Application of dynamic mechanical analysis for the study of viscoelastic properties and identification of softwood and hardwood species

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Abstract. Based on experimental studies using dynamic mechanical analysis (DMA) methods, the storage modulus E', loss modulus E", and mechanical loss coefficient tan δ were determined. A comparative characterization of static and dynamic viscoelastic-plastic properties of softwood (European spruce – Picea abies) and hardwood (English oak - Quercus robur) species was provided, depending on the frequency and amplitude of probing harmonic oscillations, to determine the possibility of their identification. The contribution of various frequencies and amplitudes of harmonic oscillations to the formation of the total viscoelastic-plastic response of softwood and hardwood species was determined, and the effect of additional oscillating load on the dynamic parameters of wood was evaluated. The fundamental possibility of using dynamic viscoelastic property parameters for the identification of softwood and hardwood species was demonstrated.

1 Introduction

It is known that wood is a complexly structured, highly porous, fibrous natural polymer material that is widely used in various spheres of practical activity. Wood is used as construction and finishing materials, materials for making musical instruments, packaging, and other applications [1-3]. The key components of wood of all species, determining its basic structure and properties, are the most common natural polymer - cellulose, as well as hemicellulose, lignin, pectin, and water [4]. In this hierarchy, it is customary to distinguish several structural levels - atomic-molecular [5], nanolevel (nanocrystals, nanofibrils) [6], microlevel (microfibers, cell walls) [7], mesolevel (cells, large vessels, radial rays, etc.) [8], and macrolevel (annual rings, macroscopic structural defects, cracks, etc.) [9]. These are what form all the diversity and uniqueness of practically important wood properties [10]. For example, the fundamental differences between the macroscopic structure of softwood and hardwood species are directly related to the different types of cells in these two groups [8, 11-12]. The recent increased interest in the practical use of wood stimulates more intensive study of its structure and properties at different scale levels, applying not only optical microscopy methods, but also other modern materials science methods: scanning and

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transmission electron microscopy [13], near-IR spectroscopy [14], Raman spectroscopy [15], X-ray diffractometry and tomography [16], nanoindentation [17-18], scratching, and others.

This made it possible to obtain much detailed information about the macro-, micro-, and nanoscale features of wood. At the same time, significantly less attention has been paid to experimental studies of the viscoelastic-plastic behavior of wood. However, it is these properties of wood that determine its creep and relaxation, which depend on time, temperature, and humidity [19-20]. Usually, to solve problems of studying the time-dependent behavior of materials in physical materials science, impedance methods are used, such as internal friction and related dynamic mechanical analysis (DMA), non-contact atomic force microscopy methods, dynamic NI (CSM) modes, etc. [23-24]. Common to these methods is the registration of the response to the combined action of the main quasi-static load causing one of the types of deformation (tension, compression, or bending) in the material and additional low-amplitude probing harmonic oscillations [23-25]. Considering that the key components of wood, such as cellulose, hemicellulose, and lignin, are polymeric viscoelastic materials, the use of the DMA method for characterizing the time-dependent properties of wood can be considered a natural choice [26, 27]. At the same time, despite the widespread use and long history of studying wood properties, literature data on the application of impedance methods for its study remain very limited [2], and information on the use of DMA methodology for wood identification is practically absent.

In this regard, the aim of the work was to determine the dynamic viscoelastic-plastic characteristics of wood depending on the frequency and amplitude of probing harmonic oscillations using typical softwood (European spruce) and hardwood (English oak) tree species as examples.

2 Materials and methods

The following objects were selected for the study:

a) Hardwood species: English oak (*Quercus robur*) 90 years old;
b) Softwood species: European spruce (*Piceaabies*) 90 years old.

