Terahertz Paint Thickness Measurements: from lab to automotive and aeronautics industry

M.Picot, H. Ballacey, JP. Guillet, Q. Cassar and P. Mounaix

Laboratoire IMS- UMR 5218 CNRS, Université Bordeaux, 33405, FRANCE patrick.mounaix@u-bordeaux.fr.

Abstract

For terahertz sensors to enter the industrial sector, idealized measurements on simplified samples are not sufficient. It must be efficient on any type of substrate. Then, a noncontact sensor to determine the individual thicknesses of multiple paint layers is highly recommended. We report on development of terahertz systems for measuring the thicknesses of multiple paint films for non-contact quality control. The principle of operation is comparable to pulsed ultrasonic imaging but uses broadband terahertz-frequency pulses to which many materials are partially transparent. Then, analysis of time-domain data is described, and the results of measurements made on real industrially-applied structures containing up to five layers found in both automotive and aeronautic industries are presented.

Keywords: terahertz, multi-layer thickness, coating thickness inspection, non-contact

1 Introduction

The thickness of paint films is one of the most critical quality parameters in the paint process for automotive or aeronautics. Then, it's a critical limitation amongst the most essential quality parameters in the paint process. The thickness of individual paint layers is a critical factor in determining their capacity to appropriately satisfy their functional and other requirements. Paint upper and lower film thickness limits must be met to guarantee those necessities, for example, film functioning, appearance, cost and natural effect are all the while fulfilled. Each paint layer is deposited with the expectation that when film organisation is finished at the end of the process, each painted layer will present a thickness inside determined target limits defined by the designers. Consequent safe testing of the dry film thickness is performed to guarantee the individual layers have the right optical and geometrical properties. Numerous measurement techniques exist for quantifying coated film thickness but many are unsuited to deployment in industrial environments or cannot meet the requirements of real-time quality control. With the introduction of robust, turnkey systems in recent years, time-resolved terahertz pulsed sensing has matured sufficiently to find application in providing quantitative analysis of physical properties to a range of industries, including semiconductor package inspection [1] and non-destructive testing of larger scale composites, (e.g. for wind turbine materials development) [2].

2. Technique

For many applications, layer thickness determination can be accomplished using a simple time-offlight approach, whereby the thickness of individual layers can be estimated from the successive reflections from front and back interfaces of each layer. This may be applicable to thick paint layers where the interface reflections are well separated in time, as in marine paint systems [3], but modern automotive and aircraft paint systems can involve the application of thin paint layers, making accurate identification of individual interface reflections difficult. For aeronautic industry, paint is more than aesthetics; it affects the weight of the aircraft and protects also the integrity of the airframe. The top varnish is applied to protect the exposed surfaces from corrosion and deterioration. Also, a properly painted aircraft is easier to clean and maintain because the exposed surfaces are more resistant to corrosion and dirt, and oil must not adhere as readily to the surface.

Many materials that are opaque at visible and other wavelengths appear partially transparent to terahertz frequency light, making it ideal for use in non-destructive testing applications. Furthermore, the use of broadband terahertz generation and detection makes possible time-of-flight measurements through such materials, as illustrated in Figure 1 for a single semi-transparent layer on a perfectly reflecting substrate. When a broadband pulse of terahertz light is incident on an interface between two adjacent materials of different refractive index, the incident light is split into a forward propagating transmitted component and a backward travelling reflected component. Time-gated detection gives very accurate values of the time at which reflections (from this and any subsequent interfaces) occur. This coherent detection scheme makes possible the acquisition of a time-domain trace containing reflections from the various interfaces within the object under test. Converting the units of time into distance yields information on the depth at which reflections occur, thus allowing for thickness determination of layered media.

In the case of a multi-layer object, the measured reflected signal is a superposition of individual reflections from each interface, where the position and amplitude of reflections in the measured time-domain waveform are dependent on the optical properties and thickness of each layer. The separation between reflections, from the front and back surfaces of a single layer, is relative to layer thickness and inversely proportional to refractive index. The polarity and strength of reflection indicates the relative change in refractive index between adjacent layers. Clearly, the greater the reflection, the greater the change in refractive index across an interface between two adjacent layers. In the case of thin layers, reflections from subsequent interfaces can be closely spaced thus making the task of detecting individual reflection peaks non-trivial.

