Air-Coupled Ultrasonic Inspection Technique as NDT Tool for Evaluation of Porous Wound Oxide/Oxide Composite Ceramics

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Abstract. Contactless air-coupled ultrasonic (ACU) inspection technique is used for non-destructive evaluation of oxide/oxide composite ceramics (CMC). The main material (WHIPOX™) inspected within this work is made of Al2O3-fiber filaments (Nextel 610) which are embedded in a porous ceramic alumina matrix. Some variations with mullite fiber filaments and/or mullite matrix are investigated as well. Production of the CMC is performed via winding of the infiltrated fiber filament around a mandrel with 45° or 60° winding angle.

Some material specimens are examined before and after sintering process revealing the microstructural changes during the sintering stage. Extra thick plates (up to 16 mm) are investigated demonstrating the penetration/resolution limits of the ACU technique. The most significant defects that are identified by non-destructive ACU are delaminated areas showing a unique response while interacting with the acoustic waves. Furthermore, compared to the undisturbed material, highly porous regions within the CMC demonstrate higher acoustic transmittance for the ultrasonic wave. Consequently, further systematic investigations are performed to reveal the nature of the observed effects. The investigations presented here include ACU scans of the material specimens with various parameters (e.g. scanning frequency) in transmission mode. The ACU results are compared to data from computed tomography (CT) scans to ensure the meaningfulness of the information obtained with ACU.

Introduction

Ox-Ox composite materials are increasingly used for aerospace applications [1]. This type of material possesses both oxidation resistivity in comparison to other type of ceramic composites (e.g. SiC/SiC) and mechanical stability in terms of damage tolerance that is typical for fiber-reinforced ceramics [2]. Mechanical properties of Ox/Ox-CMCs, however. are strongly dependent on the material quality and local microstructure. Previous investigations demonstrated that production-related defects negatively affect the mechanical properties, therefore can lead to an unexpected component failure in operation [3]. Therefore, quality control of the produced material is an important issue in order to guarantee a reliable operation of an Ox-Ox component. Quality control should be performed with non-destructive tools for further usage of the inspected material (or component).

In this work the investigations are performed with non-contact (air-coupled) ultrasonic technique (ACU). The advantages of this technique are the absence of the contact medium (time and costs issues) and the costs of the device in comparison to other state-of-the-art techniques (e.g. computer tomography).

Previous investigations demonstrate the usability of ACU technique for the as-produced material with artificial defects [3]. Next step is the application of the air-coupled ultrasonic technique for quality control purposes. Firstly, this requires the definition of production-related defects and their detection in the as-produced material. Secondly, in order to avoid further unnecessary production costs defects need to be determined as soon as possible.

1. Material and Experimental Arrangements

1.1 Material

Material investigated within this work is mainly WHIPOX® made of alumina fiber filaments (Nextel 610) embedded in a porous alumina matrix. During the manufacturing the fiber roving is firstly infiltrated with the ceramic matrix suspension and then wound on a mandrel. After the winding process the cylindrical body is cut along the winding mandrel and taken off the mandrel to form a plate and left drying. Finally, after the drying process the “green” plate is sintered.

An ideal wound material can be described by 2 regions: unidirectional laminate (UD-laminate) and cross-lines. In the UD-laminate regions tightly wound fiber bundles are stacked without any structural distortions [4]. In cross-line regions are formed of numerous single cross-over points which are a typical feature of any wound or weaved structure [3].

Additional variations of the material manufactured for the investigation are alumina fibers in mullite matrix, mullite fibers in mullite matrix and mullite fibers in alumina matrix. An overview of the investigated materials with the corresponding documentation is given in Table 1. Composition notation is structured in the following way: fiber type + matrix type. Fiber types are alumina Nextel 610 (N610) and mullite Nextel 720 (N720). Matrix types are alumina (A524) and mullite (A534) as well.

Table 1. Investigated material.

Specimen name	Composition
W1520	N610 + A524
W1586-1	N610 + A524
W1611	N720 + A534
W1612	N720 + A524
W1616	N610 + A534
W1617	N610 + A524
W1619	N610 + A524
N2	N610 + A524

1.2 Air-coupled Ultrasonic Set-up

The set-up used in these investigations consists of the scanning system FlatScan 1000 AirTech with USPC 4000 AirTech module (Ingenieurbüro Dr. Hillger). The transducers are AirTech 120, AirTech 200, AirTech300 and BAM 696/697. The investigations are performed in transmission mode. The frequency spectra of the transducers are demonstrated in Fig. 1.

