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## Scanning optical coherence tomography applied to the characterization of surfaces and coatings

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### Abstract

This paper shows the technique of optical coherence tomography in the frequency domain (OCT-FD) to obtain surface topography and tomography of surface coatings. The setup is based on a superluminescent diode centered on 849 nm with an spectral wide of 50 nm as the light source, a single-mode fibre Michelson interferometer and a spectrometer as the detector system. This scheme allows having compact and robust equipments that was designed to be used in an industrial environment. With this system it can be obtain a tridimensional image acquired point by point with a maximum range of 20 mm width and length and 2 mm depth. Images obtained from the topography of metal samples and tomography of polymer based coatings is presented. Also it is shown that is possible to obtain, from these images, parameters of interest of the sample, such as roughness, flatness and characteristic distances. As an example to show the potential of the method the dimensions of a metal surface modified after laser ablation were measured from a topography image obtained with this technique. A second example presented was the image obtained of a roughness pattern made on a metal surface. Finally it was presented a tomography image of a modified polyurethane coating deposited on a metal surface. Thickness and refractive index of the coating were obtained after the image analysis.

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## 1. Introduction

The characterization of surfaces and coatings with high spatial resolution using techniques to obtain the tomography or the topography of a sample is a topic of interest for many applications in the area of materials. Among the great variety of available techniques, the optical interferometry methods have been traditionally used because of they offer high spatial resolution combined with non-contact with the sample. In the last years there have had a great development of this techniques based on low coherence interferometry, Leach et al. (2011) or coherent interferometry scanning (CSI), Ferraro et al. (2011). There is a set of experimental devices based on this principle which are commonly known with different names in the literature by Huang D. et al. (1991). In this report we employed one of these techniques, that is named optical coherence tomography (OCT) and offers the possibility to obtain 3-D images of the surface (topography) or of the inner of a material (tomography), in an analogous way to ultrasound, but using light. Since 1991, the technique has been developed in different fields of application, Huang D. et al. (1991), mainly in medicine Drexler et al. (2008), particularly in the fields of ophthalmology, cardiology and oncology by Bouma et al. (2002). In the industrial area, its application is more recent and has relevance in the analysis of surfaces and shapes and the study of the interior of various transparent materials.

In this paper the objective is to develop equipment, aimed at the characterization of surfaces and coatings. Although there are commercial systems that offer similar performances like say Vakhtin et al. (2002) and Stifter et al. (2008), they generally requires limited samples sizes and special conditions (insulation in vibration, environmental conditions, mechanical isolation, etc.). The aim of this work is to design a setup that can be used in industrial applications, suitable to perform measures in different environments from a laboratory or metrology room up to a production line. So the design is able to measure “in situ”, with a robust and stable structure and with a setup configuration that can be adapted to different ambient and in a limited space.

There are different experimental schemes for the implementation of OCT but the basic idea in all cases is similar and uses the interference of two light beams (one from the sample and another from a reference surface), Jayaraman et al. (2011). Analyzing the interference pattern generated due to optical path difference, it is possible to obtain images by scanning point by point the sample. Throughout this paper, we refer to topography, when the image is obtained with light reflected only from the surface of the sample and to a tomography, when the image is obtained with light coming also from reflections at internal interfaces.

### 1.1. OCT in the frequency domain

In this paper the experimental setup was designed to use OCT in the frequency domain (OCT-FD) like Hu et al. (2003) and Jayaraman et al. (2011) so the spatial resolution and maximum depth range are mainly defined by the spectral width of the light source and the detector characteristics (an spectrometer), Rabiner et al. (1975). With our experimental scheme, the axial and lateral spatial resolution is in the order of 1 micron, while the range of depth is 2 mm. The technique uses a Michelson interferometer where the test sample is placed in one arm and a mirror, used as a reference surface, in the other. A general scheme is shown in Fig. 1.

