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Effective use of transient vibration damping results for non-destructive measurements of fibre-matrix adhesion of fibre-reinforced flax and carbon composites

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ABSTRACT

Fibre-matrix adhesion affects fibre-reinforced composites' mechanical properties, a process which can be improved by applying appropriate sizing on the fibre. Transverse bending tests and Scanning Electron Microscopy (SEM) can help quantify this effect. This paper investigates if modal damping measurements are a reliable alternative for quantifying fibre-matrix adhesion. When a composite sample is vibrating, part of the dissipated energy is due to the internal friction. More internal friction and slipping at the fibre-matrix interface is expected with a weaker fibre-matrix bond, hence increasing the amount of dissipated energy, which in turn is proportional to the modal damping value. This paper researches two different cases to validate this hypothesis. In the first case, we will use two composite samples of flax fibre, one with and one without sizing. In the second case, we will compare flax and carbon fibre laminates. If the only variable is fibre sizing, better adhesion is related to significantly lower damping and higher resonance frequencies. If composite laminates with different fibre and matrix type are compared, lower adhesion is not necessarily related to increased damping and lower resonance frequencies. However, when combining the damping result with SEM microscopy, it is possible to assess the relative contribution to the internal energy dissipation of the fibre, the matrix and the fibre-matrix interface individually.

1. Introduction

In fibre-reinforced composites, mechanical properties do not only depend on the properties of the separate constituents and their respective fraction of the composite material, but also on how both the fibre and the matrix material interact with each other. This measure for how the matrix binds itself to the fibres is known as interfacial adhesion. Generally, fibre surface treatments have been shown to improve interface adhesion compared to their neat-fibre surface. Different measurement techniques exist to evaluate the fibre-matrix interface. These mechanical tests can be subdivided into two main categories, (i) single-fibre tests and (ii) tests on composite specimens [1]. Within the latter category, transverse bending tests and Scanning Electron Microscopy (SEM) are often used to measure fibre-matrix adhesion quality. In the transverse bending test, it is mainly fibre-matrix adhesion and composite laminate fibre volume fraction that determine mechanical stiffness and strength [2–4]. This is independent of fibre mechanical properties. With the transverse bending test, maximum strain is located at the outer layers of the specimen so

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that only a small volume is loaded with critical strain. As such, fracture probability due to voids and other defects in the material is minimized. Because of this, the transverse bending test is preferred to a transverse tensile test because the latter has a uniform strain distribution [2]. SEM microscopy is used for qualitative interpretation of the interface's quality. With a weak interfacial bond, fibres and resin are separated easily from each other, resulting in a fibre surface that is almost-resin free. In the case of a strong fibre-matrix bond, however, the matrix will be attached to the fibre. Strong adhesion is characterized by fracture across the fibre or the resin, whereas weak adhesion is characterized by a fracture at the fibre-matrix interface [5]. Measuring the material damping is a method to characterize the effect of modifications to the internal structure of the material [6–12]. As such, fibre-matrix adhesion also affects the specimen's dynamic properties. Friction and slip at the fibre-matrix interface increase in the case of a weak interface bond, leading to higher internal energy dissipation and, consequently, improved composite damping behaviour. In the case of a strong fibre-matrix bond, microscopic friction is minimal and results in lower damping capacity of the material. As an added bonus, modal damping tests are non-destructive, which is advantageous if the sample has to be used for other experiments.

The purpose of this paper is to examine whether modal damping measurements from transient vibration excitation are suited for detecting adhesion and fibre type effects in fibre-reinforced composites. This will be verified with results from transverse bending tests and SEM microscopy. Two different cases were evaluated. In the first case, two composite laminates were produced with the same manufacturing process. One sample was made with coated flax fibres for improved fibre-matrix adhesion whereas the other had neat, uncoated fibres. We will discuss if damping results can replace the two other methods for assessing interface quality. In the second case, composite laminates with flax and carbon fibre were investigated. These samples were made with pre-preg (pre-impregnated) fibre-matrix laminae. The damping results reflect the properties of the laminate itself, which is not necessarily related to the fibre-matrix adhesion only. We will, therefore, examine if the findings on damping measurements from the first case are still valid if two different composite materials are compared with each other. By combining three independent measuring techniques, we gain access to additional information that can help determine the contribution of the fibre, the matrix and fibre-matrix adhesion to the composite damping.

