Advancing Industrial Imaging: Contactless THz Domain Spectroscopy for Topography and Chemistry

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Abstract: ***This research introduces a dual-functional contactless imaging technique based on terahertz time domain spectroscopy (THz-TDS) for simultaneous topographic and chemical analysis. The method was tested on pharmaceutical and food industry samples, providing accurate non-destructive measurements of thickness, roughness and chemical composition. The results demonstrate its potential for real-time quality control on industrial production lines.***

Keywords: THz time domain spectroscopy, contactless imaging, topography analysis, chemical composition, non-destructive testing

Surface topography, particularly to evaluate thickness or roughness, could be measured using many technics such as contact methods, including profilometer or AFM [1], as well as contactless methods, including optical profilometers [2]. AFM offers the highest level of accuracy for Z resolution in open space but it is limited to a surface that is limited to 100 µm x 100 µm [3]. Topographic optical imaging methods present higher interest for large surface measurements and application in industry production lines. The high demand in industry motivates strong research to increase the speed of acquisition and processing [4] to allow real time characterization of fast-moving samples, pellets of 1 to 5 mm thickness on conveyor for instance. However, optical profilometers do not provide any information about the chemical composition of samples. In this case, near infrared NIR [5], infrared IR [6] and Raman [7] imaging spectroscopies are widely used for spatially chemical analysis but they do not provide thickness or roughness of samples. For fast comparison of materials, using classical imaging spectroscopic technics such as infrared or Raman spectroscopies, it is mandatory to use only flat samples exhibiting the same thicknesses. However, this induces a really time-consuming process to prepare sets of identical samples, that is not compatible for continuous analysis in industrial conditions, for instance for tablets moving on conveyor. Terahertz (THz) imaging spectroscopy is an emerging method for fast material identification and is more and more applied in the fields of biology [8], detection of explosive, hidden objects and weapons [9], quality inspection of materials [10], and pharmaceutical [11] or food inspection [12].

This study addresses critical industrial needs for faster, more accurate quality control by integrating non-destructive methods. This study aims to develop and validate a contactless imaging method using THz-TDS spectroscopy in reflection mode to enhance industrial quality control by integrating topographic and chemical analysis. For reflection configuration, as shown in Fig. 1, the emitted THz beam is directed to the surface of a sample with an 8-degree incident angle and the reception antenna is placed symmetrically (cf Figure 1).

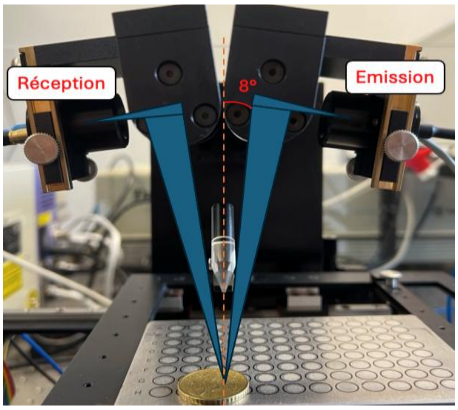

Figure 1: Picture of THz spectrometer with the sample in a reflection configuration

To validate our contactless topologic and chemical analysis, 18 pellets were prepared showing different thickness range (A: 1 to 1.5 mm; B: 1.8 to 2.1 mm and C: 2.2 to 2.4 mm) and variable concentration of maltose in polyethylene PE (0 (i.e., 100 % PE), 20, 40, 60, 80 and 100 % of maltose (i.e., 0 % PE)) (cf Figure 2).

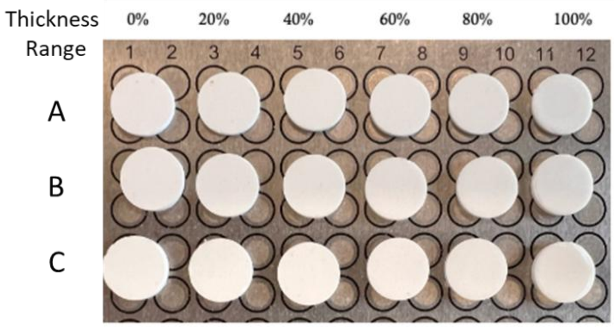

Figure 2: Picture of 18 pellets with different thickness range and ratio maltose/polyethylene.