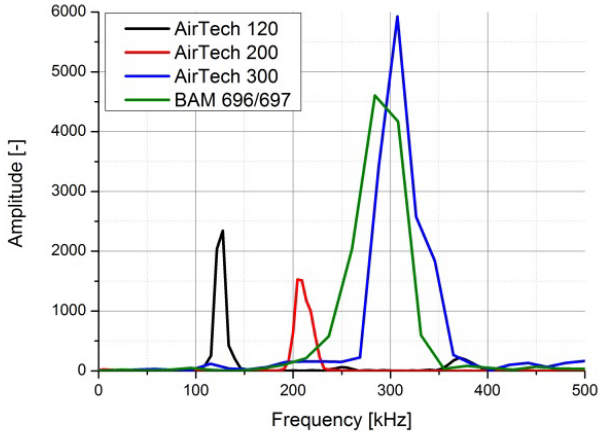

Fig. 1. Frequency spectra of the transducers used for the investigation.

The data obtained in investigations is demonstrated in the form of amplitude-based C-scans (logarithmic scale) with corresponding statistical data. The time window for the recalculation in a C-scan is the first pulse. Comparison of the C-scans is performed in terms of amplitude-based statistical analysis of the regions of interest with Oculus 1.43 Deluxe (Ingenieurbüro Dr. Hillger).

2. Results and Discussion

2.1 Technique-related Investigations

2.1.1 Computed Tomography Reference Investigation

First aspect to be clarified for an application of ACU as non-destructive technique is to investigate the type of the detected defects, their sizes and positions. For this purpose a plate with defects is produced and investigated via computer tomography. The results are presented in Fig. 2.

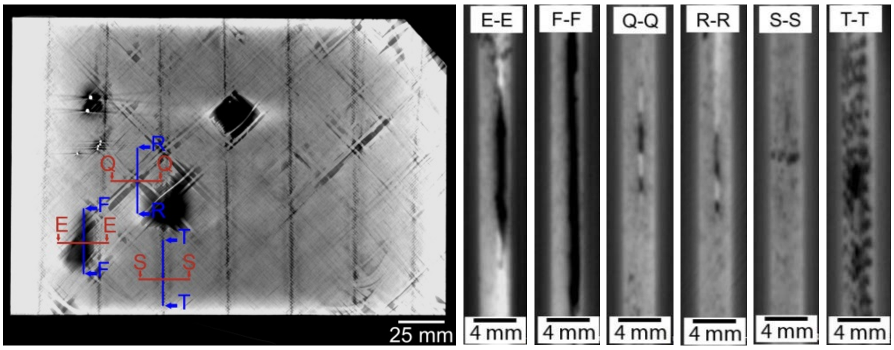

Fig. 2. 2D-image of the CT-scan (W1520) with slices E-E, F-F, Q-Q, R-R, S-S and T-T.

CT-scan reveals the presence of 8 delaminated areas, e.g. slices E-E and F-F. Some additional areas with material gaps are observed within a plate, e.g. slices Q-Q and R-R. These materials gaps are empty spaces between fiber bundles in the material. Another detected production-related feature (cross-line) is depicted in slices S-S and T-T. Therefore, 3 relevant detected microstructural features of the materials are: delaminations, material gaps and cross-lines. Cross-lines, however, cannot be avoided within the composite and could be found optically; for this reason their detection is not in focus of this investigation.

2.1.2 Investigation of Various Material Regions

Next step is the comparison of the CT- and ACU-results. For this purpose W1520 is investigated with transducer AirTech 200. The results are demonstrated in Fig. 3.