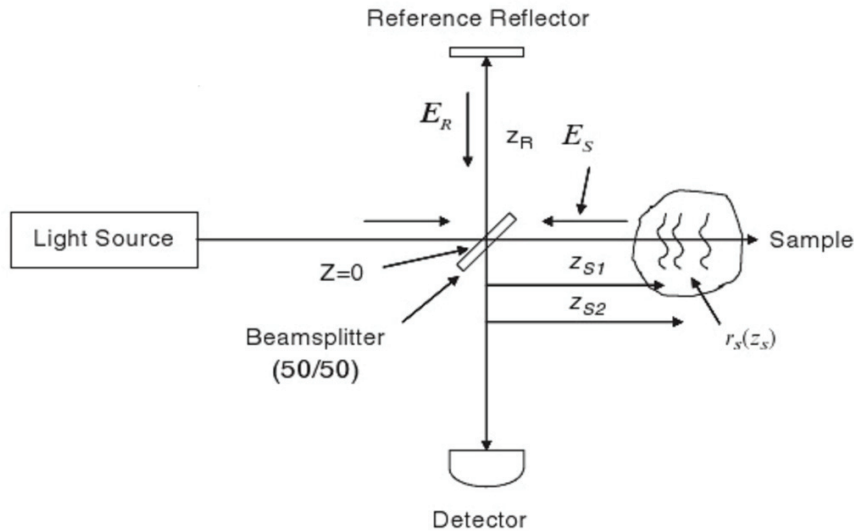


Fig. 1 Scheme of general OCT setup

In this configuration the arms remain fixed, avoiding displacements needed in other variants such as in the OCT-Time Domain. To obtain the reflectivity profile of the sample the spectrum of the light source is measured and the optical path difference of each measured point is obtained after an Fourier analysis of the interference signal.

Assuming a Gaussian intensity profile to the incident beam  $S(k, \omega)$  (where  $k$  and  $\omega$  are the spatial and temporal frequency) and that the sample has a set of  $N$  interfaces, it can be shown that Fourier transformation of the intensity of the light spectrum measured with the spectrometer ( $I$ ), as a function of the position of the surface reference and the position of the interfaces in the sample, is given by the Eq. 1:

$$\begin{aligned}
 I(z) = & \frac{\delta}{8} \left[ \gamma(z) \left[ R_R + R_{s_1} + R_{s_2} + \dots \right] \right] + \dots \\
 & \frac{\delta}{4} \sum_{n=1}^N \sqrt{R_R R_{s_n} \left[ \gamma \left[ 2(z_R - z_{s_n}) \right] + \gamma \left[ -2(z_R - z_{s_n}) \right] \right]} + \dots \\
 & \frac{\delta}{4} \sum_{n \neq m=1}^N \sqrt{R_{s_m} R_{s_n} \left[ \gamma \left[ 2(z_{s_n} - z_{s_m}) \right] + \gamma \left[ -2(z_{s_n} - z_{s_m}) \right] \right]}
 \end{aligned} \quad (1)$$

Where  $\rho$  is the detector response factor,  $\gamma(z)$  is the Fourier transform of  $S(k)$ ,  $R_{s_i}$  and  $R_R$  are the reflectivities respectively of the interfaces of the sample and the reference mirror, while  $z_{s_i}$  and  $z_R$  are its corresponding positions.

## 2. Experimental Setup

The experimental setup used is shown in Fig. 2. It is a Michelson interferometer mounted on optical standard single-mode fibre. The light source is a superluminescent diode with a spectrum centered at 849nm and a spectral width of 50 nm. The focusing system on the sample is a microscope objective (Olympus 10X) used to collect light reflected from the sample. The optical system and the sample are mounted in a positioning stage which allows automated controlled sweeps. The detector is a spectrometer (HR4000 OceanOptics) with a linear array of 2048 pixels.

