Dynamical mechanical characterization of a nanostructured vibration damping layer

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Abstract. A vibration damping layer can dissipate relatively large amounts of energy. A known effect of this dissipation of energy is the reduction of the response of a structure to sound and vibration excitation and the reduction of the transmission of sound through structures at high frequencies. Added damping also increases the impedances of structures at their resonances and thus may improve the effectiveness of vibration isolation at these resonances. It has been observed that the molecular behavior of new nanomaterial composites may have significant influence on their shock and vibration insulation properties. Dynamical mechanical characterization of these nanostructured materials is important for determining the potential application as a viscoelastic vibration damping layer. In this work, inclusion of several nanostructures to thermostable polyurethane has been made and their main elastic properties have been measured using current standard methods. It is observed that specific amounts of nanostructures can improve the impact damping capabilities of the material, with the advantage of reduced thickness and weight as compared to traditional materials.

1. Introduction

One of the recent experimental and research objectives is to improve the thermal, electrical and mechanical performance of a polymer by adding nanostructures. They include reinforcement nanotubes, nanoparticles (NP) and nanofibers that, depending on their characteristics, improve either the physical or chemical polymer properties [1,2]. The advances in nanotechnology have provided several new materials with diverse mechanical properties which can be used in both acoustic and vibration applications. In particular, the molecular behavior of these new nanocomposites may have a significant influence on their impact and shock absorption [3].

This work aims to evaluate, through vibration and mechanical testing, nanostructured materials with different types of nanoclusters at different mass quantities. The main objective is to identify nanostructured materials for their potential use as viscoelastic layers to reduce impact noise in floating slabs.

2. Nanostructured damping layers

To synthesize the nanostructured samples, four types of clay were used: cloisite 15A, cloisite 30B, hectorite, and laponite. Initially, the clays were dried for 8 hours in an oven to 110°C. A polymer matrix thermostable polyurethane (PU 455-203 # 1) was used and proportions of 0.5%, 1%, 2%, 5%
and 10% in mass for each clay where chosen to synthesize the films. Subsequently, the polymer in pellet form was mixed with tetrahydrofuran (THF) and N, N’-dimethylformamide (DMF), with mass ratio of 20% for the polymer, THF to 40% m/m and DMF to 40% m/m. The dried clay was then added to the mixture and samples were shaken during 30 min in an intensive mixer. Subsequently, the samples were mixed again during a period of 12 hours in a magnetic stirrer.

After the mixing process, the stirring process was repeated for 30 min in the intensive mixer. Then, the samples were agitated by applying sound energy during 30 min in a sonifier Branson with 10% intermittent amplitude as well as pulsed (30 s on, 30 s off). Finally, the samples were subjected to 30 min of stirring in the intensive mixer and, again, in the sonifier during 30 min. Samples were poured into teflon molds and cured in an oven at 45°C for 12 h. Additionally, single wall (SWCNT) and multiwall (MWCNT) carbon nanotubes samples with concentrations of 0.5% and 21% m/m were made for comparison purposes. The process of mixing and drying was the same for the case of samples with NP and no additional chemical modifications were made.

3. Experimental results

In order to evaluate the mechanical properties of the nanostructured materials, a dynamic mechanical analysis (DMA) was used. The working principle of DMA is to apply a controlled sinusoidal deformation or strain to a sample of known geometry [4]. Thus, to study the mechanical properties through the DMA, it is possible to separate the main mechanical properties of a material into two parts: the elastic modulus corresponding to the elastic response under an applied strain, and the loss modulus, which indicates the damped response to the applied strain. Finally, the loss factor shows the exchange rate between the loss modulus and elastic modulus [5]. In addition, the procedure defined in ISO 9052-1 [6] was used to evaluate the dynamic stiffness of the material samples. It is known that the noise reduction properties of a viscoelastic layer are related to its dynamic stiffness [7].

3.1. Mechanical test using DMA

After evaporating THF and DMF solvents from the samples in the oven, both density and thickness measurements of each nanostructured film were performed. Subsequently, tests were carried out using a commercial dynamic mechanical analyzer (DMA). The samples were cut with dimensions of 7 ± 2 cm long, 0.5 ± 0.1 cm wide with a thickness of 0.35 ± 0.05 mm. Furthermore, to have more reliable values, five samples of each nanostructured polymer were measured for each experiment.