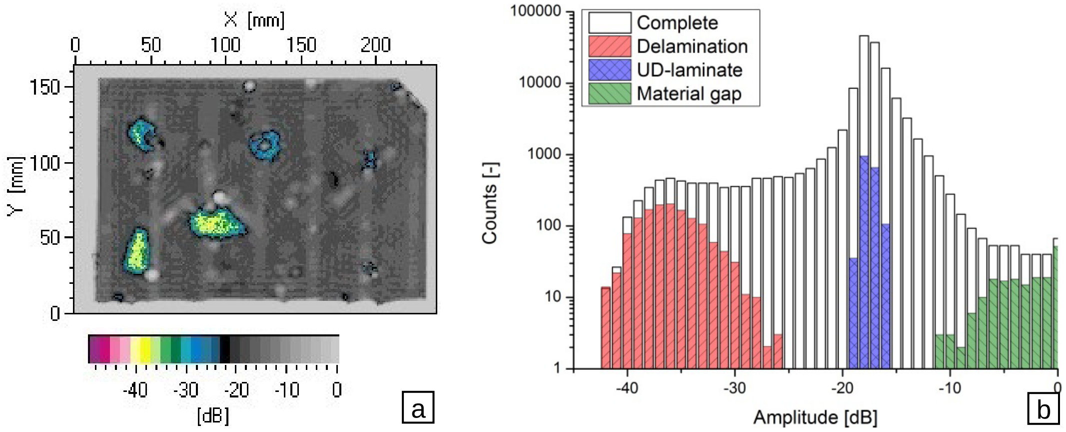

Fig. 3. C-scans of W1520 scanned with AirTech 200 (a) and statistics of the ultrasound signal in UD-laminate, delaminated and material gap regions (b).

Statistical analysis of W1520 reveals that the signal amplitude distribution of the entire plate can be decomposed in 3 constituents: the first one related to the UD-laminate region (X = 168…189 mm, Y = 103…140 mm) is the largest one; the second is related to the delaminated regions with lower transmissivity compared to UD-laminate (X = 81…102 mm, Y = 52…64 mm); the third one is related to material gap regions (X = 92…98 mm, Y = 73…79 mm) with higher transmissivity compared to UD-laminate. UD-Laminate region demonstrate a narrow distribution with a pronounced peak (-18 dB). Delaminated region show up as a wider distribution with an identifiable peak (-36 dB). Material gap region does not provide sufficient statistical data for a clear statistical distribution and could be identified as a signal between -11 and 0 dB.

Therefore, it is concluded that the production-related defects (delaminations and material gaps) can be detected and distinguished from the UD-laminate region by ACU technique.

2.1.3 Investigation with Various Transducers

Next investigation step is the inspection of a plate with defined defects (W1520) with various transducers in order to determine the best one for further quality control. The corresponding C-scans are demonstrated in Fig. 4.

In this investigation 2 main factors describing the quality of ACU as NDT tool are taken into account: 1) the contrast between the UD-laminate material and possible defects (delaminations and material gaps); 2) the defect shape recognition based on the qualitative comparison of the detected defect size to the real one (CT-scan). The contrast between the UD-laminate and delaminated regions is quantified via statistical data in terms of the difference (delta) between the distribution peaks at UD-laminate (X = 81…102 mm, Y = 52…64 mm) and delaminated (X = 81…102 mm, Y = 52…64 mm) regions. The defect shape recognition is performed with the delaminated region (X = 114…136 mm, Y = 102…120 mm) since its sharp rectangular shape (Fig. 2) gives a possibility to qualify the transducers precision in terms of closeness to rectangular shape.