This research is limited to investigating the fibre-matrix interfacial properties of plate specimens with rectangular or square geometry. This is required prior to drawing conclusions if the modal damping technique could also be applied to actual geometries and sizes. If so, this technique could be used as an NDT method to detect the fibre-matrix interfacial properties. However, fibre-matrix adhesion is not the sole damping parameter, but also other influences such as boundary conditions, exact excitation method, local variations in part geometry and thickness, play an equally important role, and are very difficult to filter out when using it as an NDT method for real structures.

2. Sample preparation

Fibre-matrix adhesion was characterized by different composite materials and production techniques, the manufactured composite laminates had a unidirectional (UD) fibre direction.

Sample preparation with the RTM (vacuum-assisted resin transfer moulding) production process allows the production of composite laminates with neat and treated flax fibres. The flax fibre material was provided by Lineo and has a density of 150 g/m^2. The treated flax fibre was, in contrast to the neat flax fibre, coated for improved fibre-matrix adhesion and to prevent water absorption. The RTM process also makes it possible to produce neat epoxy samples, which will later be used for comparison with the composite laminates. For this production technique, the combination of epoxy resin RIMR 135 and hardener RIMH 137 is ideal.

Composite laminates with flax fibre and carbon fibre were compared with each other based on samples made with the autoclave process. The flax fibre pre-preg material was also available from Lineo, with the same fibre density and coating as with the RTM manufacturing process. The epoxy resin was Araldite LY5150, Young's modulus is 3.52 GPa and the tensile strength is 68–78 MPa, corresponding with a maximum strain of 2–3%, according to the manufacturer's specifications. The carbon fibre pre-preg material consists of M55J carbon fibres and the M18 epoxy resin. The epoxy has a Young's modulus of 3.50 GPa, a tensile strength of 81.1 MPa and failure strain of 3.7%, as specified by the manufacturer. The carbon fibre surface was treated with sizing 50B from Toray to improve the fibre-matrix adhesion.

Table 1 lists the mechanical properties of the different laminate types. These properties were assessed from tests according to the standards ISO 527-4 and ISO 14129. In the case of plane stress analysis, four engineering constants are sufficient to describe the composite material's mechanical behaviour: Young's modulus in the 1- and 2-direction E_{11} and E_{22} (cf. Fig. 2), the Poisson's Ratio ν_{12}, and the shear modulus G_{12}.

3. Experimental test procedure

To verify if modal damping measurements are suitable for determining the influence of the fibre-matrix adhesion and the fibre type, it was necessary to have a reliable reference that can be related directly to the fibre-matrix adhesion. Therefore, our analysis procedure first evaluates the results from the transverse bending test and the SEM method. These results will then be used for comparison with the modal damping results.

The sample thickness was 2 mm for all tests. The width and the height depends on the selected test method, which will be discussed in the following sections. The samples for the different test methods were cut out from the same base material plate to eliminate scatter in the results due to small variances in the production process.

3.1. Transverse bending test

With the transverse three-point bending test, the composite laminate was positioned as shown in Fig. 1. Direction 1 is parallel with the fibres, direction 2 is perpendicular to the fibres in the laminate's plane, and direction 3 is perpendicular to the fibres and the plane of the laminate. The sample used two supports at distance l_S and the force was applied in the 3-direction in the middle of the specimen. The plane defined by the 2–3 directions is the translaminar view of the specimen, the plane defined by the 1–3 direction is known as the interlaminar surface. The tests were executed according to ISO 14125. The sample thickness t was 2 mm, the width w was 12.7 mm and the length l was 100 mm. The support length l_S was 32 mm, the overlap at each support should be at least 10% of the support length with a minimum of 6.4 mm. Prior to testing, it was necessary to polish the translaminar surface for better quality of the microscopic analysis (Section 4.1.2 and 4.2.2). The finest grain size applied was #4000. It should be noted that quantitative interpretation with bending tests is difficult. The friction at the supports significantly contributes to the result and the slope at the contact points is possibly too large for correct use of linear elastic theory of bending [13]. Consequently, the Young's