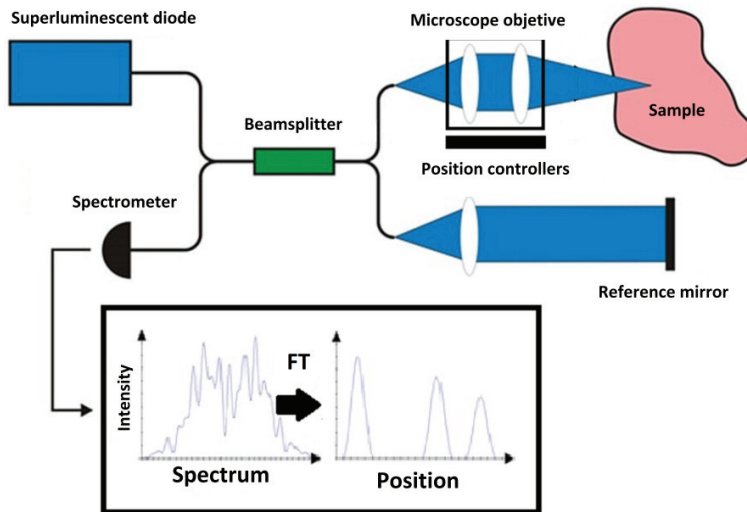


Fig. 2 Experimental setup. After detection, the Fourier transform (FT) of the interference signal provides the position of each interface in the sample.

Once the interference signal is acquired, the Fourier transform is performed to obtain different values of optical path difference. The spatial resolution is improved using the transformation Chirp Z in the Fourier analysis by Rabiner et al. (1975).

The system presented allows obtaining three-dimensional images of up to 2cm (width) x 2cm (length) x 2mm (depth). Although the same system could have better spatial resolution, is a priority in the design to obtain a robust and compact device, even if it means sacrificing a higher resolution (see subsection 3.1). The system was proven in different materials as metal surfaces, lubricant films, multilayer polymer films, glass slabs, among others. Some of these results are shown in the following sections

### 3. Experimental Results

The experimental system supports two configurations, one in fiber optics and other one in air. To characterize the axial spatial resolution in both configurations a set of measurements were performed that resulted in a standard deviation of 0.02  $\mu\text{m}$  (air) and 0.41  $\mu\text{m}$  (fiber). We attribute the difference to the optical fiber instabilities (variation in the refractive index by mechanical stress or temperature changes.). Results presented in the following sections were obtained mainly with the fiber optic system for the reasons mentioned above.

#### 3.1. Surface topographies

In Fig. 3 it is shown a first example in which it is measured the dimensions of a crater produced after laser ablation on a metal surface. A scan of 2.56 (width) x 3.00 (long)  $\text{mm}^2$  with steps of 0.02 mm was performed. Fig. 4 shows an enlargement of the image obtained with details of the topography of the crater. The images were processed with software developed specially for this application.

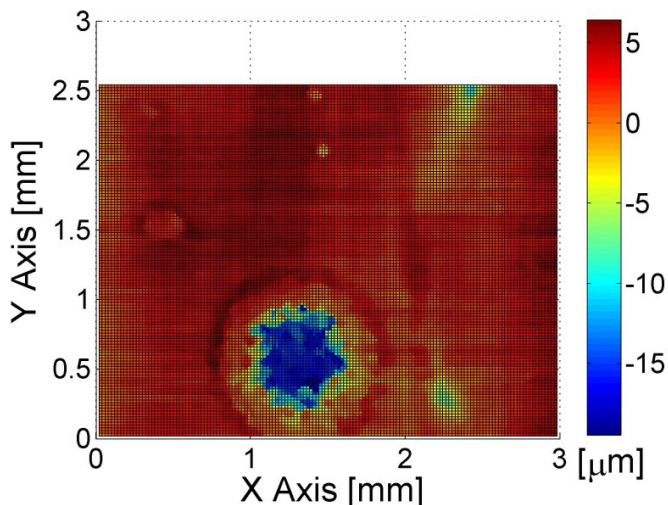


Fig. 3 OCT image of a crater on a metal sample..

From this image it was determined that the crater had an elliptical shape with the following dimensions:  $(720 \pm 40) \mu\text{m}$  of larger diameter,  $(540 \pm 40) \mu\text{m}$  of smaller diameter and an average of  $(16 \pm 1) \mu\text{m}$  of depth. Also, it was determined the dimensions and shape of a ring produced on the crater edge, as a consequence of the laser ablation.

