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Characterization of nanoparticles doped composites using ultrasound

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ABSTRACT

The aim of this work is the non-destructive automatic mechanical characterization of nanoparticles doped composites using ultrasound in order to understand and control the dispersion of the dopant nanoparticles in the final product. We present a method which is able to measure the elastic constants of composites (Youngs, Bulk, Shear Modulus and Poissons ratio), in addition to other parameters as density, sound velocity and thickness, providing information of the nanoparticles dispersion in the samples. All results are obtained with a single ultrasonic measure at each point of the samples' surface in an immersion setup with both pulse-echo and through-transmission measurements simultaneously, obtaining detailed information for all the samples' surface in a XY scanning. All the analysis is performed automatically, that is, no manual correction or adjustment is needed at any stage of the process. To validate the results, a polyester based resin has been analyzed with different concentrations of graphene nanoparticles as dopant. The method has shown to be very accurate and reliable. The resolution of the values obtained for the elastic constants is limited by the resolution in the velocities measurements, for which we have achieved a resolution in the order of cm/s, thus providing very accurate measurements of the elastic constants.

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1. Introduction

Nowadays it is almost impossible to find an area in the industry in which composites are not used. From aircraft and aerospace industries to dental restorative composites we will find a wide set of applications: wind mills, containers, automotive and marine structures, chassis and hulls, helmets, glasses, wearables, etc. They can also be designed with specific physical or chemical properties, as photo-sensitivity, electromagnetic-sensitivity or thermo-sensitivity, that make them suitable for the design of sensors and actuators. Even ballistic protective layers of polymers can be easily applied to all of the above items. One of the pioneers and most successful uses of composites are those related to mechanic structures. Most of the structures developed nowadays share the same principle: a polymer matrix reinforced with fibers. The possible combinations extend to a wide variety of solutions, but mainly using either epoxy, vynilester or polyester resins reinforced with glass, carbon or aramide fibers, processed and formed in many different ways: Autoclave, Poltrusion, Resin Transfer Molding, etc. In the last few years, the revolution of nanotechnology has spread to the field of composites resulting in a new set of plastic and resin-based composites [1–5].

These nano-filled polymer composites are obtained adding nanoparticles to the polymer matrix to enhance its properties. It is easy to find hundreds of research works related to the use of nanomaterials and polymers [2,6–8], but it is very difficult to apply those in commercial products like aircrafting or boating industries. One of the reasons is that it is very difficult to integrate the nanomaterial (in the form of nanoparticles or nanotubes) in the manufacturing process, mainly due to two factors. On the one hand, the mixture in industrial volumes of the polymer matrix and the nanoparticles does not always result in a homogeneous product, which could be due to several factors, such as the integration of the nanoparticles in the local chemistry of the polymer chain [6,9–11], or simply the functionalization of the nanoparticles before the mixing [9,12]. On the other hand, even if the mixing is homogeneous, nanoparticles (especially in the case of nanofibers and nanotubes) tend to agglomerate around the fibers of the fabric used as reinforcement, impeding the correct diffusion of the

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mixture throughout the material. As a result, the homogeneity of the mechanical behavior in the final product is compromised, especially in the construction of large structures and surfaces, as windmills wings, boat hulls or containers.

Due to the above, it is essential to conduct a study to analyze the dispersion of nanoparticles in the final product. This can be done using the elastic moduli of the materials, closely related to their mechanical properties. Usually, this is made in the manufactures' laboratories using tensile stress tests, which provide information about the elasticity modulus. Unfortunately, these methods have some drawbacks that make them not suitable when the goal of the test is the analysis of nanoparticles dispersion within the samples, or in other words, how homogeneous is the mechanical behavior of the sample along its surface. These measurements provide an average of the specimen properties, but not information about the real dispersion of the nanoparticles. Furthermore, specimens crack in the weakest point, which could be due to an improper dispersion of nanoparticles in that particular point. To have an idea of the product behavior, it requires the analysis of a high number of specimens, and even then the result can be both inaccurate and ambiguous. Finally, these measurements are highly time consuming, require expensive equipment and trained personnel.

