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Characterization Multiscale of Poly(propylene) Nanocomposites Made up with Modified Nanoclay

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Summary: This paper shows the characterization carried out in polypropylene nanocomposites made up with different proportions of modified nanoclays (1% 2.5% and 5%). Modified nanoclay (montmorillonite) has been added to improve the mechanical properties of the original matrix and increase its heat resistance. The objective of this work is to characterize multiscale nanocomposites, from nano-scale to macroestrucutural scale using thermography, nanoindentaton and mechanical testing.

Keywords: clay; mechanical properties; nanocomposites; poly(propylene); thermography

Introduction

The application of composite materials is widely extended; it presents some advantages respect to other materials.[1,2] The characterization of mechanical properties in these materials is usually made by measuring macroscopic properties,[3] but macroscopic properties are mainly influenced by the microstructure and by the properties of the individual phases.[4,5]

The purpose of this work will be to study the influence of microstructural parameters of multiphase materials on macrostructural behavior. This study is divided in three parts. First, an experimental study of mechanical properties, subsequently, a study of the influence of microstructural parameters. Finally, an thermographic study.

By means of this characterization is possible to establish patterns of behavior and multiscale relations.

Experimental Part

Material was considered as a biphasic composite with a matrix (a copolymer of polypropylene and polyethylene) and modified nanoclays, in different proportions, uniformly distributed in the matrix.

To characterize the samples, a set of four test specimens has been used, one for each percent of nanoclays. All specimens were manufactured according to UNE-EN ISO 527-2 1997[6] using an injection process to obtain type 1A samples. The shape and dimensions of the samples are shown in Figure 1.

Mechanical Characterization

Was done in accordance to UNE-EN-ISO-527-1: 1996 ‘‘Determination of tensile properties - Part 1: General principles’’,[7] UNE-EN-ISO 527-2: 1997, ‘‘Plastics - Determination of tensile properties - Part 2: Test conditions for moulding and extrusion plastics’’,[6] and ISO 5893:2002 ‘‘Rubber and plastics test equipment - Tensile, flexural and compression types (constant rate of traverse) Specification’’.[8]

For the accomplishment of tests has been used a universal testing machine Instron 5582 and an extensometer according ISO 5893 (Figure 2). Universal Testing Machine allows for measurement of tensile and elastic modulus (In this case, load operating range: 2N to 100 kN and speed: 0.05 to 500 mm/min). The load was applied to 0.5 mm/min for calculating the elastic modulus and to 50 mm/min for the rest of parameters.

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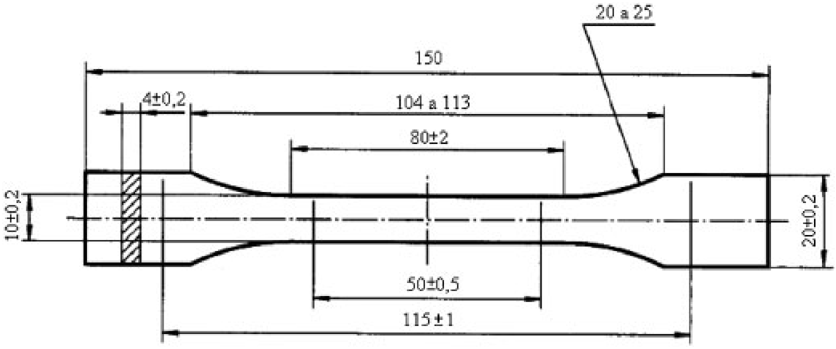

Figure 1.
Shape and dimensions of the samples used in the tests.

Before the tests, the specimens were inspected by ultrasound to ensure that the samples have no internal defects that could produce erroneous results.

Nanostructural Characterization

Instrumented-indentation testing has been developed for the purpose of probing the mechanical properties of very small volumes of material. Nanoindentation also provides the ability to map the spatial distribution of mechanical properties with good resolution. Hardness and elastic modulus are the properties most frequently measured by nanoindentation.

The equipment used has been the Nano Indenter G200 (Figure 3) with an indenter type Berkovich, operating in ''Continuous Stiffness Measurement'' (CSM) that allows the continuous measurement of the contact stiffness during loading and not just at the point of initial unload.

As the indenter is driven into the material, both elastic and plastic deformation cause the formation of a hardness impression conforming to the shape of the indenter to some contact depth, hc. As the indenter is withdrawn, only the elastic portion of the displacement is recovered; this recovery enables one to determine the elastic properties of a material.

