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USE OF NON-DESTRUCTIVE TESTING (NDT) METHODS FOR THE CHARACTERIZATION OF NOVEL SUSTAINABLE COMPOSITES AND JOINTS IN RENEWABLE RAW MATERIALS

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Abstract. *Due to increasing demands on sustainability, manifold efforts are made to reduce CO_2 emission. Renewable raw materials have even a negative CO_2 balance so that their use can be very valuable e. g. as construction material or in architecture not least for optical reasons. New material concepts with raw materials, that are currently under development, are presented. The raw materials wood and bamboo, as well as composites of both, additionally with glass fibre, are described in such a way that important aspects and special requirements for the selection of a suitable non-destructive testing method can be identified. Every later production process must be monitored regarding to the quality. The use of NDT methods offers special opportunities in terms of reliable quality assurance and cost savings. NDT methods with particular potential as a testing technique are selected. These include advanced NDT methods like radiographic computed tomography, ultrasonic testing, microwave or terahertz testing as well as thermography, each with their own aspects, strengths and limitations. The focus is on understanding the specific demands of the application and selecting appropriate NDT. Factors such as material composition, joint geometries, testing configurations and environmental conditions are taken into consideration. Assessments about the testability of typical testing tasks are discussed by means of transferable examples as close to the application as possible. First results of practical experiments with passive and active thermographic NDT methods were shown, evaluated and further proceeding for development is outlined. The shown results provide the basis for further exploration, the findings help to reduce the effort for introduction of NDT for renewable materials and composites.*

Keywords: *Non-destructive testing (NDT), renewable materials (wood, bamboo), sustainable composites, reducing CO_2 emissions, joining technologies, friction welds, materials characterization, failure detection, thermography*

1. INTRODUCTION

The emerging challenges brought about by sustainability requirements are leading to new applications of renewable resources. Unlike most materials used for recent products, they have the great advantage not to consume CO_2 but store it and thus even have negative CO_2 balances. Manifold trends exist for example for highly advanced construction materials like lightweight car body components made from bio-based composites in automobile industry (Hansen et al., 2020), or a lot of activity is done to improve the capabilities of building materials in the construction industry (BuFAS, 2018), accompanied by new concepts that could be attractive in architecture, not least for visual reasons. New material concepts always require joining technologies to be able to produce sustainable components like sandwich materials, constructions, etc., so that current development also focus to this field of research (Loth and Förster, 2022; Förster, 2020; Ziani et al., 2019).

At the same time as new applications together with their production processes are developed, appropriate testing techniques will be important to monitor the production of new components and new composites materials. Due to great advantages of non-destructive testing (NDT) methods for the quality control in later production, the aim of following investigations is to identify potential NDT methods that enable the characterization of sustainable, renewable raw materials and composites including joint properties. Starting with a brief insight into new material concepts and related joining technology development, suitable NDT methods for later quality control shell be identified, specific challenges and chances will be analysed in order to reduce the effort for later introduction of NDT.

2. SUSTAINABLE MATERIAL AND JOINING CONCEPTS

The material and joining concepts considered in this paper consist of renewable raw materials like wood, bamboo or a combination of both or with glass fibre reinforced plastic. Partly used adhesives also are mentioned. The materials will be briefly described and an overview of new composite and joining concepts are given, focusing on relevant aspects for later NDT applications.

2.1 Reniewable Raw Materials

Wood is an organic material consisting of cells and chemical compounds such as cellulose, hemicellulose and lignin. The cell wall is composed of many layers: Microfibrils in a lignin matrix glued together by polyoses. The microfibrils make up approx. 40-50 % of the wood mass and are oriented roughly in the direction of the fibres. They are responsible for the very high tensile strength of more than 1000 N/mm^2. The lignin content is approx. 25-30 % and fills the cavities between the fibrils, acting as a supporting material and providing the compressive strength, which is increased by the layered cell wall structure (Bühler, 2008).

Fig. 1 shows the inner structure of frequently used wood species softwood spruce and hardwood oak. The illustration exemplifies the large differences in structure between different types of wood. The annual rings shown for pine can be found in general, they complement the foregoing statement for wooden materials.

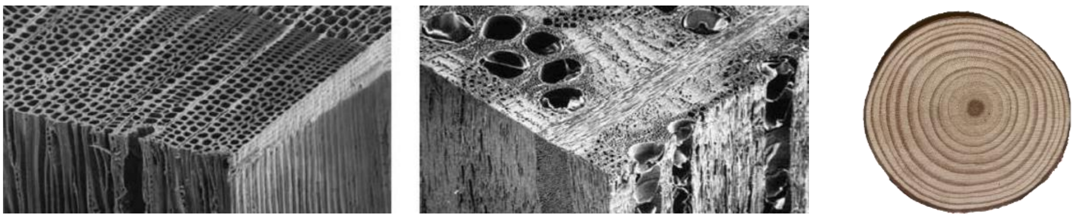


Figure 1. Inner structure of softwood (left) and hardwood (middle) (Bühler, 2008), tree slice with annual rings (right).

