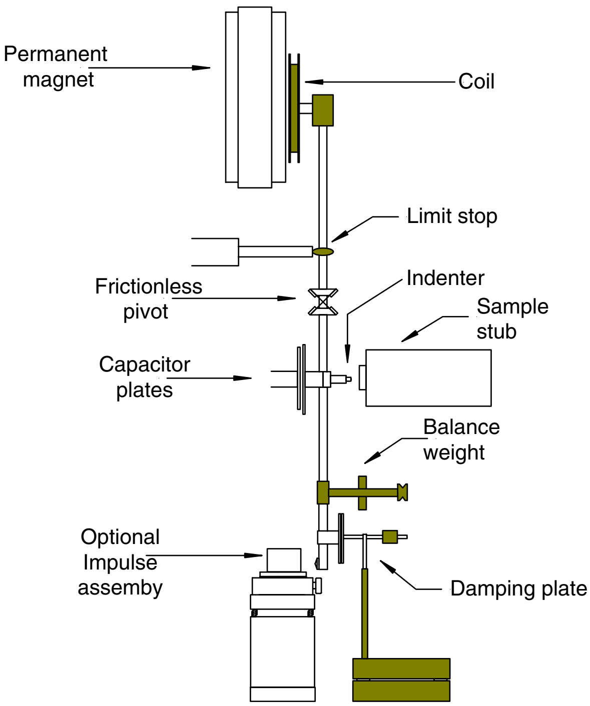

Fig. 1. Schematic of the nanotest system.

2.2. Materials

Nanoindentation experiments were carried on four different nanocomposite systems using an organically modified layered silicate (referred to as LK-EP-C) as reinforcement. The matrix material was based on unsaturated polyester (UPE), Trade Name NORPOL 444-M888, mixed with a curing catalyst, methyl ethyl ketone peroxide (MEKP) at a concentration of 0.01% w/w of the matrix, supplied by Reichhold UK Ltd. This resin system was chosen for its low viscosity and low shrinkage.

2.3. Processing

Four different types of nanocomposites sample (containing 0, 1%, 3% and 5% by weight layered silicate reinforcement, respectively) for nanoindentation behaviour characterisation were fabricated using a hand lay up method.

The layered silicate nanoclay was first dried for 30 min at 100 °C to remove any moisture present. Then the varying concentrations of dried nanoclay were dispersed into the UPE matrix. The mixture