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3D Printed Al₂O₃ for Terahertz Technology

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ABSTRACT In this work we demonstrate that 3D printed Al₂O₃ is a promising material for prototyping and precise fabrication of quasi-optical devices in the terahertz frequency range. The 3D printed Al₂O₃ exhibits a low absorption coefficient ($\alpha < 2\text{cm}^{-1}$ at 1 THz) and a high refractive index ($n > 3$). The printing resolution in the sub 50 μm range allows for the implementation of structures in the 0.3–3.0 THz range on the subwavelength scale. Furthermore, the printing process enables the realization of crystalline solids, which allows the use of the Al₂O₃ birefringence effect. Here, a $\Delta n \approx 0.05$ was achieved and used for the implementation of $\lambda/2$ -wave plates working at ~ 1 THz. The material properties and wave plates were characterized using a terahertz time-domain spectrometer.

INDEX TERMS 3D printing, ceramics, Al₂O₃, terahertz, wave plate.

I. INTRODUCTION

During the last twenty years, terahertz (THz) technology has made tremendous progress. Now, on the one hand THz systems are used for basic research in labs worldwide [1]–[3] and on the other hand many practical applications are foreseen for THz technology. These applications are predominantly in the fields of non-destructive material and structural testing [4]–[6], as well as sensing [7]–[9]. Further, short-range wireless communication is expected to become a mass market in a few years from now [10]–[12]. Consequently, THz sources and THz detectors were further developed in recent years and a multitude of passive devices to guide or manipulate THz waves have been demonstrated. This includes filters [13], reflectors [14], lenses [15], diffraction gratings [16], waveguides [17] and couplers [18], beam splitters [19] and last, but not least wave plates [20], [21].

A promising way for fast prototyping and fabrication of THz devices is 3D printing (additive manufacturing) [15], [17], [18], [22]–[25]. The main advantage of 3D printing is the monolithic fabrication of complex structures at low cost, high accuracy and high fabrication speed for small volume production [26]. 3D printing of different polymers and their applications in the THz range have been studied by various researchers [15], [18], [23], [24], [27]–[29]. Until now, most demonstrations of 3D printed THz devices are restricted to

fused deposition modeling (FDM) manufacturing. The two main limitations for this technology is the poor resolution of more than 100 μm and the limited availability of highly transparent printable polymers [30]. Further, all highly transparent polymers have a refractive index on the order of 1.5, which prevents the fabrication of ultra-thin devices as well as photonic crystals with large bandgaps, where a large contrast in refractive index is beneficial. Further, material properties which can increase the functionality of the devices, such as birefringence are not known for FDM based 3D printed materials.

A material class which has the potential to overcome these challenges are ceramics. Compared to polymers, ceramics usually have a higher refractive index and some ceramics, such as Al₂O₃, show a low absorption coefficient at THz frequencies [31]–[33]. Furthermore, the crystalline form of Al₂O₃ also known as sapphire, is known to exhibit large birefringence in the THz range [34]. This could lead to birefringence of a 3D printed Al₂O₃ ceramic samples, if some degree of crystallinity is induced during the printing process. Therefore, the 3D printing of ceramics offers new possibilities for precise fabrication of low-loss and compact THz devices of complex shapes depending on the manufacturing method.

Various methods, including: SLS (Selective Laser Sintering), LOM (Laminated Object Manufacturing), IJP (InkJet Printing), Two-Photon Polymerization (TPP) and LCM (Lithography-based Ceramic Manufacturing), have been

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invented for 3D printing of ceramics [35]. However, of these techniques, the LCM technology has the highest potential for THz applications, due to the combination of high material quality and a potential structural resolution in the sub 50 μm range. LCM utilizes ceramic particles suspended in a photocurable resins as the printing material (i.e. printing slurry). While printing layer by layer, polymerization of the suspension through controlled UV irradiation will form a green body, which consists of the polymerized polymer and ceramic particles, of the desired shape. After printing, the green body is debinded and subsequently sintered to form the final ceramic part [36]–[38]. The LCM method has been successfully employed for the fabrication of ceramic parts such as: turbine blades and gear wheels [37], as well as first mm and sub-mm wave applications such as wireless RFID (radio-frequency identification) tags [39]–[41].