Bamboo is a fast-growing plant of the grass family with wood-like properties. There are over 1250 species worldwide with different properties. The anatomical structure of bamboo appears relatively uniform compared to wood. The bamboo culm consists of nodes and internodes, the intermediate sections. The thin partitions in the nodes are called diaphragms. The lengths of the internodes as well as the thickness of the culm walls differ considerably between the different bamboo species. Fig. 2 shows the wide variety of different bamboo kinds, the culm arrangement with nodes and internodes and an microscopic intersection, illustrating the inner structure in an internode section. The bamboo culm consists of approx. 50 % parenchyma cells, 40 % fibres and 10 % vascular bundles. The cross-section shows the dark vascular bundles with their fibre agglomerates in the parenchyma tissue. All bamboo species show an analogous pattern in the distribution of their cells. The advantage of bamboo is its great hardness, but the origin, moisture content, age and diameter of the cane have a great influence on the material properties (Liese and Köhl, 2015; Minke, 2023).

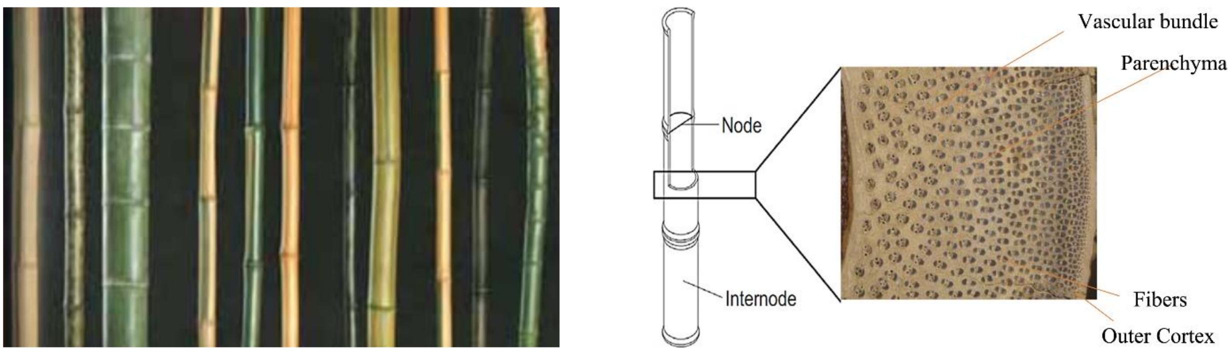


Figure 2. Bamboo variety (left) (Tönges, 2002) and culm structure (right) (Goh et. al., 2019 apud Correal, 2016).

Glas fibre reinforced plastic (GFRP) in detail is a composite, but in this paper it is simplified as one material component. It consists of a plastic matrix (polyester or epoxy resin) and glass fibres of different sizes, from approx. 1 mm up to more than 50 mm length. The glass fibres serve as reinforcement and give the material high strength and stiffness combined with low weight. The material is also characterised by its corrosion resistance and good thermal insulation. Due to its mouldability it can be brought into different shapes and enables complex designs. GFRP is used in many areas, including vehicle construction, aerospace, boat building, the construction industry and sports equipment manufacturing.

Adhesives glue components to each other in a materially bonded manner without the need for further mechanical fasteners. The interaction of surface adhesion and the internal strength of the adhesive (cohesion) results in a force-transmitting effect. The structure of the parts to be joined remains essentially unchanged. Depending on the use of the respective wood material, different types of adhesives are used. In general, adhesives consist of non-liquid components such as binders, fillers and extenders, and volatile components such as solvents, thinners and dispersants. Adhesives are classified into physically setting and chemically reacting adhesives (Wagenführ and Scholz, 2018). The adhesive used was a glue on a 1-component polyurethane basis OTTOCOLL® P84 (Otto, 2022).

2.2 New Composite and Joining Concepts

Composites of renewable materials are both already widely used as for example building materials as well as they are object of research activities due to their excellent eco balance or opportunities for lightweight construction.

Cross laminated timber (CLT) is a widely used material for load-bearing constructions. It is a layered construction of solid wood boards in different orientations as shown in Fig. 3 schematically and as real object with for example 5 layers.