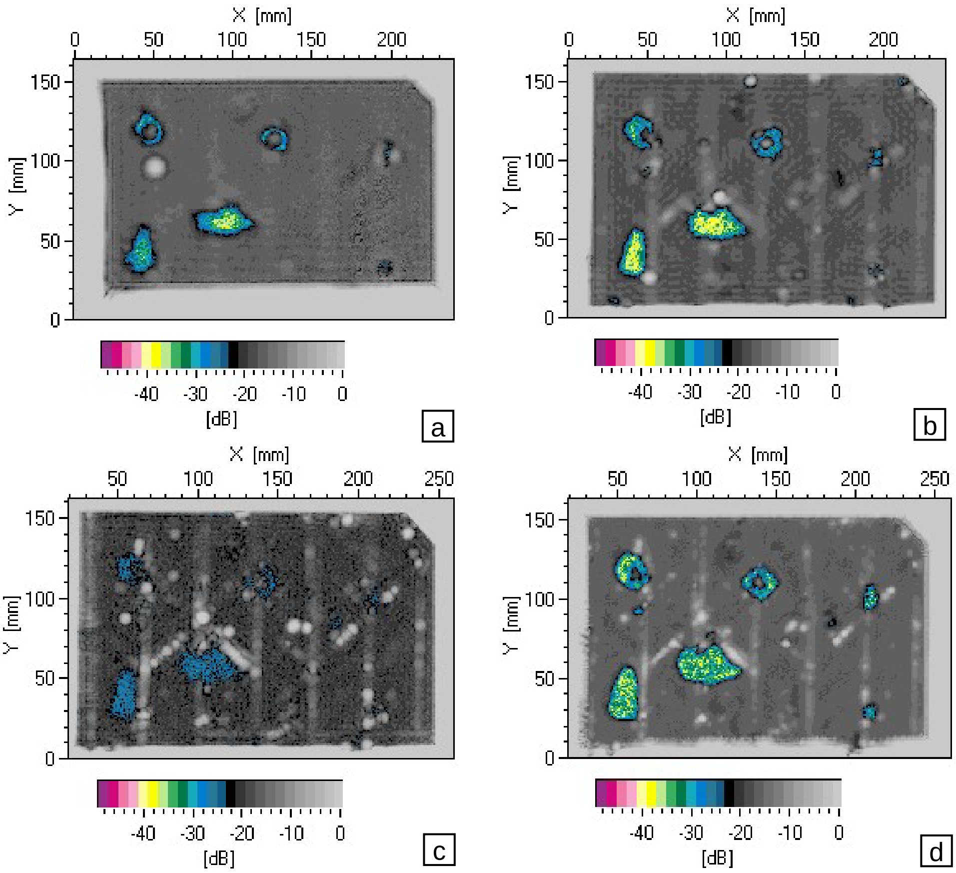

Fig. 4. C-scans of W1520 with AirTech 120 (a), AirTech 200 (b), AirTech 300 (c, courtesy of Ingenieurbüro Dr. Hillger) and BAM 696/697 (d, courtesy of Ingenieurbüro Dr. Hillger) transducers.

All transducer types demonstrate similar detection of the delaminated regions except AirTech 120 for the defect 2 (X = 44…57 mm, Y = 89…100 mm) that is identified similar to a material gap region. In most of the delamination cases, the response is high damping of the ultrasonic wave. The reason of the particular response of defect 2 to the acoustic signal generated by AirTech 120 is still in investigation.

Statistical data are summarized in terms of the peak position of the distribution for the UD-laminate and delaminated regions in Table 2.

Table 2. Amplitude-based statistical analysis for various transducers.

Transducer	UD-laminate region [dB]	Delaminated region [dB]
AirTech 120	-17	-35
AirTech 200	-18	-36
AirTech 300	-20	-25
BAM 696/697	-17	-34

The contrast between UD-laminate and delaminated regions is between 17 and 20 dB for 3 of 4 transducers. AirTech 300 shows the difference of about 5 dB making it less suitable for the detection of delaminated regions. Defect shape recognition quality for the transducers is ordered as follows (from high to low): BAM 696/697, AirTech 200, AirTech 120, and AirTech 300. Transducer BAM 696/697 is able to resolve the edges of the delaminated region very sharp resulting in a high-quality match between CT- and ACU-scans. Basing on the results of contrast and precision, the transducer BAM 696/697 is concluded to be the best option for the detection of delaminated regions.

Material gaps are detected with transducers AirTech 200, AirTech 300 and BAM 696/697. In all cases these regions demonstrate lower damping of the ultrasonic wave. The same effect is observed in the region of cross-lines and can be explained with similar microstructure. Transducer AirTech 120 demonstrates lower resolution compared to other transducers due to its lower frequency band (Fig. 1), i.e. resolution capability. The quality of transducers for material gap detection from high to low is ordered as follows: BAM 696/697, AirTech 300, AirTech 200 and AirTech 120.

Based on the observations, the best transducer among the tested ones for detection of both delaminated and material gap regions is BAM 696/697. The second best is AirTech 200. Further investigations are mainly performed with AirTech 200 due to availability issues.

2.2 Production-related Investigations

2.2.1 ACU Application at Successive Production Stages

The next important step is to determine the optimal stage for the quality control of a fabricated specimen. As it is described in Section 1.1 the material is produced in several successive stages: winding, drying and sintering. Despite the fact, that machining negatively affects the mechanical behaviour of a fiber composite, in some cases (e.g. engineering tolerances must be met for a component) the material has to be treated for further application, e.g. by grinding. Therefore, four stages are typically involved in the production process of an oxide/oxide CMC component. After the winding process while still wet, the plate is mechanically unstable and cannot sustain its own weight making its ACU investigation in transmission mode impossible. Therefore the material is investigated after drying, sintering and grinding stages. The corresponding C-scans are given in Fig. 5.