In order to further establish ceramic additive manufacturing for mm and sub-mm wave applications, a quantification of the dielectric properties for 3D printed ceramics in the THz range is vital. However, to the best of our knowledge, these properties are currently unavailable for most of the 3D printed ceramic materials. Hence, the aim of the research discussed in the following has been the investigation of the dielectric properties for LCM Al₂O₃ in the frequency range between 0.3 and 2.5 THz. Here, we discuss Al₂O₃, as it is one of the most widely used ceramics [42]. Based on the observed birefringence phenomenon in the sample, we further demonstrate a λ/2 wave plate application in the THz range.

II. EXPERIMENTAL SECTION

A. 3D PRINTING OF Al₂O₃

Cylindrical Alumina samples were fabricated using the LCM method. For this purpose, a Lithoz CeraFab 7500 [43] printer was used with a X, Y and Z resolution of 40 μm, 40 μm and 50 μm, in combination with LithaLox HP350 [43] UV curable slurry containing 49 vol% of high purity Al₂O₃ (~99.8%).

During printing, layer by layer specific UV exposure (t: 4.4 s/layer, energy: 220 mJ/cm²/layer) initiated the polymerization reactions in the slurry, and brought about the formation of the 3D green bodies. Fig. 1 a) shows the schematic image of a printed sample. It should be noted, that the sample was built perpendicular (i.e. standing on) on the building platform. This minimizes its contact area with the building platform and facilitates the device detachment after the completion of the printing step. Subsequently, the support structure was carefully removed and the sample was cleaned using a LithaSol 20 [43] cleaning fluid, in order to wash out the residue of the slurry (i.e. unpolymerized slurry).

Conversion of the green samples into the final parts was achieved using an electric furnace with the following temperature cycle programming: I) Preconditioning: T_{Max} = 120 °C, heating rate = 0.2 K/min, dwell time = 3 days, II) Debinding: T_{Max} = 1100 °C, heating rate = 1 K/min, dwell time = 5.8 days and III) Sintering: T_{Max} = 1700 °C, heating

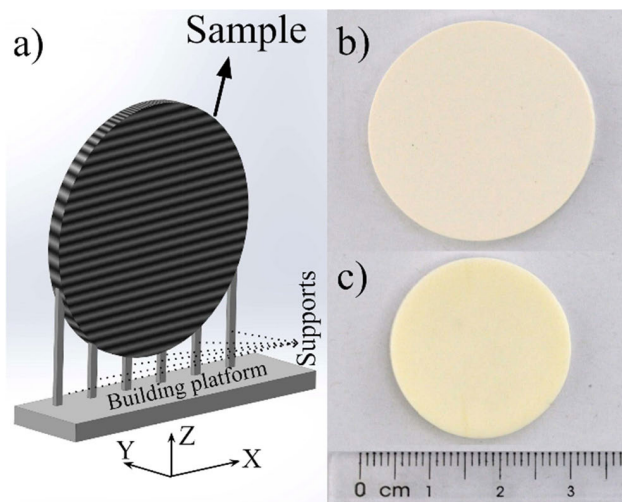


FIGURE 1. Schematic image of the a) printed sample as well as photographs of b) green and c) sintered Alumina.

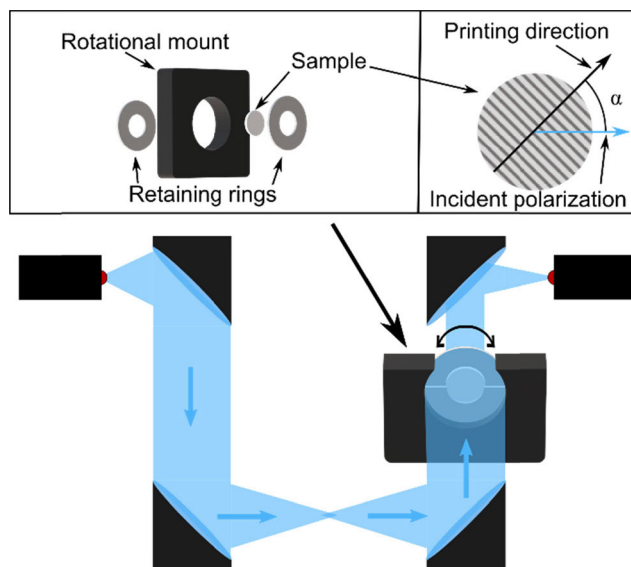


FIGURE 2. Sketch of the setup. The arrows indicate the propagation direction of the THz radiation. The inset shows how the sample was mounted into the rotational mount and defines the angle α between the printing direction and the polarization of the THz radiation incident on the sample. The retaining rigs with clear aperture of approximately 22 mm were covered with aluminum foil to block part of the THz beam.

rate = 0.8 K/min, dwell time = 4.3 days. IV) Cooling: The furnace turned off and cooled naturally. In Fig. 1 images of a green and a sintered 3D printed sample are shown. It can be seen, that the sample has shrunk about 21 Vol% during the sintering step, mainly due to the polymer burn-out during the debinding step.