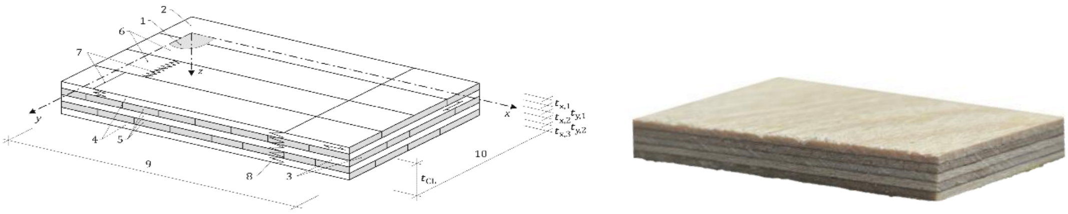


Figure 3. Structure of cross laminated timber (left) and a real sample with 5 layers (right) (DIN, 2021).

Comboo is a new type of sandwich composite structure with a bamboo-based core material consisting of a honeycomb-like arrangement of bamboo ring sections covered with various top and bottom layers. Fig. 4 shows the structure of a comboo composite, the inner bamboo comb layer and the section of a 3- or 11-layered comboo composite with two or ten cover layers of wood. This is called CCLT like comboo cross laminated timber.

Figure 4. Structure of central layer in CCLT (left) and 3- or 11-layered samples with cover layers of wood.

The use of different face layers thus results in stiffer or more ductile composites. Results of previous tests with the novel bamboo honeycomb arrangement show that very good mechanical properties can be achieved compared to conventional materials (Loth and Förster, 2023; Loth and Förster, 2022; Förster, 2020). Another composite that is recently under investigation consists of inner bamboo rings and GFRP outer layers (see Fig. 5). Since round structures can also be reproduced with this material and transparent structures are also possible, this material is already gaining a high level of attractiveness for decorative purposes.

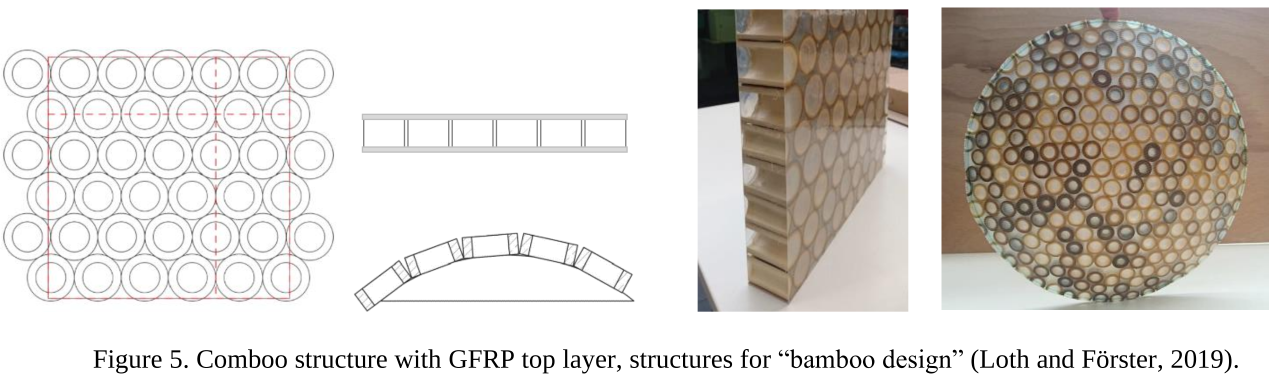


Figure 5. Comboo structure with GFRP top layer, structures for “bamboo design” (Loth and Förster, 2019).

Joints are important in order to be able to produce components, to built up structures, etc.. In this paper novel friction welded samples are considered as shown in Fig. 6 for wood and bamboo. For such samples initially determined toughness showed good results compared to ordinary connection types like glued joints. Currently, up to 60% of the final strength of wood-based materials is achieved by friction welding joints. The advantage of friction-welded joints is the absence of chemical, possibly toxic substances that make sustainable use difficult or impossible.

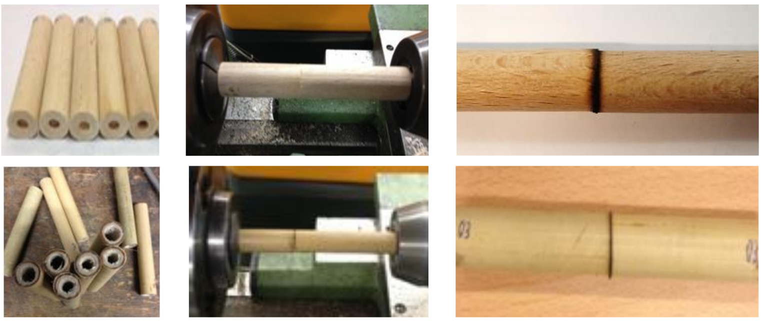


Figure 6. Friction welding on bars of wood (top) bamboo (bottom).