The values obtained were consistent when they were compared to those measured with a commercial profilometer.

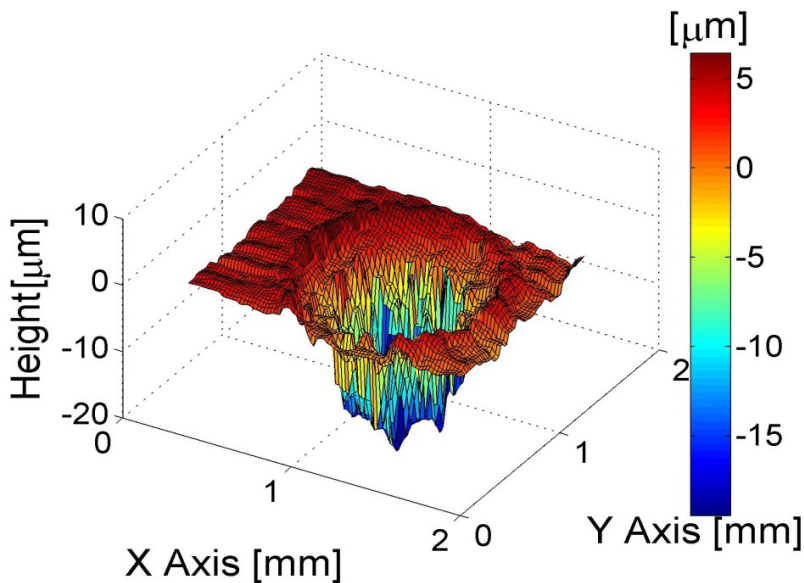


Fig. 4 3D image generated from the topography showed in figure 3.

Another example is shown on Fig. 5 where a topography of a roughness standard surface of electroformed solid nickel was measured. The surface has two distinct areas which we call Zone 1 and Zone 2, separated by a step of  $38 \mu\text{m}$ . To obtain the image a scan of  $0.35 \text{ mm} \times 5 \text{ mm}$  with steps of  $50 \mu\text{m}$  (y-axis)  $\times 5 \mu\text{m}$  (X-axis) is performed. In

zone 1 a non-repetitive ripple with an approximate amplitude of  $1\ \mu\text{m}$  and  $500\ \mu\text{m}$  separation is observed, while in zone 2 a triangular roughness with an average peak amplitude of  $(21.5 \pm 0.9)\ \mu\text{m}$  and a separation between peaks of  $(134.2 \pm 0.8)\ \mu\text{m}$  is observed (See Fig. 5).  $R_a$ , the roughness parameter, was calculated from the image generated of zone 2. The value obtained was  $6.7\ \mu\text{m}$  against the value declared by the surface manufacturer that is  $6.0\ \mu\text{m}$ .

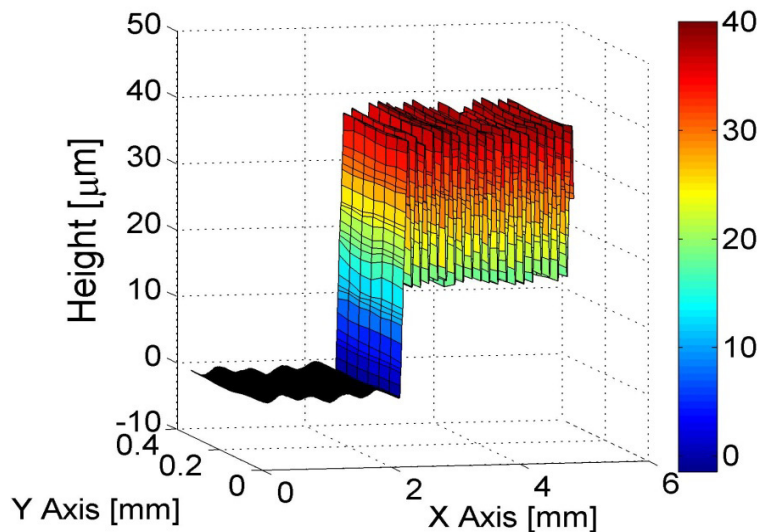


Fig. 5 OCT image of the topography of a roughness surface pattern.