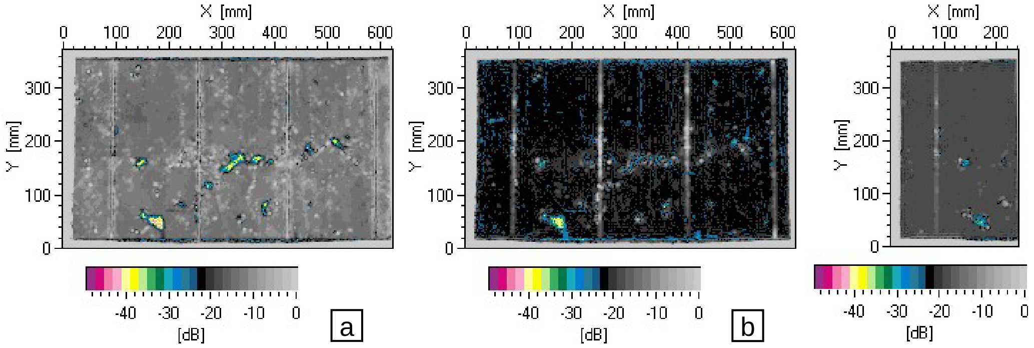

Fig. 5. C-scans of W1617 after drying (a), sintering (c) and the left part of W1617 after grinding (c) (for grinding purposes the plate was cut in 2 parts).

After drying process the plate contains some delaminated areas that are depicted in colour in Fig. 4a. After sintering stage some of the delaminated areas lose their contours, e.g. in the middle region. After grinding the delaminated regions remained visible, additionally, background “noise” decreases (distribution of the transmitted signal becomes narrower) that is related to the removal of the excess matrix from the plate surface.

A possible change of the delamination microstructure or morphology during the sintering stage is suspected. Further investigations are going on for clarification of the possible changes.

Statistical data corresponding to the contrast between the UD-laminate and delaminated regions with respect to the productions state are given in Table 3.

Table 3. Amplitude-based statistical data for various production stages.

	Position of the distribution peak	
Production stage	**UD-laminate region [dB]**	**Delaminated region [dB]**
After drying	-14	-41
After sintering	-22	-39
After grinding	-19	-29

Basing on the observations, the best visibility of the delaminated regions (highest distance between the laminated and delaminated regions peaks) is determined for the material after the drying stage. Therefore, non-destructive inspection is defined to be performed after drying.

2.2.2 Investigation of material with fiber and matrix variations

In some application cases oxide/oxide ceramic composites should be manufactured with fibers or/and matrix other than pure alumina. Therefore, the applicability of the ACU technique is tested on plates with further variations: mullite fibers in mullite matrix (W1611), mullite fibers in alumina matrix (W1612), alumina fibers in mullite matrix (W1616). These materials are compared to a standard variation: alumina fibers in alumina matrix (W1619). The results are presented C-scans with statistical analysis along with a SEM image of the composite in Fig. 6.

