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Test method

Extension and application of dynamic mechanical analysis for the estimation of spatial distribution of material properties

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ABSTRACT

The paper presents a unique extension and application of the dynamic mechanical analysis (DMA) technique for the determination of heterogeneous mechanical material properties. Their values as well as their spatial distribution are major interest when analysing properties of post-damage materials. The proposed procedure incorporates longer than typical beam-shaped test samples enclosing the zone with locally varied material properties. The mechanical properties are estimated for the extended sample at defined positions while the sample is iteratively shifted along its axis. The procedure was successfully validated in various tests incorporating standardised and extended samples.

The preliminary investigations regarding the application of the proposed procedure were conducted on composite specimens with heterogeneous distribution of material properties introduced by impact load. The results show, on the one hand, a monotonic relation between the storage modulus and the damage extent. On the other hand, a non-monotonic relation between the loss modulus and damage severity has been observed.

1. Introduction

The increasing demand for high performance products with a simultaneous requirement for low price has stimulated the development of numerous efficient simulation techniques [1,2]. This can be accounted on a significant price difference—due to time and material savings—between physical and virtual experiments. Nevertheless, in order to provide any authoritative simulated results, accurate input information of the material parameters remains unavoidable [3–5]. Hence, reliable characterisation techniques for their determination have to be provided. One of many well-established methods that enable accurate quantification of material thermo-mechanical properties is dynamic mechanical analysis (DMA) [6].

In DMA, a small volume material sample—typically in a form of beam with rectangular cross-section—is fixed in the analyser's clamps. The sample is then excited to defined vibrations either with controlled force or displacement. Based on the sequences of excitation and response (v. Fig. 1), the material mechanical properties viz. loss and storage moduli can be estimated. Since DMA is a laboratory test, i.e. conducted under fully controlled conditions, the results are normally very accurate.

1.1. Motivation

A fundamental assumption of typical DMA is a homogeneous distribution of material properties within the sample. However, in specific cases the spatial distribution is of some concern, e.g. while analysing properties of a locally damaged material [7–9]. Since this vital issue has not been addressed until now, an attempt to develop an extended DMA procedure that combines the advantages of conventional DMA i.e. accuracy and the small required material volume with the possibility of spatial distribution characterisation of material properties is presented in this paper.

1.2. Determination of material properties in the dynamic mechanical analysis

In DMA, the phase lag δ_G between the amplitudes of the oscillating excitation force F and the resulting deflection d (or vice versa) (cf. Fig. 1**A**) is analysed in order to calculate the mechanical material properties. To determine the storage E' and loss E'' moduli from the sequences of force and deflection, first the complex stiffness k^* is calculated as [10]:

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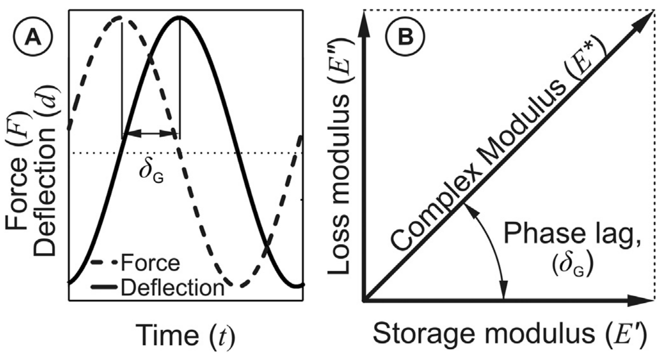

Fig. 1. The schematic principle of material properties determination in the dynamic mechanical analysis. **(A)** The force (excitation) and deflection (response) sequences as recorded by dynamic mechanical analyser. **(B)** The vectorial representation of the complex moduli describing the mechanical material properties.

$$k^* = \frac{F}{d} \tag{1}$$

Taking into account the calculated value of complex stiffness, the complex modulus E^* can then be determined as:

$$E^* = k^* c_g \tag{2}$$

where c_g is a geometrical correction factor specific for every deformation mode, e.g. for the dual cantilever deformation mode c_g is equal to:

$$c_g = \frac{1}{c_c}\left[\frac{l^3}{192\, I} + S(1+\nu)\frac{l}{2A}\right] \tag{3}$$

where c_c is the clamp-specific correction factor (approx. 0.9); l is the length of the sample between the fixed clamps in m; I is the area moment of inertia in m^4; S is the shear correction factor (typically approx. 1.5); ν is the Poisson's ratio; and A is the area of cross-section normal to the beam's axis in m^2.