Due to the lignin present in both bamboo and wood, both bamboo and wood (different types such as ash, spruce, pine, beech or oak) can be joined together without adhesives. In order to be able to reliably assess the quality of the friction welded joint, 100% non-destructive material testing is needed, especially for safety-related applications.

2.3 Testing tasks for quality control

For the later use of the materials and joints that were shown, it is important that no defects impair the properties. Therefore, defects must be monitored in later production processes or during possible life cycles of e. g. load-bearing structures. For this purpose particular NDT applications are essential.

Specific defect patterns that influence mechanical properties, reduce strength, are shown in Fig. 7. For the different composites these are horizontal delamination from layer to layer and differently oriented cracks inside the layers.

Figure 7. Exemplary failure pattern of delamination and cracks for cross-laminated timber (left), comboo structures with wooden top layer (middle) and with GFRP top layer (right).

Fig. 8 shows cross-section surfaces of samples after tensile testing. Possible adhesion defects would arise in the fracture plane area, where the connection is partially interrupted.

Figure 8. Friction welded wooden bars (left), cross-section surface after tensile break of wood (middle), bamboo (right).

These specific joining compounds together with individual material properties of the renewable raw materials and composites are mostly new and unfamiliar compared to conventional ones, posing specific challenges for inspection.

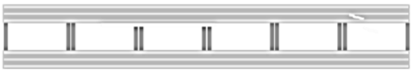
3. NDT TEST METHOD DEVELOPMENT

In order to find an appropriate NDT method, that has the potential to be used for later quality process control, at first, the NDT inspection task must be described in detail that are needed to evaluate the capabilities of potential NDT testing techniques. Most promising methods are selected, and similar applications will be presented and analysed to identify anticipated challenges.

3.1 NDT Inspection Task Definition

Typical inspection tasks for NDT were derived from the previous considerations and are shown in Tab. 1. Test geometries, materials or their combination, as well as related failure mechanisms or patterns are categorized into different types for initial analyses and investigations that will be relevant for the selection of a particular NDT method.

Table 1. Variants of inspection tasks.

	Geometry	Material(-combination)	Failures
1		wood	cracks
2		wood / wood + adhesive (cross-laminated timber)	delamination, cracks
3		wood / bamboo + adhesive (comboo)	delamination, cracks
4		cross-laminated wood / bamboo + adhesive (comboo)	delamination, cracks
5		glass fiber / bamboo (comboo)	delamanation, cracks
6		wood / wood	no adhesion, cracks
7		bamboo / bamboo	no adhesion, cracks
8		wood / wood	no adhesion, cracks

Typical defects that influence mechanical properties or reduce strength must be monitored in later production with regard to their quality. In the following, the selected defect patterns are shown for which an analysis should be carried out in a first step. Initial tests will be analysed for the following failure types: delamination or no adhesive and cracks.

3.2 NDT Methods Evaluation

There are a variety of non-destructive testing techniques, all with their very specific testing capabilities, limitations, advantages and disadvantages. Consideration of the testing principles shows which methods are fundamentally unsuitable for use on the materials considered here and which should be considered further, as they have potential for solving the testing tasks listed before. A detailed analysis of the capabilities of the NDT methods, taken also findings from recent research into account, leads to a first preselection for further investigation of the following NDT methods as shown as an evaluation in Tab. 2.

Table 2. Evaluation of NDT methods for considered renewable raw materials.

NDT Method(1)	Principle suitable?	Remarks
Visual Testing (VT)	-	no inner failures detectable
Ultrasonic Testing (UT)	(+)	…
Acoustic Testing (AT)	?	…
Penetration Testing (PT)	-	not for highly absorbing materials
MT (MT)	-	only for permeable materials
Eddy Current Testing (ET)	-	no electric current inducible
RT (RT)	+ / -	mostly limited to detect volume failures
Computed tomography (CT)	+	very expensive
Thermal Testing / Thermography (TT)	+	active and passive testing possible
Microwave Testing (MW)	(+)	…
Terahertz Testing (THz)	(+) (2)	considered together with microwave testing

(1) (…) method shortcut (2) somewhat similar to microwave testing

For further consideration, the methods CT, TT, UT are preselected, which are expected to have potential either due to their test principle and for which applications/new applications/adaptations have already been found in the literature. AT is excluded for the moment, because the potential is considered to be less as for the selected. Even though the usability for online testing while production process or offline testing capabilities in laboratory will be evaluated.