B. SAMPLE CHARACTERIZATION

A THz time domain spectroscopy (TDS) system [44] with a pair of fiber-coupled photoconductive antennas was used for the characterization of the samples. Four off-axis parabolic mirrors (OAPM) were used to guide the THz beam from

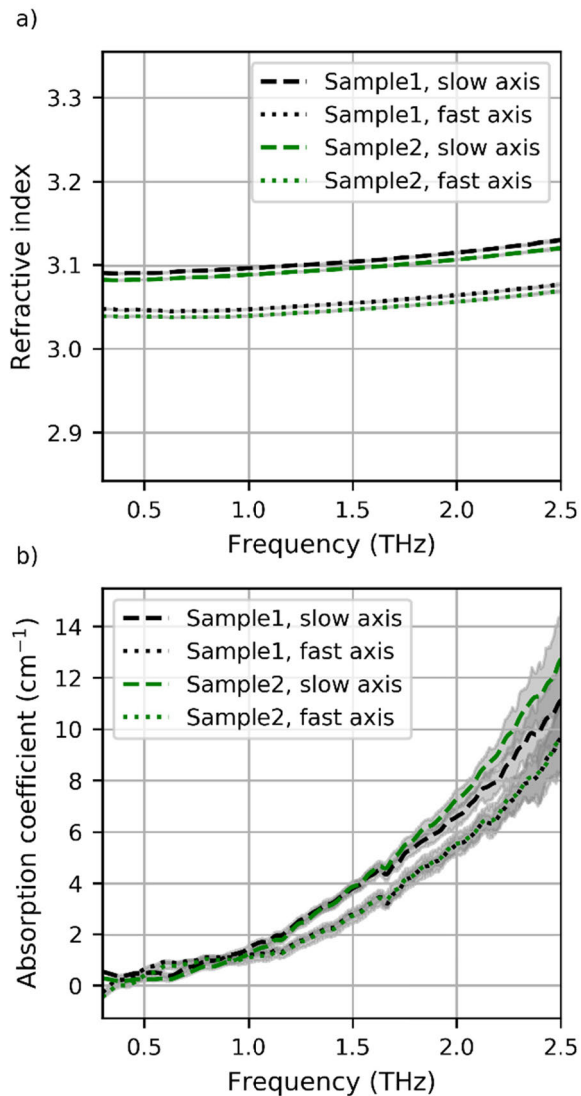


FIGURE 3. a) Refractive index and b) absorption coefficient of 3D printed Al₂O₃ along the fast and slow axis. The grey shaded area represents the measurement uncertainty due to the system instability. The corresponding time-domain and frequency-domain data as well as the imaginary part of the refractive index are available in the supplementary material.

the emitter antenna to the detector. Samples were inserted into a motorized rotational mount and placed between the last two OAPM before the detector, where the THz beam is collimated (Fig. 2). The reference measurements were taken with the rotational mount and retaining rings still in the beam path, however, without a sample in it. This was done, since the collimated beam size was greater than the sample size. During the measurements, the whole setup was flooded with nitrogen to minimize the absorption from water vapor present in the ambient air. The temperature in the lab was approximately 20.5 °C.

Preliminary investigation of the first sample revealed that the sample is birefringent and the approximate directions of the slow and the fast axes were marked. After a set of reference measurements, the sample was inserted into the