Here, we must use a combination of numerical simulation and optimization in order to determine parameters that best describe reflections from a given multi-layered paint sample. The reflected terahertz time domain waveform is simulated using a matrix formalism of Fresnel's equations, where each paint layer is represented by a set of numerical parameters that describe both the thickness and the optical response, described by a complex-valued, frequency-dependent refractive index, of each layer. Numerical optimization is then employed to find a set of values for those parameters that best fits the measured waveform in a least-squares sense



Figure 1 : Illustration of the reflected time domain waveform from a single layer on a metallic substrate. The multiple reflections produce multiple peaks in the reflected signal, the amplitude and spacing between peaks depending on the optical thickness of the layer .

Measurements were made using two terahertz systems: The TeraView's TPS 4000 and TeraView's TeraCota instrument (TeraView Ltd., Cambridge, UK), which has been specifically designed to measure coating thickness. It could be integrated in both hand-held and robotically positioned tools for industrial applications. The 100-mm focal length gives sufficient depth of field to ensure that layers remain in focus, and a spot size small enough for moderately curved surfaces to be considered locally planar, while large enough to cope with inhomogeneity of basecoat pigments. TPS Spectra 4000 includes two remote fiber heads and it could be implemented in a reflection or transmission configurations.

3 Results

Successful coating thickness extraction relies on a robust calibration method. Calibration is performed on sample training sets. Once the calibration process has been performed, the system can extract thicknesses from new samples containing the same paint layer combination. These waveforms are from samples that contain each of the mentioned paint



layers in isolation on aluminium, or if instead each subsequent waveform in the sequence corresponds to a sample that contains an additional paint, e.g. is waveform fig2. (f) from Clearcoat-aluminium only, or from a panel containing all mentioned paint layers.



Figure 2: Time domain reflected terahertz waveforms measured from 1: a) aluminum substrate, b) primer, c) external primer, d) intermediate primer, e) basecoat, f) clear coat g) description of the measured sample

We are also working on aeronautical paints to apply the technique to other industrial fields. Four or five individual paint layers are applied, different primers (for surface levelling and protection), basecoat(s) (containing coloured and/or iridescent pigments and/or metallic flakes), and clear coat (to enhance the desired finish and to protect against mechanical damage and UV exposure). For automotive industry, an initial electro coat is deposited for corrosion protection.

Typically, the layers could have a wide range of thicknesses: the minimum and maximum thicknesses for individual layers were on the order of 5 μ m (primer) upto 80 μ m (clear coat), respectively. The paint application method also varies from customer to customer, while the remaining layers are sprayed (the

primer either as a powder or liquid, and the clear coat and basecoats wet-on-wet). While the mid-coat has a thickness that would be below the instrument resolution (less than $10\mu m$) if it were isolated, it is even detectable in the film build thank to the interface effect onto the final signal.

To validate the instrument performance, we will compare measured thicknesses with those produced by established instruments. Total layer thickness was verified using an eddy current gauge and optical contactless profilometer. Individual layer thicknesses were also measured by profilometry.

Firstly, we have to simulate and to check the consequences onto the temporal profile of each new paint layer results. Although a simple analytical approach has been developed [4] to predict the peak separation to perform time-of-flight measurements, such an approach is limited to relatively optically thick paint layers. The measurement calculations in Figure 3 highlight the limitations inherent in analysing properties of reflection peaks alone – a task that depends on individual layers producing a single corresponding reflection peak and which may not be possible for three reasons.

1: Individual layers may not be optically thick enough to produce a reflection peak that can be clearly distinguished from neighbouring peaks all the reflections signal is mixed into the main peak. Depending on the thickness and refractive index of individual paint layers, one or more shoulders in the reflected waveform can emerge from the metal substrate below (see fig.2d, e).

2: For two neighbouring layers that are optically very similar (with similar refractive index profiles), a reflection from the lower layer will be relatively weak or absent. The only noticeable difference is an increased separation between the reflection peaks from the surface of the layer and the metal substrate or a drift of the substrate peak with respect to the measurement the metal substrate alone.

3: Time-of-flight measurements fail to account for absorption and scattering by individual layers, which are usually frequency-dependent terms. They can affect the frequency amplitude over the spectrum and then lead to a significant attenuation of the time domain waveform.

To extract the terahertz response of individual layer, we simulate and compare to measured data the impact of the main parameters onto the waveform, namely the thickness and the refractive index. For clarity, we present the results obtained for a single primer layer on a metallic substrate.