4. STATE OF DEVELOPMENT AND PRACTICAL RESULTS

The test capabilities are shown for the individual NDT methods, and the results of initial tests are shown in order to greatly reduce the effort required for later introduction. Test constellations are described in such a way that the relevant points for perspective test procedures are already available as far as possible.

4.1 3D computed tomography

Among the industrially applied NDT methods, 3D computed tomography (CT) plays a significant role, as it allows very precise insights into the internal structure of almost all materials. Unfortunately, however, it is also one of the most cost-intensive due to the need for extensive testing technology and peripherals. The application for wood or bamboo materials enables high penetration depths into the interior of the material and also excellent resolution capabilities down to the nanometre range. Pure detachments can only be detected with a high resolution, as there are hardly any density differences and thus only the smallest of contrasts when layers lie closely together. Testing times are considerably long and depend strongly of the desired resolution. Friction welded samples were analysed before the tensile test with a 3D CT system (phoenix nanotom® m, GE Sensing & Inspection Technologies GmbH), see Fig. 9. The relatively even, compressed lignin layer, which is responsible for the bond strength, is clearly visible. Possible damage should be with previously described restrictions reliably detected in a 3D CT scan.

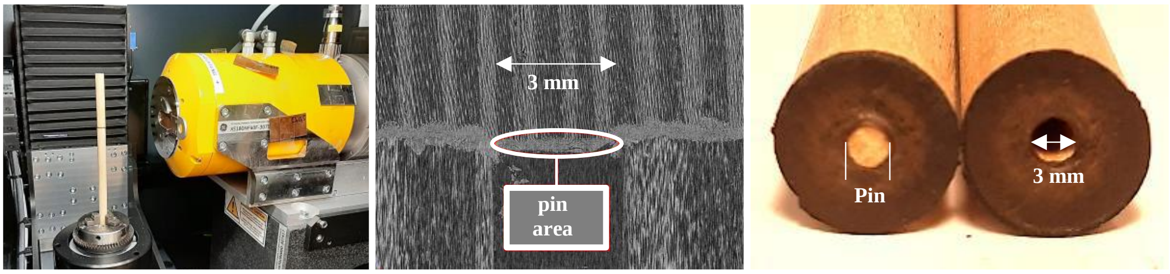


Figure 9. Pine sample in CT system (left), CT image (middle) and sample after tensile test (right).

NDT method evaluation: high resolution (+++), all materials (++), expensive (---), long testing duration (--).

4.2 Ultrasonic Testing

Conventional ultrasonic testing is a well-known method being used in industrial NDT for many years. It bases on the coupling of an acoustic wave into a component and its reflection at interfaces, which in turn is received and interpreted.

In previous work (Hasenstab et al., 2005; Hasenstab, 2006) the use of low-frequency ultrasound (approx. 50…200 Hz) was investigated for wood-based materials to determine the ability to detect e. g. voids, delamination and reduced thicknesses. The propagation speed of the ultrasound is strongly influenced by the fibre direction of the resin. There was measured a difference of more than two times in different directions. Therefore, this must be considered separately. Defects with an extension in direction of the fibres can be located more reliably than those perpendicular to the fibres direction. Pulse-echo techniques (contact coupling of the ultrasound perpendicular to sample surface) or non-contact sound transmission (non-contact technology with transmitter and receiver opposite each other) as shown in Fig. 10 can be used. The test arrangement in reflection offers particular advantages when there is only one-sided accessibility to the component. Transmission configuration allows working with air coupling, so that no coupling medium is required. The non-contact coupling enables testing when coupling medium cannot be used.

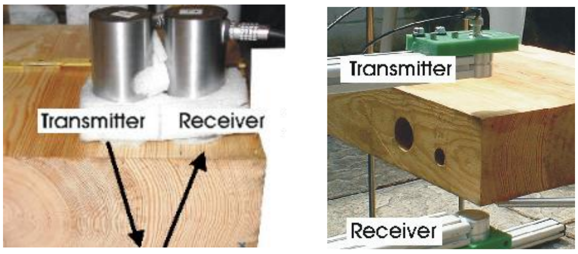


Figure 10. Ultrasonic puls-echo technique (left) and non-contact transmission mode (right) (Hasenstab et al., 2005).

Fig. 11 shows exemplary measurement results, which were generated on special test sample. A cavity with extension of approx. 40 mm is reliably detected with different ultrasound configurations. Not least due to relatively long wavelengths of ultrasound and a propagation speed in wood of about 4000…6000 m/s perpendicular to the fibre direction, minimum detectable defects can be assumed in a range of 10…60 mm.