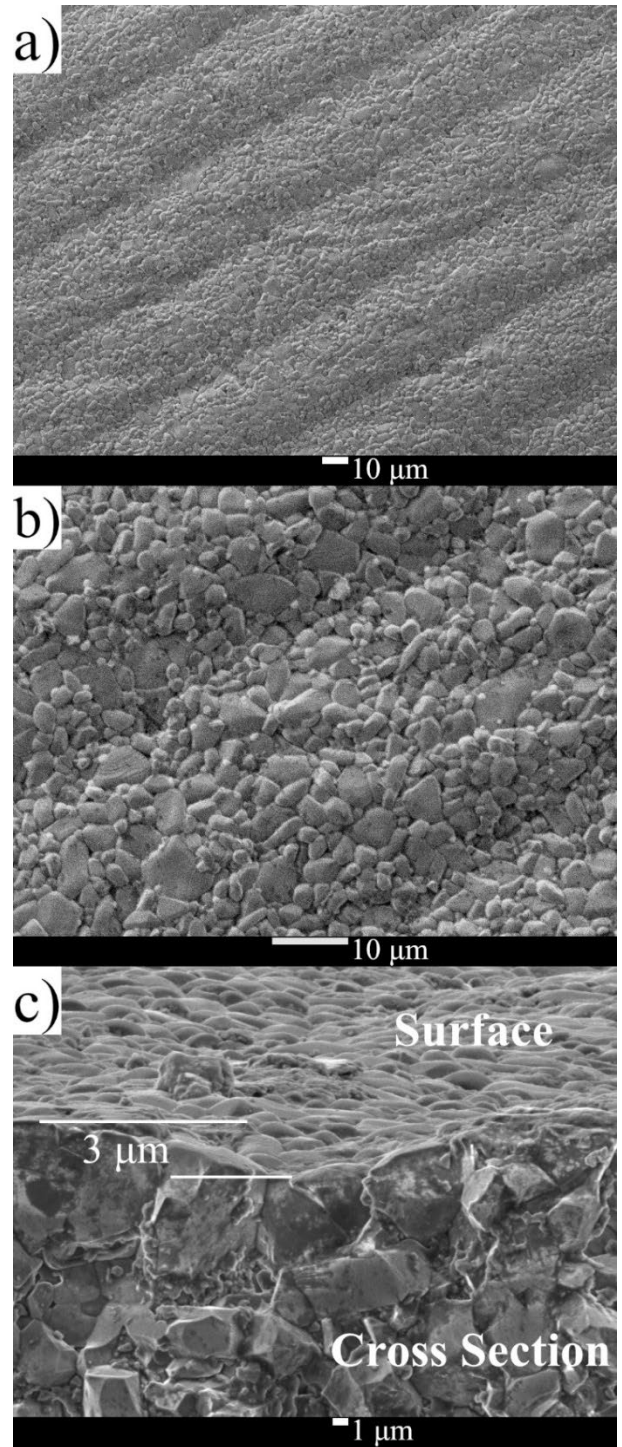


FIGURE 4. SEM images of the a) surface b) surface at higher magnification and c) the cross-section of 3D printed Al₂O₃ sample. The periodic surface structure resulting from the layer by layer additive manufacturing of the sample is approximately 3 μm thick.

rotational mount. The sample was orientated in such a way that at the initial position the two marked axes were either parallel or perpendicular to the polarization of the incident THz radiation. Then the sample was rotated for 360° in steps of 1° and for each angular step the sample transmission was