Figure 3 a) Influence of changes in refractive index on terahertz signal, insert: zoom on the first peak

b) Influence of changes in layer thickness on terahertz signal, insert: zoom on the first peak



We can observe the dramatic influences of both parameters. Each peak is strongly affected in amplitude and in temporal position when we varied the thickness in the $10\mu m$ range. To accelerate the convergence of the numerical procedure, we can constrain first the thickness to a reasonably range and converge to an optimal optical index using a non-linear least squares fitting routine in frequency domain and in time domain in a repeated sequence, then, we optimize the thickness and iterate until the best correlation is found. At last, we add linear or non-linear absorption to adjust the time domain amplitudes of the entire signal since it affects mainly this parameter.



Figure 4: calculation and measurement of the reflected time domain waveform

Figure 4 represents the calculation and the measure signals of a measured primer layer deposited on a metal substrate. That corresponds to the measured and calculated data of Fig.3. In that case, we found a thickness of 25μ m and a constant complex-valued refractive index of 1.90 + i0.06. We establish a very good agreement between the calculation and the measurementin amplitude and in phase as shown in Fig.4. We translate in X and Y the two curves for clarity. So, this procedure is repeated for each layer deposited onto a substrate. Calibration of the layers of a multi-layer paint stack is a starting point to determine the refractive index profile of each paint type. Numerical simulations have been developed to predict how terahertz is refracted and absorbed by that multi-layered material. For numerical optimisation to return reliable refractive index profile values, the thickness of the paint layer at the point on the paint surface where the terahertz measurement was made needs to be optimized.

Then, full-stack samples of the paint process could be tested. The results presented in Fig.5 were performed on automotive multilayers samples [6]. Two sets of test panels were prepared: one with a solid basecoat and the other a metallic basecoat. The thickness prediction is performed while supposing the refractive index of each layer is fixed and individual thicknesses are variable parameters for fitting.





Figure 5 : Stacked bar charts of individual expected and measured paint layer thickness for test panels 1 to 3 containing solid basecoat (left), pearlescent basecoat (centre) and a 3-stage top coat (right), **Top row**: thicknesses of all paint layers in each system; **bottom row**: thickness of the top coat only (clear coat plus basecoat).

Overall, the predicted thicknesses are calculated and we found very good agreement with nominal thickness given by Eddy current. New generation of paint meter are tested by industrials partners on site to test the reproducibility and the stability with time.

Conclusion

In this work, we demonstrate that time domain terahertz imaging is capable of measuring the thickness of individual layers on multiple paint system for automotive or aeronautics industry. Since the terahertz reflection from a substrate material can be simulated by attributing optical properties in the same manner as is done for paint layers, the technique can also be used for non-metallic substrates that are not perfect reflectors. The flexibility of the terahertz systems can easily take into account any change made to an individual paint layer within a multi-layer paint stack (e.g. due to a change in paint formulation), because the calibration process is rapid and can be achieved using the same instrument, without having to send samples away for independent calibration. The results presented in this work are from measurements made on painted steel and aluminum substrates, which are considered perfect reflectors. Future work for recent composite aircraft panels will include testing non-metallic substrates to extend the applicability of our terahertz instrument.



References

- [1] M.Y. Tay et al, "Advanced fault isolation technique using electro-optical terahertz pulse reflectometry", 19th IEEE International Symposium on the Physical and Failure Analysis of Integrated Circuits (IPFA), pp 1-5, Jul 2012.
- [2] J.W. Park, et al, "Terahertz radiation NDE of Composite Materials for Wind Turbine Applications", *Int. J. Precision Engineering and Manufacturing*, vol. 15(6), pp. 1247-54, June 2014.
- [3] W. Tu, S. Zhong, Y. Shen and A. Incecik, "Non-destructive testing of marine protective coatings using terahertz waves with stationary wavelet transform", *Ocean Engineering*, vol. 111, pp. 582-592, January 2016.
- [4] Tae-In Jeon and D. Grischkowsky, Characterization of optically dense, doped semiconductors by reflection THz time domain spectroscopy Appl. Phys. Lett. 72, 3032 (1998)
- [5] H. Balacey, B. Recur, J. B. Perraud, J. B. Sleiman, J. P. Guillet, and P. Mounaix, "Advanced Processing Sequence for 3-D THz Imaging," IEEE Transactions on Terahertz Science and Technology, vol. 6, pp. 191-198, 2016.
- [6] R.K. May, I.S. Gregory, TeraView Ltd, Cambridge UK," Automotive Paint Thickness Analysis by Terahertz Sensing", 12th Coatings Science International, 2016, Amsterdam NE