Figure 2.
Universal Testing Machine.

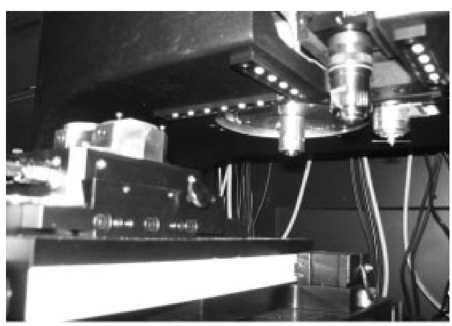

Figure 3.
Nano Indenter G200.

Table 1.
Preparation parameters.

Method	Surface	Abrasive	Force	Rotational speed	Time
Grinding 1	SiC 320	Water	20N	300 rpm	20 seg.
Grinding 2	SiC 800	Water	20N	300 rpm	60 seg.
Polishing 1	MD-DAC	DiaproDAC 3	30N	150 rpm	15 min.
Polishing 2	MD-NAP	NAP-B	20N	150 rpm	5 min.
Polishing 3	MD-Chem	OP-U	15N	150 rpm	5 min.

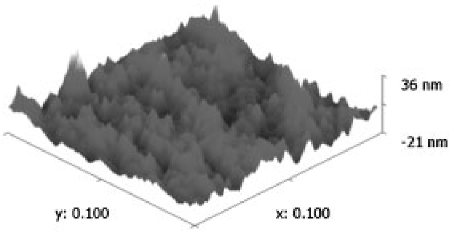

Figure 4.
Map of sample surface of polypropylene.

Before nanoindentation tests, the surface was prepared by polishing. Surface smoothness is important because contact areas are calculated from the contact depth and area function rather than observed directly. Thus, the degree of required smoothness depends on the magnitude of the measured displacements and the tolerance for uncertainty in the contact area.

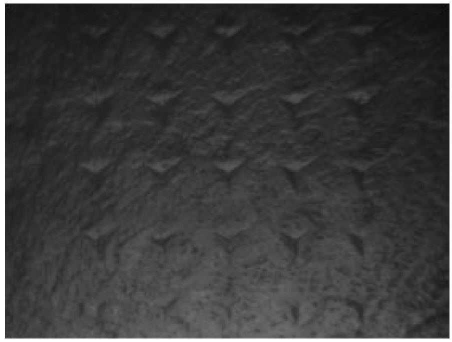

Figure 5.
Indentations on polypropylene.

The Table 1 shows method used for the preparation of the surfaces.

After surface preparation, roughness was determined using maps. For this type of nanocomposites, the average roughness obtained was 65 nm (Figure 4).

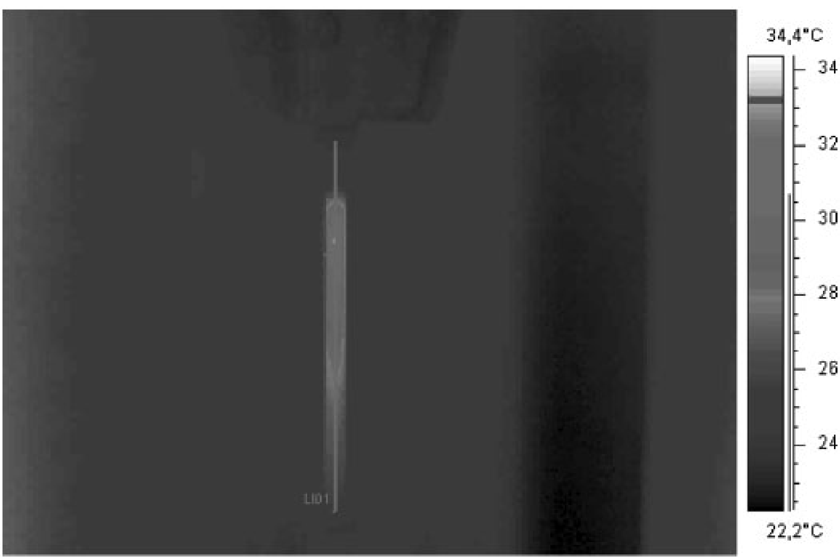

Figure 6.
Thermographic imaging for the study of thermography, with the line of traction.

Table 2.
Test results of multiscale characterization.