To evaluate the Young’s modulus E of the samples, mechanical traction on the DMA test was performed for each nanostructured film under a constant extension rate of 10 mm/min at room temperature. In addition, all samples were brought to the point of breakage. After obtaining their responses against static tensile strain, the Young’s modulus was extracted from the linear elastic section of each sample.

To evaluate the dynamic mechanical properties of polymers (dynamic modulus and loss factor), the experimental conditions were a constant room temperature and a frequency sweep signal from 0.1 Hz to 90 Hz.

Table 1 shows the increase of the Young’s modulus of each nanostructured sample in relation to the pure polymer matrix thermostable polyurethane (E = 1.542 ± 0.183 MPa). We notice that E increases with an increase in the amount of NP in all samples (except for cloisite 30B which remains constant from amounts larger than 2% m/m). The increase in Young’s modulus is caused by the exfoliation that the material experiences and the bonds generated between the organic and inorganic nanoclay. Adding NP to the polymer matrix causes the high-E of NP to modify the low-E of the polymer matrix, which results in an increase of the Young’s modulus of the nanostructured material. Thus, as reported previously, the greater the amount of NP in the PU, the higher the E in the nanostructured film [8]. However, at higher values of E, the fragility of the material increases (which increases with the slope of the elastic region of the static tensile curves), which causes breakage more easily [4].
Figure 1 shows the values of loss factor as function of frequency for all samples of NP and CNT with 0.5% m/m concentration. It is noted the apparent increase in the loss factor for SWCNT and MWCNT samples. This is due to the mobility of the CNTs and their Van der Waals bonds with the polymer chains [9]. All loss factor curves showed a slight drop in the slope due to the viscoelastic behavior of materials generated by an increase in the elastic behavior [10]. When the frequency is increased, the vibration of the viscoelastic material is inhibited by the polymeric chains, resulting in a stiffer material.

Table 1. Increase in percentage of the Young’s modulus of each nanostructured sample in relation to the pure polymer matrix thermostable polyurethane (E = 1.542 ± 0.183 MPa).

| Material’s concentration (% in mass) | Cloisite 15A (%) | Cloisite 30B (%) | Laponite (%) | Hectorite (%) |
|-------------------------------------|-----------------|-----------------|--------------|--------------|
| 0.5                                 | 110             | 47.4            | 86.7         | 101.6        |
| 1.0                                 | 295             | 66.3            | 96.9         | 164.7        |
| 2.0                                 | 340.9           | 100.6           | 135.2        | 164.5        |
| 5.0                                 | 984.1           | 83.4            | 227          | 657.5        |
| 10.0                                | 4646.5          | 92              | 2495.1       | 1820.1       |

Figure 1. Loss factor $\eta$ versus frequency from 0.1 Hz to 90 Hz under constant temperature of nanostructured polymers at 0.5% concentration of added nanostructures.

3.2. Dynamic stiffness test
Based on the standard ISO 9052-1 [6], which specifies the process for determining the dynamic stiffness, 8 ± 0.5 kg-steel plate of dimensions 200 ± 3 mm x 200 ± 3 mm was used. The samples were placed and centered on two heavy marble plates 400 mm x 400 mm which avoided vibration interference measurement between the nanostructured films and direct surface. Data acquisition and frequency responses were performed by a FFT real-time signal analyzer connected to a laptop via a LAN port. The sensors used were an accelerometer and an impact modal hammer (with a steel, rubber
and hard plastic end). Handling the data acquisition process and data analysis was performed by specialized software. Results obtained for single layer of these nanostructured materials have been published before by the authors [11].

Figure 2 shows the results of the dynamic stiffness using nanostructured materials made up of 10 layers, resulting in approximately 3.5 mm thickness for each sample. We observe that the stiffness changes with an increase in the concentration of NP present in the samples. In addition, the behavior of the dynamic stiffness of materials at 0.5% m/m concentration is like the polymer matrix. However, increases of NP beyond 1% m/m for all samples show a dissimilar behavior between the different nanostructures.

For samples with cloisite 15A, 30B and hectorite with NP ≥ 2% m/m the dynamic stiffness presents a slight fall as the amount of NP is increased. Laponite has a remarkable fall in the dynamic stiffness value below the corresponding value of pure polyurethane. In laponite films, there is a clear decrease in dynamic stiffness for 5% and 10%. These films demonstrate the best stress forces distribution between the laponite and the polymer matrix. It is presumed that the laponite morphology can intervene in the increase of energy dissipation because this nanoclay has the highest loss factors.