There are many other techniques that can be used, as Dynamic Mechanical Analysis (DMA) [12,13], Thermomechanical Analysis (TMA) [13], Thermal Gravimetric Analysis (TGA) [13,14], Atomic Force Microscopy (AFM) [15], UV–vis Absorption Spectra [15], Raman Spectra [15], IR Spectroscopy [14,17], Fourier Transform Infrared Spectroscopy (FT-IR) [13], X-ray Photoelectron Spectroscopy (XPS) [14], X-ray Diffraction (XRD) [11,14–17], Transmission Electron Microscopy (TEM) [11,13–16] and Scanning Electron Microscopy (SEM) [11,13,15]. Unfortunately, they all have serious shortcomings, including one or more of the following:

1. Required equipment is very expensive.
2. They require very specialized and trained technicians.
3. They provide information about the inner structure of the materials, but not about their mechanical properties.
4. They do not provide information locally, and/or only from the surface.
5. They required specific manipulation of the samples (which can affect the actual mechanical properties of the product) and/or additional facilities (thermal chambers, vacuum, etc.).
6. Results are difficult to interpret, and could even be biased by the samples manipulation required.

In conclusion, all those techniques are good, but do not allow whole sample inspection, sometimes use dangerous radiation, use complex equipment, are quiet expensive, and most relevant for our goal, they do not provide information about the distribution of the mechanical properties locally.

Using ultrasound it is possible to control the process in situ in the laboratory or in the manufacturer workshop, which is a qualitative advantage for the end product, plus it can result in the reduction of costs and production time. Ultrasonic techniques can provide the elastic constants of the materials as a function of the longitudinal and shear propagation velocities and the density of the samples. Several methods can be found for the accurate calculation of the velocities at any point in the material [18–20], but in the case of the density it is not so simple. Although there are numerous methods to obtain the density of solids in the laboratory (densitometer, pycnometer, etc.), these require additional laboratory equipment and preparation of the samples, and results may be inaccurate. On the other hand, there are the methods that use ultrasound, using the relationship between the acoustic impedance, propagation velocity and density. There are numerous well known techniques for obtaining these parameters [21–25], but they require complicated processes for correcting the diffraction due to the difference in the propagation path of each pulse as well as parallelism between surfaces.

In this work we present a simple and fast method to calculate all the parameters needed to obtain the elastic constants of doped resin samples produced in the laboratory, and therefore to estimate the dispersion of the nanoparticles within the sample. With this method, in a single measurement and at each point on the surface of the analyzed specimen, it calculates automatically the propagation velocities, the thickness and the density with high accuracy, and without the need of any prior knowledge about the samples besides its mass. It is automatic and does not need any specific calibration nor the action of the user except changing the samples between measurements.

The work is organized as follows. It starts in Section 2 with a short review of the mechanical properties of the materials to introduce the relation between elastic constants and sound velocity. Section 3 describes the method followed for the calculation of the ultrasonic parameters involved in the analysis. Section 4 describes the different set-ups designed for the experiments and the results obtained after the analysis of the samples. Finally, conclusions will be summarized and discussed in Section 5.

2. Review of the mechanical properties of the materials

2.1. Stress, strain and stiffness

The elastic properties of solids are key properties for the design of mechanically loaded components [2,26–28]. Specially, the elastic modulus and Poissons ratio, which describe the elastic behavior of isotropic materials. The elastic moduli measure the resistance opposed by a material to being deformed elastically, and is measured as the ratio between the stress applied and the resulting strain. The stress is the force causing the deformation divided by the area to which the force is applied and it is measured in Pascals, and strain is the ratio of the change in some length parameter and the original value of the length parameter, therefore dimensionless.

The state of stress at an arbitrary point P in a structure depends on the orientation of the applied force F acting on P and the orientation of the reference plane with respect to a reference coordinate system. If the point P under analysis is considered as an infinitesimal cube, the stress acting on each of the six sides of the cube can be resolved into components normal to the face and within it, as shown in Fig. 1, in which a plane oriented normal to an axes i is called the i-plane.

A stress σ_{ij} is defined as acting on the i-plane and being oriented in the j direction. Components of the stress tensor perpendicular to the considered plane are denoted as normal stress (σ_{ii}), while stress components acting in the same plane are called shear stress (σ_{ij}), resulting in six shear and three normal stress components

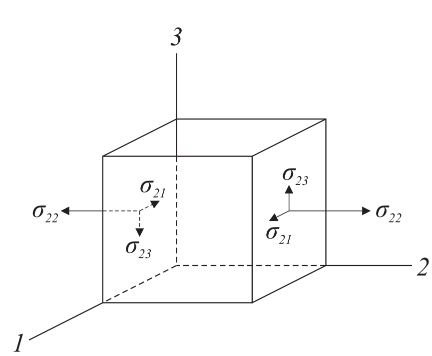

Fig. 1. Example of stress components acting on plane 2.

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