		% nanoclays			
		0	1%	2.5%	5%
MECHANICAL CHARACTERIZATION	Elastic modulus (MPa)	845.23	854.90	855.57	916,49
	Tensile stress (MPa)	15.76	15.56	15.14	15.09
	Deformation (%)	303.17	93.31	62.92	47.27
NANO-SCALE PROPERTIES	Elastic modulus (MPa)	609.50	604.22	647.65	691.23
	Tensile stress (MPa)	12.77	12.5	12.45	12.38
THERMOGRAPHY	Temperature (°C)	39.03	36.17	34.90	32.23

To determine the nanostructural properties, 13×13 nanoindentation matrices were made with a velocity of 10 nm/s and 2000 nm deep as shown in Figure 5.

Termography

The technique used is quantitative thermography.[9] The interpretations of the images are based on realistic models leading to quantitative identification of parameters, in this case, tensile stress (Figure 6).

It was studied a correlation between the level of concentration, thermal capacity and tensile properties.

The calculation of emissivity has been performed by comparing the surface temperature of a known emissivity to the temperature of the sample.

The thermograph study of the process allows us to relate the internal energy of the specimen with its concentration as well as being a tool to support the results obtained, and an auxiliary method of obtaining variables.

Results and Discussions

Table 2 shows the results for different proportions of nanoclays:

The results show that there is a relationship between the different methods of characterization. This study found that an increase in fraction of nanoclay inclusions causes an increase in the elastic modulus, and a decrease in deformation, tensile stress and in temperature (Figure 7). The relationship between macro and microstructural properties is constant for all the proportions of nanoclays.

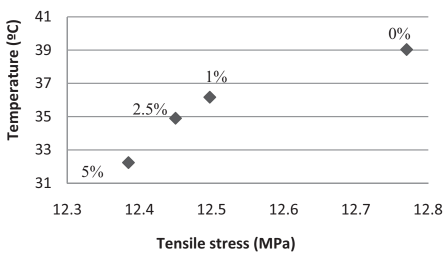

Figure 7.
Relationship between temperature, Tensile stress and nanoparticles fraction.

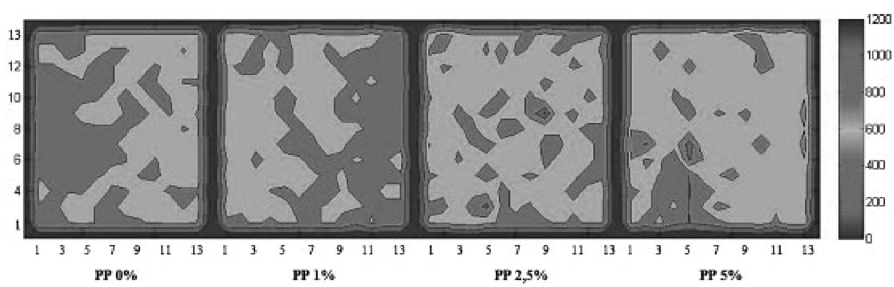

Figure 8.
13×13 Elastic modulus matrices at 2000 nm.

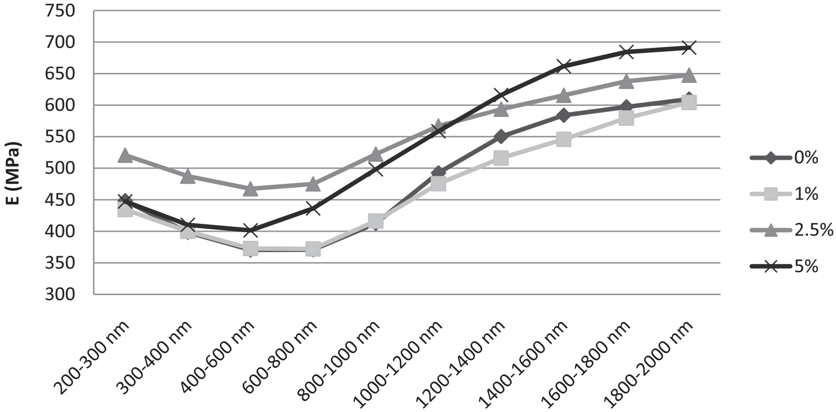

Figure 9.
E obtained at different depths between 200 nm and 2000 m.

Furthermore, this study has shown that it is important to consider the microstructural properties because through them can be obtained precise information. Next figures show the elastic modulus (E) for 13×13 indentations in the different materials (Figure 8).

The CSM operating mode allowed us to estimate the elastic modulus at different depths (Figure 9). It is important to note that the calculated elastic modulus in the area next to the surface is not considered as bulk values.

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