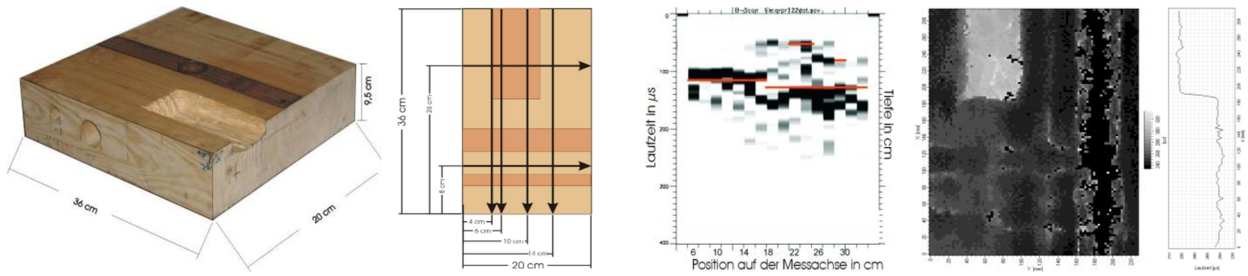


Figure 11. Test sample with dimensions (left), B-scan with echo technique (middle), C-scan non-contact measured in transmission (right) (Hasenstab et al., 2005).

Practical applicability was shown for laminated timber and solid wood using the pulse echo method, demonstrated by analysing various wooden bridge constructions (Hasenstab, 2008; Hasenstab, 2009).

NDT method evaluation: one-sided accessibility (++), medium detectability size (+/-), high penetration depth (+++), non-contact testing (++), dependence from anisotropy (--).

4.3 Microwave or Terahertz Testing

Unlike ultrasonic testing is based on acoustic waves, microwave and terahertz testing methods use electromagnetic waves, adjacent to infrared radiation towards longer wavelengths. Microwaves typically own frequencies of 300MHz…300GHz and wavelengths of 1m…1mm, terahertz radiation (THz) own frequencies of 100GHz…10THz and wavelengths of 3mm…30µm.

Microwave testing is primarily a method for detecting defects in electrically insulating, i.e. dielectric materials and components. The materials that can be tested include, for example, plastics, GFRP, plastic foams and many types of ceramics. The method is based on the propagation, reflection and other interactions between material and electromagnetic waves. Local differences in the materials dielectricity produce a refraction of the radiation and thus reflection, as in optics. For example, pores, foreign material inclusions, as well as delamination and undulation in GFRP can be detected. Fig. 13 shows an exemplary B-Scan testing result of a 600 mm x 250 mm part of a GFRP wind turbine rotor blade component. The indication comes from a depth of approx. 35 mm, it is a wetting error in the foam GFRP interface with a diameter of approx. 20 mm (Becker et al., 2021).

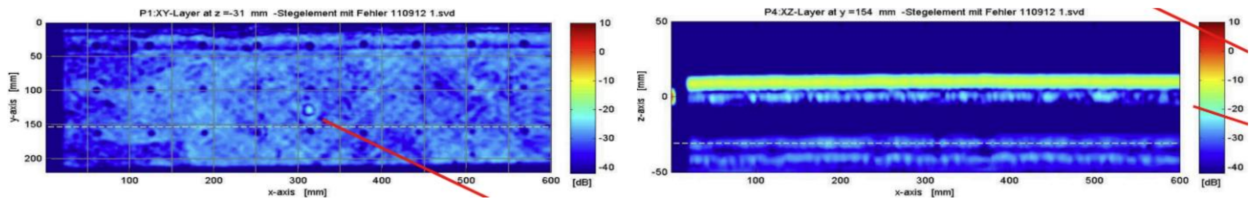


Figure 13. D-scan of GFRP component: wetting error (left), B-scan in profile line (right) (Becker et al., 2021).

Wall and layer thicknesses can also be measured. Microwave testing does not require a coupling medium and can be used in contact or non-contact reflection or transmission configuration. Unlike e. g. X-rays, microwaves are not ionizing and therefore comparatively harmless. The penetration depth at microwave testing is strongly dependent of the materials dielectric properties (higher $\varepsilon \rightarrow$ more electric damption $\rightarrow$ less penetration depth). The penetration depth for wood and similar materials varies depending on their humidity content, but it can be estimated to be not more than a few 10 mm.
Recent investigation (Hinken and Gopalan, 2020) show, that the pitch-catch method used in ultrasonic testing can also be transferred to microwave testing. Microwave tests were carried out with the also called half-transmission method for butt welds on polyethylene pipes, the test configuration is shown in Fig. 14. Those radial butt welds on polyethylene (PE) pipes can successfully be tested. Failures like through and blind holes with dimensions between 2 to 5 mm were detected as shown by an exemplary result image. The geometry is comparable to the radial friction welds on the bamboo rods, which indicates the idea that a similar test could also be possible here. Further tests should be continued. The geometry is comparable to the radial friction welds on the bamboo rods, which indicates the idea that a similar test could also be possible here. Further tests should be continued.