We have recently reported that THz time domain spectroscopy could be used to measure thickness of samples without any contact. A theoretical representation of the THz pulse interaction within the surface of a pellet showing a thickness *d* is reported in insert Fig.3. The reflected beam at the surface of the sample goes back to the detector faster than the reference peak, i.e. the peak reflected at the surface of the metallic plate measured without any sample (Time of Flight measurement [13]). The Fig. 3 shows the reference peak at 0 ps (green curve) and the reflected signals at the surface of different samples presenting different thicknesses. Δt increases when samples get thicker (Δt= 2d.cos (θ)/c, with θ the incident angle and c the speed of light in air).

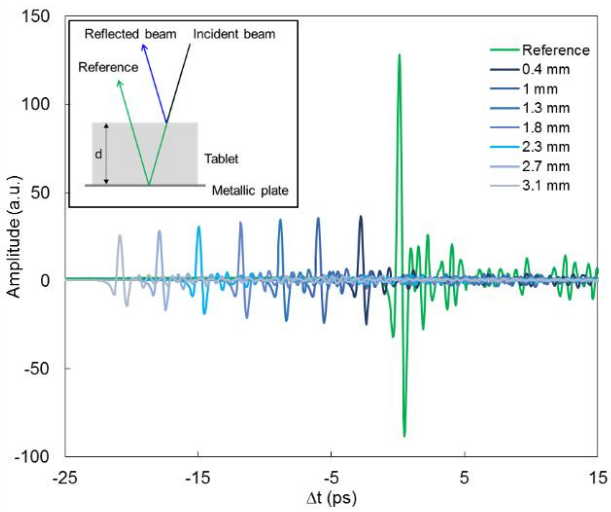

Figure 3: Amplitude of the THz time domain signals reflected on different thickness maltose tablets, and reference signal on the support plate (green peak). Insert: representation of THz pulse interaction with the sample in a reflection configuration

For comparative study, the thickness of the 18 pellets were also measured by classical micrometer (Palmer gauge).

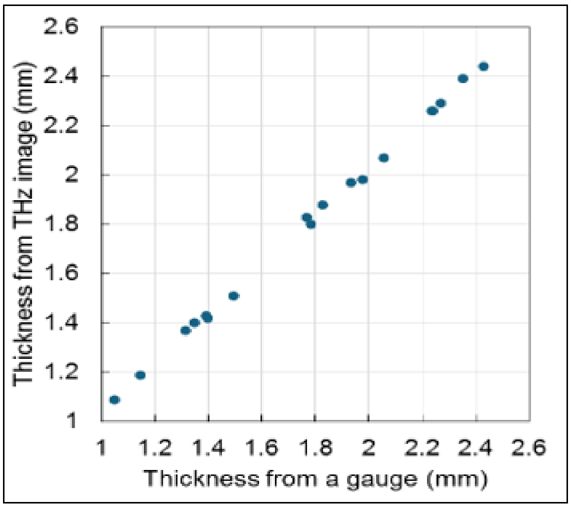

Figure 4: Comparison between the thicknesses extracted from the THz method and the one measured with a gauge

Fig.4 clearly demonstrates the correct correlation between the THz method and the classical mechanical thickness measurements for the 18 pellets. This motivated us to investigate a real topographic analysis of a model surface.

1) Topographic study

We have applied this THz time domain approach for a contactless topography imaging of a sample showing different pattern thicknesses (cf. Fig. 5, center). Using 3D deposition of polylactic acid (PLA), 4 squares of 10, 5 and 1mm side respectively (X and Y axis) and 4 different height (Z axis: 1000, 500, 100 and 50 µm) have been produced. In addition, the logo CROMA have been written in negative level (at – 500 µm). Topographic image obtained from the THz method shows clearly the different squares with positive heights (Fig. 5 left). This THz topographic method is also able to measure negative heights as shown in Fig.5 right.