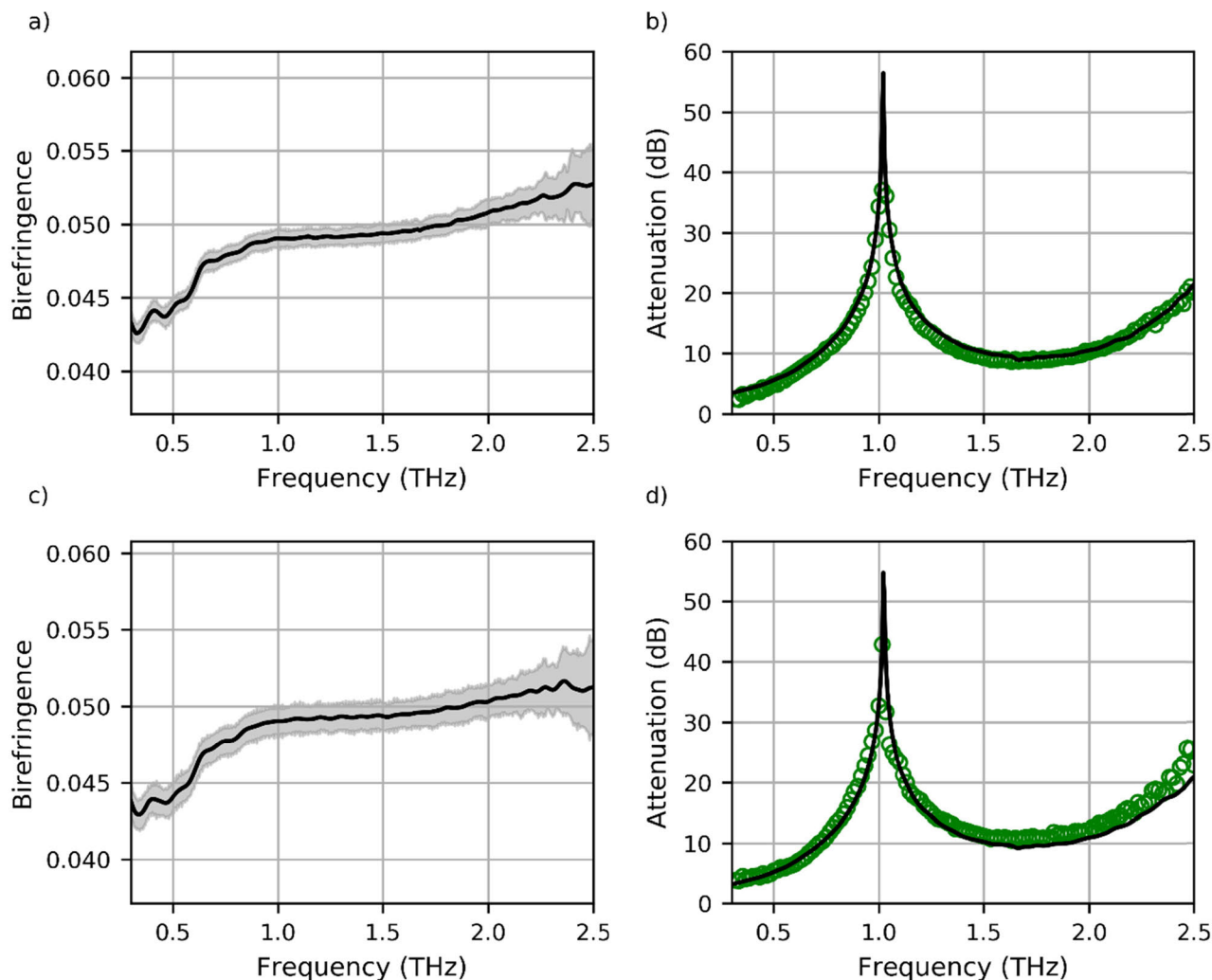


FIGURE 5. Samples 1 and 2 as $\lambda/2$ wave plate. a) measured birefringence and b) attenuation modeled and measured for sample 1 at $\alpha = 45^\circ$. c) measured birefringence and d) attenuation modeled and measured for sample 2 at $\alpha = 45^\circ$. The attenuation peak around 1 THz corresponds to the rotation of the polarization for 90° degrees due to the phase shift of π between the two polarization components. The green circles correspond to the measurement result and the black line to the modeled values.

recorded. With an analysis of these angular measurements, the orientation of the fast and slow axis was determined more precisely. Then the sample was remeasured ten times at orientations parallel to the two axes and directly afterwards ten reference measurements were performed. These data were used for the extraction of the refractive index and the absorption coefficient, which was performed using a commercially available software (Teralyzer).

The software is based on algorithms described in [45], [46]. For the evaluation of the refractive index and the absorption coefficient, the thickness of the sample needs to be determined, which was done in two steps. In the first step, the thickness of the samples was measured using a micrometer screw. This value was then used as a starting value in the second step, in which the Teralyzer software was used to further refine the thickness evaluation of the sample based on multiple-reflections of the THz radiation within the

sample using the quasi-space approach [46]. The thickness determined in this way was used for both orientations of the sample (i.e. polarization parallel to the fast and the slow axis). The determined thickness was $3000 \mu\text{m}$ and $2990 \mu\text{m}$ for sample 1 and sample 2, respectively, which agrees well with the design thickness of $3000 \mu\text{m}$.

As it will be discussed in the following section, the orientation of the two axes is related to the printing direction which could be determined from the residue of the printing supports (see Fig. 1 a)). Therefore, the second sample was mounted in such a way that the printing direction was approximately perpendicular to the polarization of the incident THz radiation. The measurements and extraction of refractive index and absorption coefficient were performed in the same way as for sample 1.

Following the THz TDS measurements, one of the Al₂O₃ samples was broken in half and its cross-sectional

microstructure as well as surface quality was evaluated using a Scanning Electron Microscope (SEM: JEOL JSM 7500F).