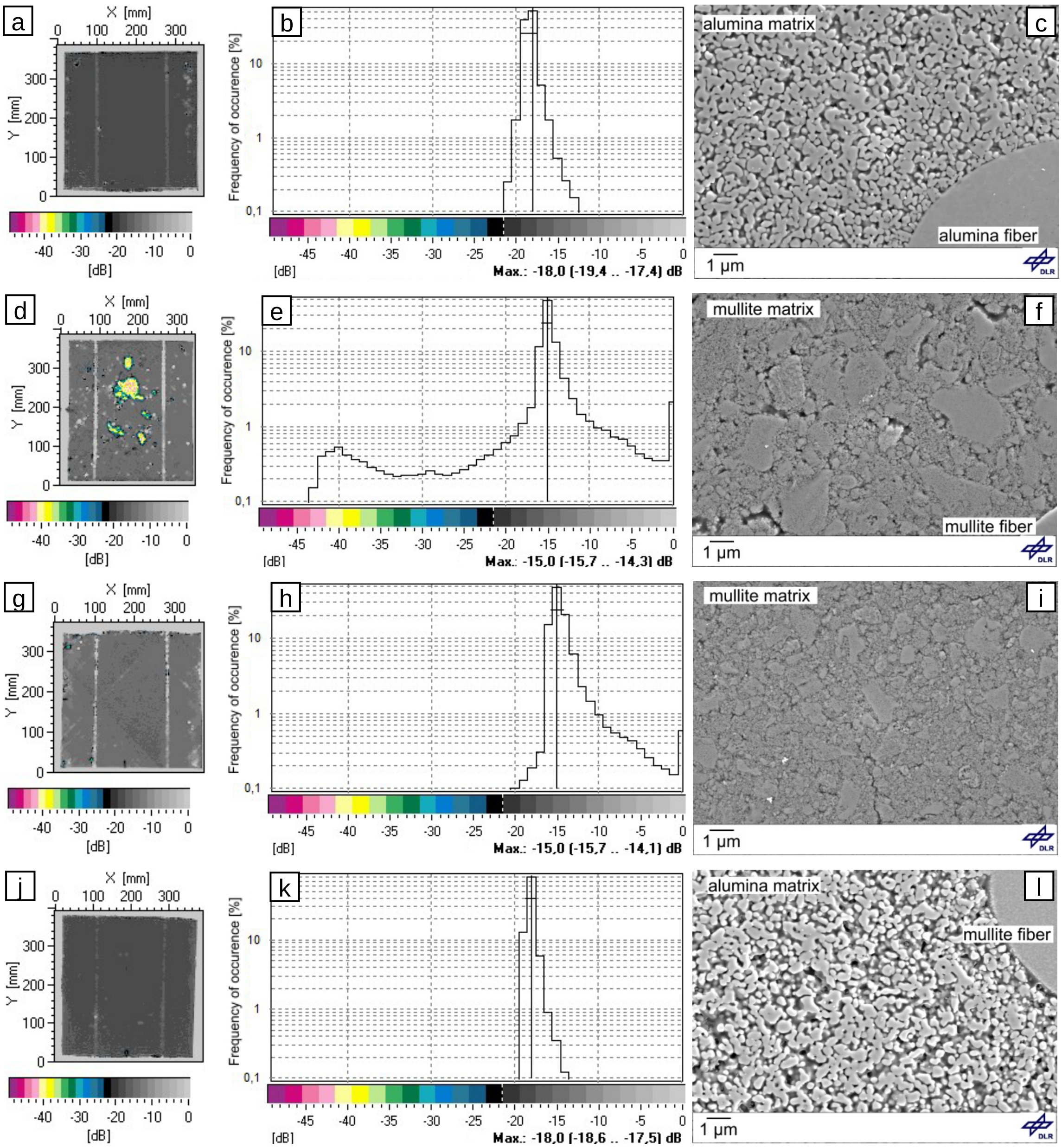

Fig. 6. C-scans, statistical data and SEM images of W1619 (a,b,c), W1616 (d,e,f), W1611 (g,h,i) and W1612 (j,k,l).

Massive delaminated regions as well as numerous regions with material gap are observed in W1616. The delaminated regions in W1616 are related to a forming process of the plate in undried state (directly after winding). This demonstrates the applicability of the ACU technique for quality control of other typical variations of the oxide/oxide CMCs. Another interesting observation is summarized in Fig. 7.

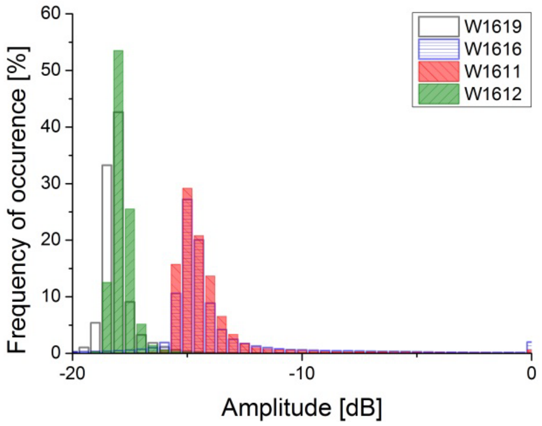

Fig. 7. C-scans, statistical data and SEM images of W1611, W1612, W1616 and W1619.

Four measurements can be classified in 2 groups with an overlapping of the largest areas (UD-laminate region): mullite matrix materials (W1611 and W1616) and alumina matrix materials (W1612 and W1619). Therefore, it is concluded that matrix-factor has dominating contribution (comparing to the fiber contribution) to the transmissivity of the entire material. Further investigation revealing the role of matrix parameters (porosity, composition, etc.) on the transmissivity to the acoustic waves are going on.