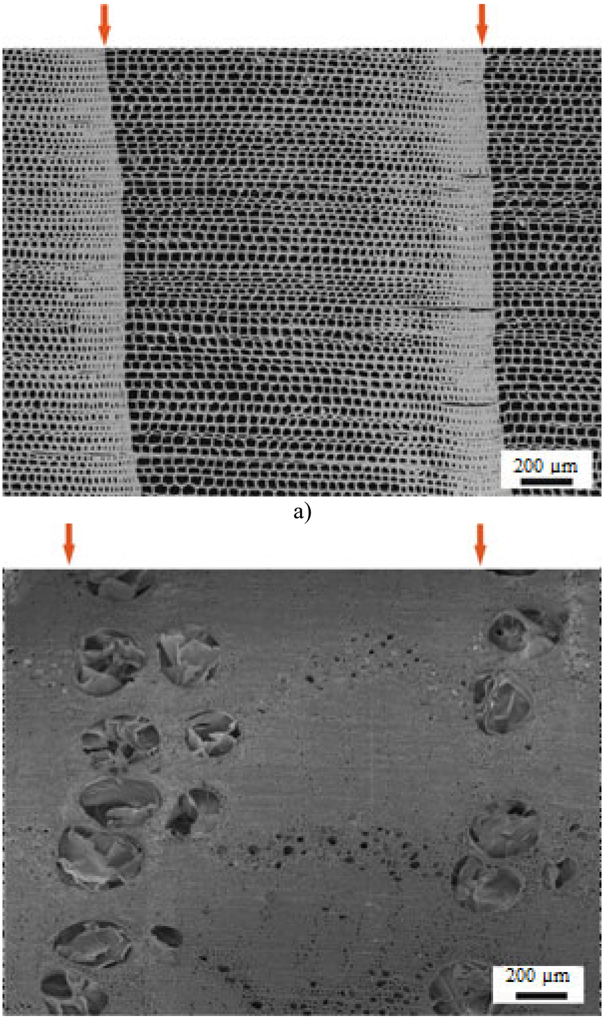
Oak and spruce samples were obtained in 2020 from tree cuts from the Vernadsky forestry in the Tambov region (Tsninsky forest area).

For the research, samples measuring 50×10×2 mm were cut from cross-sections of wood, pre-dried to 8% moisture content, which were then subjected to mechanical grinding and polishing using a grinding and polishing complex (Buhler, USA). To assess the degree of sample processing, a diInnova SPM scanning probe microscope (Veeco-Digital Instruments, USA) was used. The resulting roughness values Ra for spruce wood samples did not exceed 250...300 nm, and for oak wood - 140...180 nm. A Tescan Vega 3 scanning electron microscope (SEM) (Tescan, Czech Republic) was used to analyze the wood microstructure. For this, additional sample preparation was carried out by cross-sectioning the surface layer of wood a few tens of micrometers thick using a Slide 4004M PFM sledge microtome (Pfmmedical, Germany).

To determine the viscoelastic properties of wood, a DMS 6100 dynamic mechanical spectrometer (SII Nano Technology Inc., Japan) was used, allowing research in a wide range of frequencies f (from 0.01 to 200 Hz) and amplitudes A (from 5 to 100 μm) of probing oscillations. Sample deformation was carried out by the three-point bending method with the application of a peak bending force $P_{\max}$= 8 N. To measure the frequency dependences of the elastic modulus (or storage modulus) E', loss modulus E'', and mechanical loss coefficient tan δ, along with the stationary load $P_{\max}$, probing oscillating vibrations with an amplitude in the range of 5...100 μm were applied to the sample. The share of the oscillating load did not exceed 1% in relation to the quasi-static load. The software allows extracting the values of the complex modulus $E^* = E'+iE''= \frac{\sigma_0}{\varepsilon}(cos\varphi + isin\varphi)$, and its components - E' and E'', as well as tgδ = E''/E'. Here $\sigma_0$is the amplitude of the probing stress, $\varepsilon_0$is the amplitude of strain oscillations, φ is the phase shift angle between the vectors of oscillating stress and strain in complex form.