III. RESULTS AND DISCUSSION

Fig. 3 shows the refractive index and absorption coefficient along the fast and slow axis for both investigated samples. The grey shaded areas correspond to the measurement uncertainty due to the system instability, as calculated based on the ten repeated measurements.

For the case of sample 1, the refractive index increases with frequency from approximately 3.05 at 0.3 THz to 3.08 at 2.5 THz for the fast axis and from 3.09 at 0.3 THz to 3.13 at 2.5 THz for the slow axis. The same trend can be observed for sample 2, however, its refractive index was determined to be slightly lower compared to sample 1. This difference is attributed to the measurement uncertainty of the sample thickness, which is required for the parameter extraction from the measurement as described elsewhere [45], [46]. However, this sample thickness uncertainty does not influence the observed birefringence, since the same sample thickness was used when extracting the refractive index and the absorption coefficient for the slow and the fast axis of the same sample.

Both samples show low absorption coefficient values at lower frequencies ($\alpha < 2 \text{ cm}^{-1}$ at 1 THz). With increasing frequencies, however, the absorption coefficient also increases and exceeds 5 cm^{-1} at 2 THz. Minor differences between the determined absorption coefficient for the two samples can be observed, which are mainly within the measurement uncertainty as explained above. Yet, the diattenuation is mainly greater than the measurement uncertainty and can be observed for both investigated samples.

The determined values of the refractive index and absorption coefficient are in agreement with already reported values for ceramic Al_2O_3 [31]–[33]. However, the investigated 3D printed Al_2O_3 samples show birefringent properties, which is unusual for alumina samples. The direction of the slow and fast axis could be related to the printing direction (see Fig. 2), namely, the slow axis was found parallel to the printing direction and the fast axis perpendicular to it.

Illustrated in Fig. 4 are the results of an SEM study of our samples. Fig. 4 a) reveals a periodic surface structure, which is a consequence of the additive manufacturing layer by layer growth. Such periodic surface structures [47] as well as periodic layered samples [48] can cause a sample to be birefringent. However, in case of such a form birefringence, the slow axis would be perpendicular to the printing direction and the fast axis parallel to it, which is exactly the opposite to the measured birefringence. Therefore, this surface structure cannot be the reason for the observed birefringence. Furthermore, the expected birefringence from such a thin surface structure ($\sim 3 \mu\text{m}$, see Fig. 4 c)), is more than an order of magnitude smaller compared to the observed one. Therefore, we suggest a different origin for the observed birefringence.

As the influence of stress birefringence also seems unlikely, we suggest this effect to be a consequence of sample

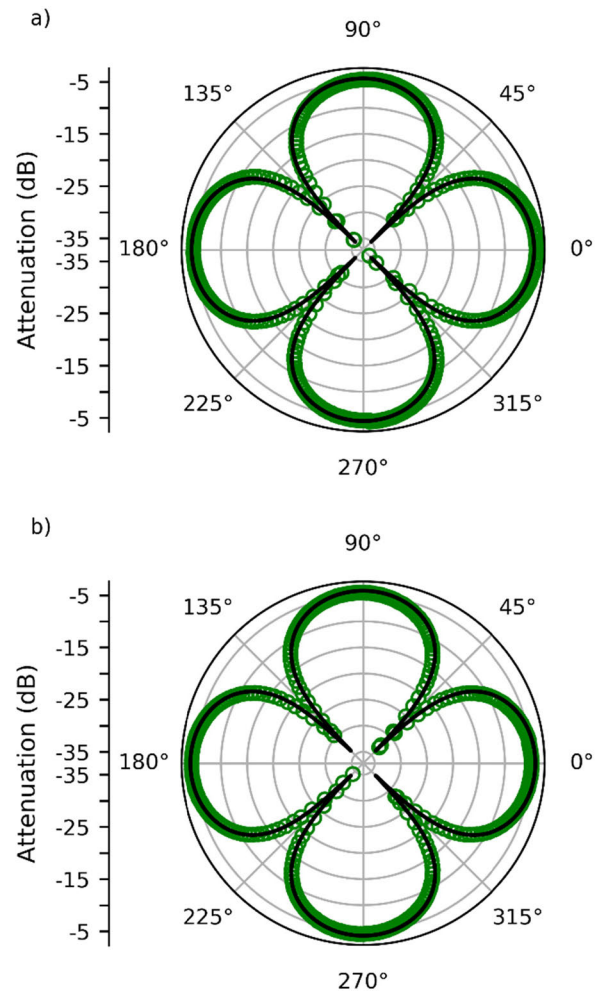


FIGURE 6. Angular dependent attenuation in the frequency range 0.98-1.04 THz for a) sample 1 and b) sample 2. The green circles correspond to the measurement result and the black line to the modeled values.