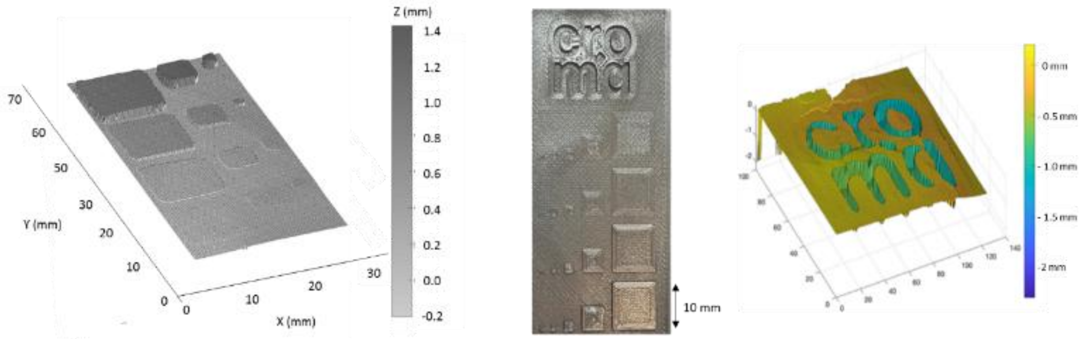

Figure 5: Picture of a 3D pattern fabricated by 3D-printing (center) and corresponding topographic image obtained from THz-TDS method (left for positive Z and right for negative Z)

The best X and Y resolution are limited by the diffraction limit and are around 100 µm [14]. However, the Z (height) resolution is limited by the time resolution of the THz pulse measurement and can theoretically reach 10 µm or less.

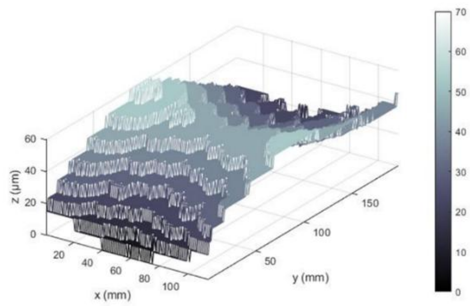

Figure 6: THz topographic image obtained from THz-TDS method with Z sensitivity.

Paper ID: SR241119135215 DOI: https://dx.doi.org/10.21275/SR241119135215

The topographic image in Fig.6 shows that different steps with 10 mm increments could be observed by the THz method, i.e., Δt measurement of each pixel to extract the corresponding d. Fig.7.

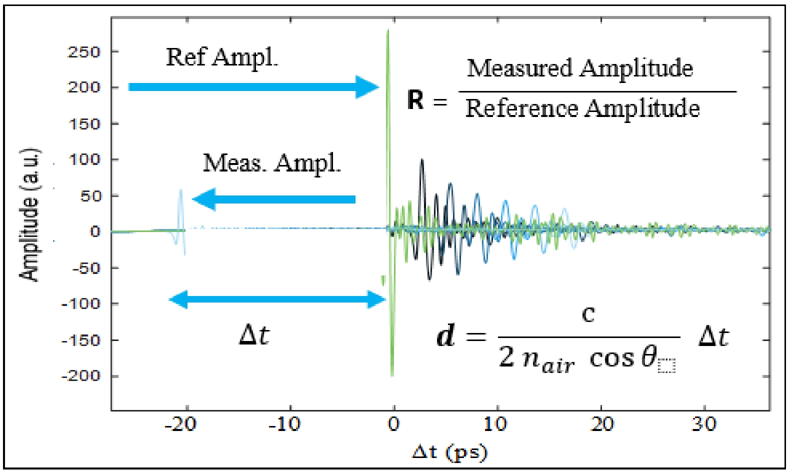

Figure 7: Extraction of height **d** of sample from Δt and reflection coefficient **R** from amplitude obtained from THz-TDS method

Classical contactless optical microscopes dedicated to 3D surface analysis can also give similar thickness measurements than our approach but do not provide chemical composition details.