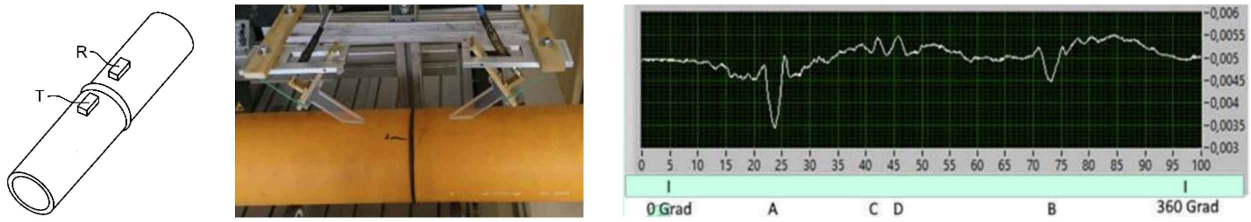


Figure 14.: Pitch-catch method setup, position of microwave couplers for transmitting (T) and receiving (R) (left), device under test for buttwelded PE pipe (middle), result of circumferential scan (right) (Hinken and Gopalan, 2020).

NDT method evaluation: contactless possible (++), no coupling agent (++), low safety measures (+), large observation depth (+++), Smaller attenuation than ultrasound (+), easy to automate (+), reflection- and transmission mode (++), transmission allows direct imaging (+), also porous, foam sandwich components, etc. (+), only insulating materials (---).

4.4 Thermographic Testing

Thermography is classified in passive and active methods. This means for active methods, that special heat sources induce a heat transfer into a component. The interaction with defects maps a heat pattern on the samples surface that can be detected with an infrared camera and then be further evaluated. Passive methods characterize that no extra heat source is used for testing. All heat transfer and infrared radiation from the sample originate from the production process of the component (Siemer, 2010).

Initial analyses with passive thermography are made for the friction welding process of the wooden bars. Fig. 15 shows the experimental setup and plotted temperatures while friction welding with several speeds. First comparisons show variations of the temperature profiles with the produced quality, but only rough statements are possible so far. Further investigations must be done to evaluate what kind of irregularities can be controlled in later production processes. Smaller defects of some mm dimensions will very likely not be detectable with this test setup.

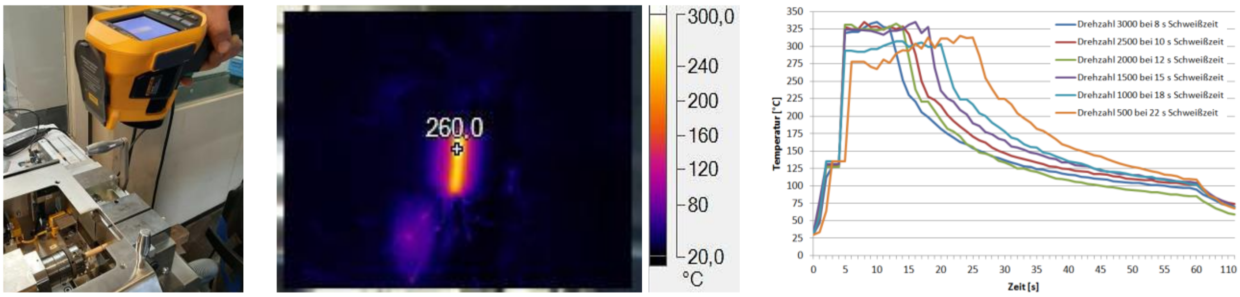


Figure 15. Passive thermography on friction welding of wooden bars (left), temperature plots for several speeds (right).

To estimate the testing capabilities of active thermography investigations with promising kinds of thermal excitation were conducted (cf. Siemer et al., 2023). Fig. 16-18 show initial results of principle tests that indicate high potential for later quality control with NDT. The round wood bar sample (Fig. 16, right) was not welded but glued for the first principle test. The applied ultrasonic excitation source provided frequencies of about 60 Hz and up to 8 Watt. As heat source an ordinary industrial hot air dryer was used. Dependent from the failure depth an accuracy of several mm can be estimated. Advantages and disadvantages of the NDT method strongly dependent on the chosen excitation, therefore they will only be described after further investigations.