crystallinity with a preferred orientation. This may be the result of the layer by layer green body growth, as the orientation of the slow axis is parallel and the fast axis is perpendicular to the printing direction. We therefore hypothesize, that the printing and cross-linking mechanism results in a preferred crystallographic orientation, which manifests itself during the debinding and sintering step of the sample (Figure 4 b) and c)). As the printed samples are still polycrystalline, the observed birefringence of $\Delta n \approx 0.05$ is approximately a factor of 6 lower than the birefringence of Al_2O_3 single crystals ($\Delta n \approx 0.32$) as described by Kim *et al.* [34].

In the following, we use the birefringence effect of the printed Al_2O_3 ceramic to demonstrate a $\lambda/2$ wave plate as a first device made out of 3D printed Al_2O_3 working at approximately 1 THz.

Consider that THz radiation normally incidents on a sample with an angle α between the polarization of the incident electric field and the printing direction (slow axis) as depicted in Fig. 2. The incident electric field can be represented by two components, one parallel to the fast and the other to

the slow axis. If $\alpha = 45^\circ$ then the two components are of equal amplitude. However, they propagate with a different phase velocity through the material, resulting in a phase shift between the two components. The phase shift can be calculated as

$$\Delta\varphi = 2\pi df \Delta n/c_0, \quad (1)$$

where Δn is the birefringence, f the frequency, d the plate thickness and c_0 the speed of light. If the phase shift equals π or its multiple and the diattenuation is small compared to the total attenuation, the initial polarization of the light is turned for 90° and the plate acts as a $\lambda/2$ wave plate. Considering that only polarization along the direction of the incident polarization is detected, the attenuation through such a sample can be modeled based on the Malus' law as [48]:

$$A = -10 \log_{10} \left(T_{fast} T_{slow} \cos^2 (\Delta\varphi/2) \right), \quad (2)$$

where T_{fast} and T_{slow} denote the transmittance for the individual electric field components. The birefringence of the two samples as well as the modeled and measured attenuation at $\alpha = 45^\circ$ are shown in Fig. 5.

If a wave plate is placed under an angle different from $\alpha = 45^\circ$, the components along the fast and slow axis are not of equal amplitude. In such a case, the polarization is turned for an angle different from 90° even if the phase shift is equal to π or its multiple. The angular dependent attenuation in the frequency range between 0.98 and 1.04 THz is shown in Fig. 6.

IV. CONCLUSION

In this contribution we report the refractive index and absorption coefficient values of 3D printed Al₂O₃ in a broad THz range from 0.3 THz up to 2.5 THz. The refractive index is found to be greater than 3 in this frequency range and the absorption coefficient to be lower than 2cm^{-1} below 1 THz, which agrees with previous reports on ceramic Al₂O₃ [31]–[33]. Compared to polymers commonly used for 3D printing of THz devices, the 3D printed Al₂O₃ has significantly higher refractive index in this frequency range and a comparably low absorption coefficient with respect to the highly transparent polymers for frequencies lower than 1 THz [15], [27]. Furthermore, the investigated samples show birefringence ($\Delta n \approx 0.05$), which we attribute to a printing process dependent crystallinity of the LCM samples. As the birefringence of the 3D printed Al₂O₃ samples is related to the printing process, the birefringence might be tunable by adjusting the printing process parameters. Here we have employed the observed birefringence to demonstrate a $\lambda/2$ wave plate as a first device made out of 3D printed Al₂O₃ working at approximately 1 THz.

Consequently, the achieved material quality with the LCM technique in terms of a high refractive index ($n > 3$) and a low absorption coefficient ($\alpha < 2\text{cm}^{-1}$ at 1 THz), in combination with high resolution digital 3D printing, opens a variety of possibilities for the implementation of complex structures

and compact devices for the THz range. Exploring these possibilities as well as investigating the potential birefringence tunability of the 3D printed Al₂O₃ are part of ongoing research.

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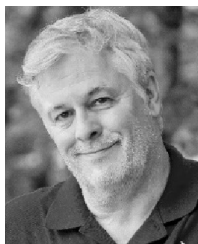
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