Based on the calculated E^* and measured phase lag δ_G between the force and deflection, the storage and loss moduli can be determined as:

$$E' = E^* \cos \delta_G \tag{4}$$

and

$$E'' = E^* \sin \delta_G \tag{5}$$

It is important to emphasise that the complex modulus is estimated based on two semi-empirically defined factors, namely clamp-specific factor and shear correction factor as well as on assumption of rigid clamps. Nevertheless, a comparison between material properties obtained within the same DMA configuration is reliable.

2. Experimental procedure

The main objective of the study was to determine the post-impact spatial distribution of composite material properties using an adapted DMA technique. In the proposed procedure, a commercially available dynamic mechanical analyser, Type Q800, produced by TA-Instruments, was used. A detailed procedure description is presented in the following section.

2.1. Phenomenological description

The proposed procedure incorporates a longer than typical test sample that includes the zone to be investigated. An extended sample is iteratively shifted along its axis while the mechanical properties are being estimated for each sample position. In order to avoid a position-dependent influence of the sample's free ends on the obtained results, a dynamic mechanical analyser with double cantilever deflection mode clamps was used (Fig. 2). It was assumed that its fixing clamps, denoted as (1) in (Fig. 2), block all degrees of freedom, and hence the parasitic oscillations of the sample's free ends could only result from their finite stiffness.

The material properties are quantified at every position of the sample as an average of the properties in the region enclosed by the DMA clamps (v. Fig. 2 region between the clamps 1). Through the successive shifting of the sample, the distribution of the material properties along the sample's length can be estimated.

The proposed procedure can be structured into six steps:

1 Preparation of the sample enclosing the interesting zone,
2 Clamping the sample in such a way that the zone of interest is fully outside one of the fixing clamps (Fig. 3**A**),
3 Conducting a single DMA test,
4 Shifting the sample by a defined distance d_s along its axis (Fig. 3**B**),
5 Iterative conduction of steps 3 and 4 so that the focused region moves step by step through the measuring zone until it is outside the other fixing clamp (Fig. 3**C** and **D**),
6 Post-test analysis of the results, e.g. involving their correction according to the aspects of digital filter theory described in Sec. 5 or graphical visualisation of acquired material properties.

Such an extended procedure enables a locally resolved estimation of the mechanical material properties using a standard DMA apparatus. A limitation in comparison to the usual test procedure results merely from the increased sample size which could hinder an application of the integrated oven. Without utilization of the oven, the investigations regarding the temperature-dependence of mechanical material properties cannot be conducted. This would require either some hardware modifications to the analyser or the use of a similar device with sufficiently large temperature controlled chamber.

3. Procedure validation

In order to gauge the accuracy of the altered determination procedure, an uncertainty budget was identified. The sources of inaccuracy in the DMA can be tracked down to the following

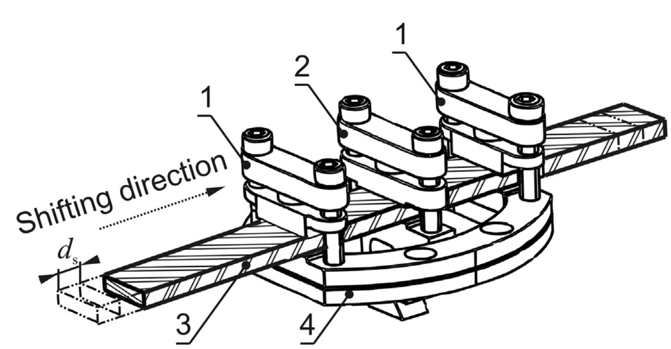

Fig. 2. Dynamic mechanical analyser with installed double cantilever clamps. 1—Fixing clamps, 2—Driven clamp, 3—Analysed extended sample, 4—Support. ds —shifting distance.