3 Results and discussion

The characteristic cellular structure of the studied samples is shown in Figure 1. The obtained SEM images clearly show the typical structure of spruce wood (Figure 1(a)) and oak wood (Figure 1(b)) with a well-defined structure of annual rings, zones of early and late wood within an individual annual ring, as well as the cellular structure of the tree.



a)

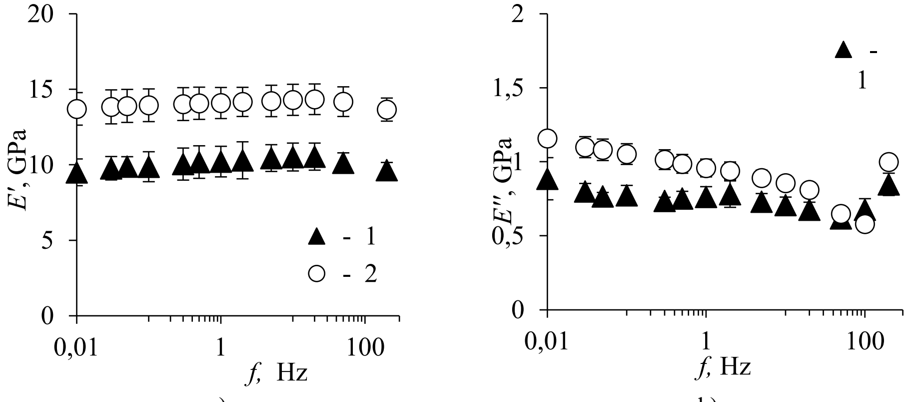
b)

Fig. 1. SEM image of the cellular structure of annual rings of wood of Norway spruce (a) and English oak (b). The arrows indicate the positions of the boundaries of the annual rings.

The conducted studies of viscoelastic properties allowed constructing dependencies of the elastic modulus E′on the frequency f of probing oscillations for softwood (1) and hardwood (2) species, at a constant amplitude of probing oscillations (A = 5 μm) (see Figure 2(a)). Analysis of the obtained data shows that in the studied frequency range, the value of E′ tends to increase weakly and monotonically with increasing frequency (in the frequency range from 0.01 to 20 Hz), after which a decreasing trend is observed, both for softwood and hardwood. At the same time, hardwood (using the example of English oak) is characterized by higher viscosity and elasticity (the characteristic value of E′for oak in the studied frequency range is 13.3 ± 0.2 GPa, while for spruce softwood it is 9.1 ± 0.15 GPa, see Figure 2(a)).

The change in the frequency of harmonic oscillations has a significantly greater effect on the mechanical loss modulus *E″*, especially noticeable for oak wood (Figure 2(b)). The lowest losses occur in spruce wood, which belongs to the softwood species with low density. In general, the loss (dissipation) modulus determined by DMA methods is responsible for the potential ability of a material to accumulate energy (due to recoverable deformation) and dissipate it (through the interaction of molecular chains, for example, in polymeric materials, due to internal friction and chain restructuring as a result of deformation).

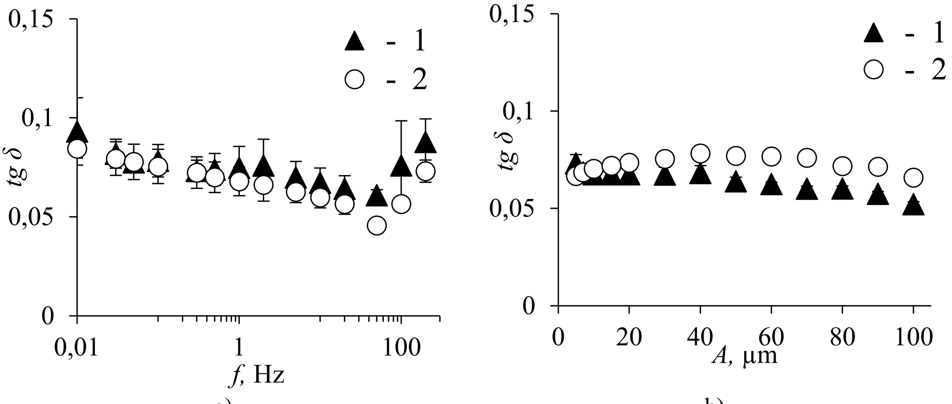
Analysis of the frequency dependencies of *E″* shows that English oak wood has greater nonlinearity among the studied wood species (see Figure 2(b)). Usually, a decrease in the loss modulus with an increase in the oscillation frequency of the load indicates a deterioration in the damping ability of the studied material with increasing frequency. However, for frequencies f > 66.6 Hz, the decrease in both *E″* and tgδis replaced by their rapid growth. This increase may be associated with localized softening in the material structure (and in the case of wood, this may be due to localized softening in the structure of amorphous polymers that are part of its composition) due to the greater amount of energy dissipated at these frequencies. Simultaneously, an increase in free volume due to increased energy dissipation in this frequency range can contribute to increased mobility of side chains, leading to a more viscous surface response.