Finally, by comparing the values of the results obtained from nanostructured materials, it is noted in Fig. 2 that the dynamic stiffness values are in some cases comparable to those of common sound insulation materials such as glass and mineral wool with thickness of 13 mm and 33 mm respectively, but requiring much smaller thicknesses.

![Figure 2: Test results of dynamic stiffness for nanostructured samples composed of 10 layers. The lines show the value of dynamic stiffness for glass wool with thickness 13 mm and mineral wool with 30 mm.](image)

By linking the high loss factor values with lower values of E (implying a low dynamic stiffness) will allow one to obtain a material with good impact sound reduction properties. In all materials, the amount of NP is proportional to E and the loss factor. Therefore, if there is a change in the amount of NP in the polymer matrix this will change both its loss factor η and their respective dynamic stiffness.

It has been shown [12,13] that the index of impact sound reduction \( \Delta L_{\text{Wt}} \), in dB, for a floating slab of mass per unit area \( m \) (kg/m\(^2\)) which is mounted on a resilient layer of dynamic stiffness \( s \) (MN/m\(^3\)), can be estimated by the equation

\[
\Delta L_{\text{Wt}} = 10 \log \left( \frac{m}{s} \right)
\]
\[ \Delta L_W = 18 + 15 \log \frac{m}{s} \]  

(1)

Table 2 reports the results of the index of impact sound reduction of a 6 cm thick homogenous concrete (144 kg/m²) floating slab with 10 elastic nanostructured layers. The layers are made up of the nanostructured polymer matrix with added laponite nanoclay for 5% m/m and 10% m/m concentrations. The results are compared with those obtained using typical glass and mineral wool [14].

For the particular case of laponite, dynamic stiffness is less than that of the polymer matrix for samples with NP ≥ 5%, which shows that although there is a high E, the loss factor indicates that nanoparticles are involved in the dissipation of vibration energy more efficiently. This is observed with the reduction in dynamic stiffness and, consequently, in increasing \( \Delta L_W \). We notice that in the case of laponite with 5% and 10% m/m concentration, the index of impact sound reduction of this 3.5 mm thick layer is 5.6 dB and 8.5 dB greater, respectively, than that of a 30 mm thick mineral wool [14].

| Material            | Density (kg/m²) | Thickness (mm) | Dynamic stiffness (MN/m³) | \( \Delta L_W \) (dB) |
|---------------------|----------------|----------------|--------------------------|----------------------|
| Laponite (5% m/m)   | 780            | 3.5            | 8                        | 36.8                 |
| Laponite (10% m/m)  | 784            | 3.5            | 5.1                      | 39.7                 |
| Cloisite 30B (5% m/m) | 1433       | 3.5            | 22.6                     | 30.0                 |
| Cloisite 30B (10% m/m) | 871        | 3.5            | 18.8                     | 31.3                 |
| Glass wool          | 36             | 13             | 28                       | 28.7                 |
| Mineral wool        | 140            | 30             | 19                       | 31.2                 |

4. Conclusions
In this study, the effects of nanostructures cloisite 15A, cloisite 30B, hectorite, and laponite on a matrix of thermostable polyurethane with various concentrations of nanostructures were determined. Based on the experimental results, it can be concluded from the mechanical characterization that the loss factor and Young’s modulus increase with the amount of nanoparticles added. Furthermore, it was found that there is a strong dependence on the type of nanoparticle added to the polymer matrix.

Material characterization by ISO 9052-1 pointed out that materials may be used as components in damping applications when percentages of NP are higher than 5% m/m. The lowest dynamic stiffness found was for laponite with 10% m/m concentration, with a reduction in stiffness of 72% when compared to the polymer matrix without nanostructures.

The index of impact sound reduction of a floating slab with a viscoelastic layer was also determined from the dynamic stiffness values, and the index of impact noise reduction for thin films of 3.5 mm thickness was obtained. Experimental results obtained for Laponite and cloisite 30B at higher proportions than 5% m/m have indicated that these materials can be used as viscoelastic layers for construction of floating slabs. These results suggest that the use of nanostructured materials may be an alternative to the use of common commercial materials and, in some cases, enhance the impact noise isolation with lower thicknesses.

Further work is planned using SEM to study other parameters of the nanostructured materials, such as their morphology and NP distribution, in order to better explain the physics behind the results presented here.

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