2.2.3 Investigation of material with various winding angles

Some applications of a fiber-reinforced composite require special fiber orientation that results in various types of the composite. Therefore, a composite with 60° winding angle is investigated via ACU technique. The results are presented in Fig. 8.

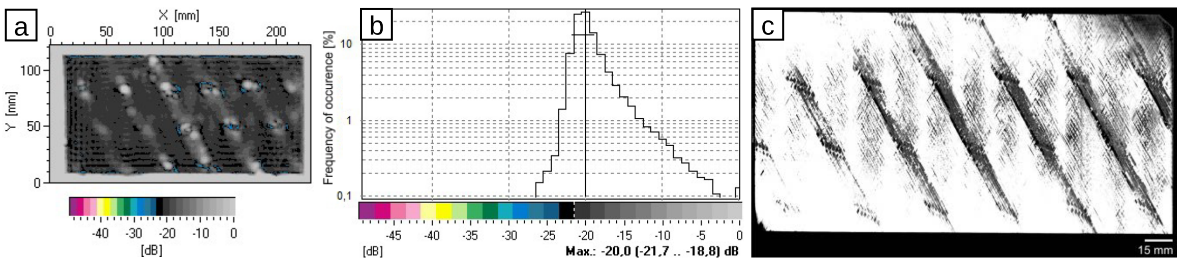

Fig. 8. C-scan (a), statistical data (b) and CT-image (c) of W1586-1.

Massive material gap regions as well as some delaminated regions are detected. To confirm their presence an additional CT-scan is performed proving the statement. Their presence is related to the winding procedure giving a hint to its optimization. No drastic difference to previously investigated material is observed, therefore materials with various fiber orientations can be inspected with ACU technique.

2.2.4 Investigation of an extra thick specimen

An extra thick specimen (~16 mm) produced via lamination of 3 wound plates in wet state (directly after winding) is inspected. A delamination between adjusted parts of the specimen can be observed without any NDT tool; however its depth and size are unclear. ACU results are presented in Fig. 9.

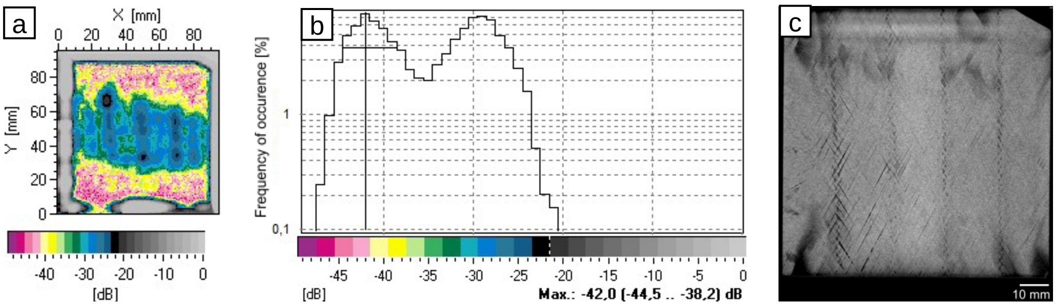

Fig. 9. C-scan (a), statistical data (b) and CT-image (c) of N2.

The increased thickness decreases the transmissivity of the acoustic signal. Nevertheless, UD-laminate intact region can still be distinguished from the delaminated one (2 separate distributions in statistical data with peaks at -42 dB and -29 dB). CT-scan confirms the presence of the delamination and its shape.

3. Conclusions

Concluding the investigations further statements could be done:

- Delaminations and material gaps can be detected with ACU technique with a good contrast to undisturbed UD-laminate material of good quality.
- Delaminations demonstrate low transmittance of the acoustic signal, whereas material gaps show a higher one (in comparison to UD-laminate regions).
- The best transducers among tested is BAM 696\697.
- For quality control of the manufactured material the specimens has to be inspected in dried (not sintered) state.
- Various fiber orientations do not affect the ACU response.
- Extra thick specimens (min. up to 16 mm) can be inspected with the same technique.
- Porous matrix is the dominating factor for acoustic transmittance of the investigated material. Further investigations are planned to clarify this observation and to correlate it to various matrix parameters (porosity, composition, etc).

References

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