### 3.2. Coating tomography

In this example a transparent film obtained from a water-based polyurethane dispersions was evaluated. While water was evaporating and the film was forming, images of the tomography of the sample were obtained. In this example a scan of  $0.9\ \text{mm} \times 4\ \text{mm}$  in steps of  $50\ \mu\text{m}$  was performed. Analyzing the light reflected from the sample it is possible to distinguish two zones. In zone 1 there is only a single reflection which corresponds to the metal surface uncoated (A) while in zone 2 there are two reflections to analyse, one is reflection (B) that corresponds to the air-film interface while the second reflection (C) corresponds to the film-metal interface. It is interesting to note that the reflection inside the polymer (C) is below the level of the substrate, that means a longer optical path produced by the refractive index of the film. The image shown in figure 6 represents the situation of a dry film, after all water was evaporated. The thickness ( $d$ ) can be calculated from the profilometry of the sample (B and A). An average thickness  $d = (60.3 \pm 0.4)\ \mu\text{m}$  was obtained. At the same time using the difference between the external (B) and internal reflections (C) the relation  $nd$  was measured, and the refractive index  $n = (1.482 \pm 0.017)$  was obtained.

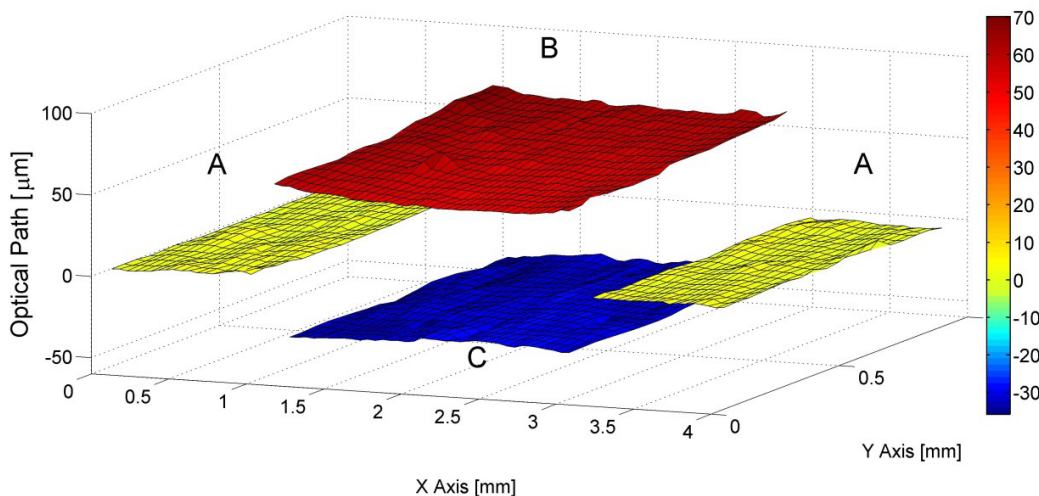


Figure 6 – OCT image of a polymer coating on a metal surface

#### 4. Conclusion

It was presented in this work an optical system based on low coherence interferometry to obtain topographies of surfaces and tomography of coating materials. The system was based on optical coherence tomography in the frequency domain (FD-OCT). The experimental results were obtained with a system developed in the laboratory and specially designed with the possibility of being converted into a device for industrial use and that can support an aggressive environment. Another important characteristic is that the optical head (optics close to the sample) can be mounted in a reduced space and in different configurations for "in situ" measurements. The characteristics in terms of spatial resolution, dynamic range of distances and sampling frequency discussed in this paper are by no means limitations of the technique, and can be improved with more sophisticated equipment. The examples were selected to show the potential of the method mainly in applications where it is necessary to obtain information simultaneously from the surface and from the inner of the material. Future work will aim to optimize the information obtained by measuring the optical path difference and its relationship to changes in the material. Mainly changes in the refractive index and its relation to structural changes and the temporal evolution of the processes of formation of coatings and thin films on various substrates.

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