a) b)

Fig. 2. Dependence of the modulus of elasticity*E′*(a) and loss module *E″* (b) on the frequency *f* of probing vibrations for coniferous – European spruce (1) and deciduous – common oak (2) tree species, with a constant amplitude of probing vibrations A (A = 5 μm).

Figure 3(a) shows the dependence of the mechanical loss coefficient tgδ on the frequency f of probing oscillations for softwood (1) and hardwood (2) species, under the condition of constant amplitude of probing oscillations (A = 5 μm). The figure shows that both types of wood are characterized by a quasi-linear decrease in tgδ as the frequency increases up to f ≈ 66 Hz, above which there is a tendency for the obtained data to increase. At the same time, in the entire remaining frequency range of 0.01–66.66 Hz, the data on the mechanical loss coefficient give a smooth curve that agrees well with the expectations of the behavior of a bulk, isotropic, homogeneous viscoelastic solid. In other words, up to this critical frequency, these data are in good agreement with the assumptions of linear viscoelasticity of the material.



a) b)

Fig. 3. Dependence of the coefficient of mechanical losses tgδ on the frequency *f* (a)and amplitude *A* (b) sounding vibrations for coniferous – European spruce (1) and deciduous – English oak (2) tree species.

Characteristic dependencies of the mechanical loss coefficient tgδ on the amplitude *A* of probing oscillations for the studied wood samples, at *f* = *const (f* = 2 Hz), are shown in Figure 3(b). The figure demonstrates that the tgδ value of the denser oak wood changes sequentially depending on the amplitude of probing oscillations, from small *A* values (oak has the smallest tgδ value among hardwoods) to *A* = 10-20 μm, at which it becomes comparable to the mechanical loss coefficient of spruce, and even exceeds it at the maximum possible amplitude *A*. Increasing the amplitude *A* of probing oscillations for softwood and hardwood species at a constant frequency of probing oscillations (*f* = 2 Hz) is accompanied by an expansion in depth of the probing oscillations' influence on the internal structure of the wood. The viscoelastic behavior of wood at different depths from the surface may vary. It is known that, for example, in polymers, a significantly more viscous, i.e., higher, mechanical loss coefficient was obtained with larger amplitude effects on the material. The observed increase in the loss modulus E'' and the mechanical loss coefficient tgδ with frequency (in the high-frequency range, at $f > 50$ Hz) indicates a noticeable improvement in the damping ability of wood polymers at these frequencies.

4 Conclusion

Using dynamic mechanical analysis (DMA) methods, the viscoelastic properties of softwood (European spruce) and hardwood (English oak) were studied. A comparison of the viscoelastic characteristics of softwood and hardwood species (using spruce and oak wood as examples) was made depending on the frequency and amplitude of probing harmonic oscillations to determine the possibility of identifying wood species using DMA methods.

As a result of the conducted research, it was established as follows.

The modulus of elasticity tends to increase weakly and monotonically with increasing frequency across the entire range of probing frequencies (from 0.01 to 20 Hz), after which a decrease is observed for both softwood and hardwood. Quantitatively, the modulus E' of hardwood is 3 to 5 GPa higher than that of softwood.

The damping capacity of hardwood species (in the studied frequency range) deteriorates with increasing frequency of probing oscillations; however, for frequencies $f > 66.6$, following the drop in E'' and tgδ, there is a rapid increase. The loss modulus of softwood decreases much weaklier in the same frequency range. The observed increase in the loss modulus E'' at frequencies above 100 Hz indicates a noticeable improvement in the damping capacity of wood species with increasing frequency.

The data obtained in this study indicate the fundamental possibility of applying DMA methods for the identification of various wood species through comparative testing.

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