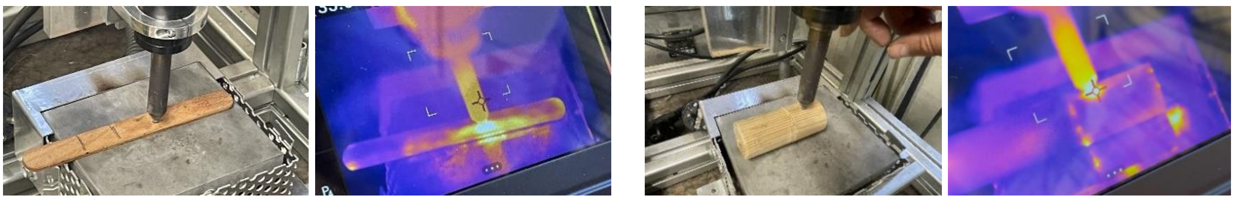


Figure 16. Principle tests for inspection task 2 (left) + 6 (right) of Tab. 1 for failure type “delamination”: test setup and result image after ultrasonic excitation - the concealed adhesive is becoming clearly apparent in the infrared image.

Figure 17. Principle tests for inspection task 3 of Tab. 1 for failure type "crack": test setup and result image after ultrasonic excitation – the crack in the wood top layer is becoming clearly apparent in the infrared image.

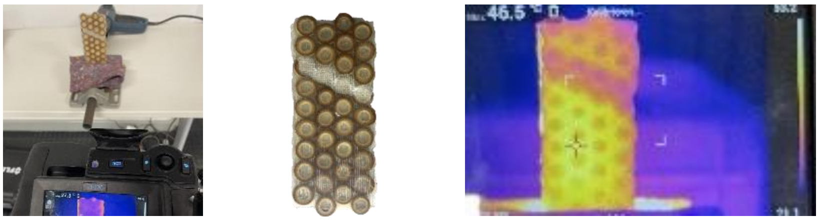


Figure 18. Principle tests for inspection task 5 of Tab. 1 for failure type "delamination": test setup and result image after hot air excitation – the delamination in the wood top layer is becoming clearly apparent in the infrared image.

NDT method evaluation: contactless possible (++), medium safety measures (+), large observation area at once (++), medium penetration depth (-), reflection- and transmission mode (++).

5. RESUMEE AND OUTLOOK

Tab. 1 shows capabilities of the selected NDT methods as a result of both considerations and practical investigations.

Table 3. Capability Overview of NDT methods for considered renewable raw materials

NDT Method	**Potential for …** 1. process integration, 2. sample geometry (1)	**Detectable failure estimation**	**Remarks**
Ultrasonic Testing (UT)	offline testing, 1-8	delamination, cracks mm … cm (2)	ability for crack detection depends on geometric alignment
Computed tomography (CT)	offline testing, 1-8	delamination, cracks µm … mm (2+3)	crack detection critical, when surfaces hardly compressed together; cost-intensive measurement limits application to laboratory analyses
Thermography (TT)	inline control +	>> mm ? (2+3)	high potential, but needs to be further investigated
	offline testing, 1-8	delamination, cracks mm … cm (2+3)	sensitivity hardly depends on failure depth and thermal excitation source
Microhertz Testing (MW)	offline testing, 1-8	delamination, cracks cm … cm (2)	sensitivity limited by radiation wavelength
Terahertz Testing (THz)	offline testing, 1-8	delamination, cracks mm … cm (2)	higher resolution as for microwave testing, lower penetration depth

(1) testing task from table 1 (2) result of theoretical assessment (3) derived from experimental results

In summary, the results show multiple specific potentials to be used for characterization of novel sustainable composites and joints in renewable materials. Further research and development are required to refine the inspection techniques and optimise their performance. It is recommended to continue with thermographic techniques both passive methods for inline control and active methods with ultrasonic and hot air excitation to gain more precise knowledge about the condition of the materials respectively the cohesion of components and parts. Another excitation source, modulated optical radiation, should also be tested. The ultrasonic and the microwave testing should be pursued. Terahertz testing could be an adequate compromise to achieve better resolutions compared to microwave testing where smaller penetration depths are sufficient for defect detection. Investigations for the mentioned NDT methods will continue.

The fundamental considerations, the analysis regarding typical testing tasks for renewable materials and composites as well as experimental results help to determine the capabilities of NDT, meeting the specific needs of the application. Identifying areas of opportunity and challenges, the results provide the basis for further exploration. The findings help to identify suitable NDT methods for specific testing tasks for renewable materials and composites. They reduce effort and time for later implementation of NDT methods for quality assurance or production control.

6. ACKNOWLEDGEMENTS

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