2) Chemical analysis study

The reflection coefficient of the THz signal at the surface of the tablet depends on the effective refractive index of the material (by Fresnel equation). Since in mixtures, this effective refractive index vary with the proportion of the compounds in the tablet, it is then possible to extract the proportion of the components of a mixture, by measuring the reflection coefficient of a given samples, i.e., the amplitude of the THz signal peaks [15]. Fig.7. After correction to correct the misalignment of the THz detector which is induced by the different thickness of samples [16], the reflection coefficient can be directly determined for each area from the ratio of the measured amplitude (at the surface of the sample) and the reference amplitude (without sample). There is effectively a linear correlation between the reflection coefficient and the composition of samples, i.e., the ratio between maltose and PE. Fig. 8. At the difference of 3D optical microscope, the THz time domain topographic approach allows also to discriminate samples according to their global chemical composition.

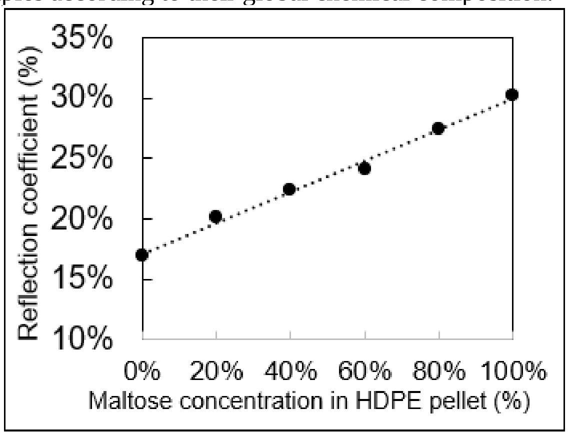

Figure 8: Linear relation between reflection coefficient R and concentration of maltose. obtained from THz-TDS method (right)

Fig.9 is the reconstructed image of the maltose proportion evaluated by the THz method, applied at each pixel for the 18 pellets. The different yellow-blue scale levels are related to the difference of ratio maltose/PE for the different tablets along the X axis from 0 % to 100 % of maltose. For each column, images of pellets are quite similar, due to the same concentration when only the thickness is changing. This new topographic and chemical composition based on THz time domain approach also allowed to visualize the heterogeneity of some tablets, top right tablet for instance.

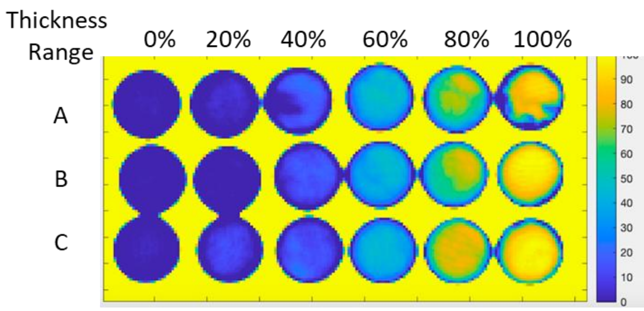

Figure 9: Image of the chemical composition and homogeneity/heterogeneity of 18 pellets from the THz-TDS method.

At the difference of 3D optical microscope, this contactless THz approach allows to discriminate samples in one shot, in order to quickly reject tablets that do not have the correct thickness, chemical composition and/or homogeneity of the repartition of active component versus excipient. This study successfully demonstrates the application of THz-TDS spectroscopy for industrial imaging, providing non-destructive and accurate topographic and chemical analyses. The results highlight its potential for real-time quality control in production lines, offering significant improvements over traditional methods. Future research should focus on expanding its capabilities for complex materials.

Acknowledgment

This work was supported by the Science for Peace NATO Grant number GS-5973 and the Région AURA Grant "AUTHANTIC". O. Lavastre thanks the CNRS (National

Center for Scientific Research, France). All the THz measurements have been performed on the characterization platform PLATERA (https://www.platera.tech/) co-financed by AURA region.

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