

# Spatially-resolved spectroscopic characterization of reflective and transparent materials at a micro-meter scale using coherence scanning interferometry

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The development of new technologies and innovative products today is often accompanied by the emergence of new micro and nanomaterials. Due to their wider use in many applications, performing accurate characterization of these materials is becoming essential. The high performance of coherence scanning interferometry for materials characterization in terms of topographic, roughness and thickness measurements as well as for tomographic analysis of transparent layers has already been well demonstrated. However, demands regarding the spectral characterization of these materials requires new operation modes using the combination of spectral measurements with high resolution imaging. In this work we present a technique for local spectral measurements by careful processing of

the entire interferometric signal over the scanned depth at each pixel in the image, so providing spatially resolved measurements in both the lateral and axial directions. Being a far-field technique, and because the sample is illuminated with a white light source, spectral information is obtained over large areas ( $150 \times 150 \mu\text{m}^2$ ) at the same time and for all the wavelengths. Spectroscopic mapping of a sample consisting of four different materials (Si, Al, Ag, Ti) and depth-resolved measurements performed through a thin layer of PDMS are reported. Spectral measurements are made over an area of about  $1\text{-}2 \mu\text{m}^2$ , with an axial resolution of  $1 \mu\text{m}$ , these features being dependent on the optical parameters of the system.

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**1 Introduction** White light interference microscopy is a widely used technique in the domain of materials since it is contactless and therefore enables accurate characterization of the samples without degrading the surface. Today, the development of many new technologies is often accompanied by the emergence of new materials with micro and nanometric dimensions, thus requiring characterization techniques with very high spatial resolutions. Measurement methods based on the analysis of white light interference fringes, such as PSM (Phase Shifting Microscopy) [1] or CPM (Coherence Probe Microscopy) [2], [3], have already well demonstrated their abilities for measuring topography, roughness, and thickness of samples with sub-micrometric spatial resolutions (lateral and axial). The latter technique is

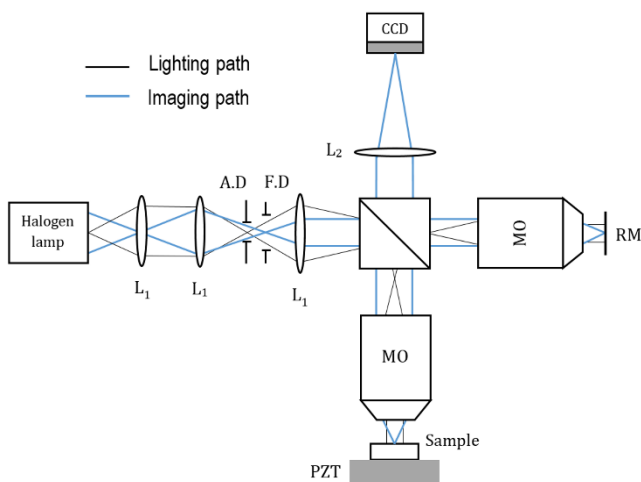
also used in the tomographic mode for investigating the structural information over the depth of transparent materials [4]. More recently, still based on the processing and analysis of interference fringes, it was shown that very thin film thicknesses ( $\sim 50 \text{ nm}$ ) can be measured [5]. To achieve a better specific characterization of materials, obtaining more information about the samples is becoming essential, mainly concerning their spectral characteristics. While an optical spectrometer, or ellipsometer, may provide spectral information about the sample, the measurement is made over a large area (several  $\text{mm}^2$ ) and cannot be used for the local measurements of micro-structured materials. Consequently, demands regarding the spectral characterization of materials increasingly requires new operation modes that combine

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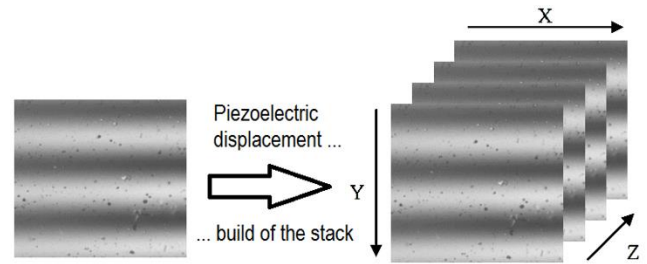
spectral measurements with high resolution imaging. For example, SNOM (Scanning Near field Optical Microscopy) [6], and hyperspectral imaging [7] allow spectroscopic measurements at high spatial resolution but require either very long scanning times to obtain information over large areas, or a high system complexity and cost. In the present work we present a technique for local spectral measurements by the careful processing of the entire interferometric signal in white light interferometry over the scanned depth at each pixel in the image, so providing spatially resolved measurements in both the lateral and axial directions. While SNOM is limited to surface analysis (not deeper than 100 nm) and hyperspectral imaging is only intend to local characterizations, the proposed method is more flexible since local (surface and depth) and global spectral measurements can be performed from the same 3D interferometric data volume. Moreover, the technique is faster than the two previously mentioned techniques, and has the advantage of being able to be used within the depth of transparent samples. To attain this requires an accurate calibration of the optical system [8]. Reflectance spectra of highly reflective materials (Si, Ti, Ag, Al) are first reported, and then reflectance spectra measurements of buried interfaces made through a transparent layer (PDMS) are presented.

**2 Measurement method** This section describes both the experimental setup to acquire the interferometric signal and the post processing applied to extract the spectral reflectance of the sample studied.

**2.1 Experimental optical setup** The optical system used to carry out the experiments is presented in Fig. 1. This consists of a modified Leitz-Linnik interference microscope with two identical x50 objectives with a numerical aperture of 0.85 and a halogen lamp illumination source.



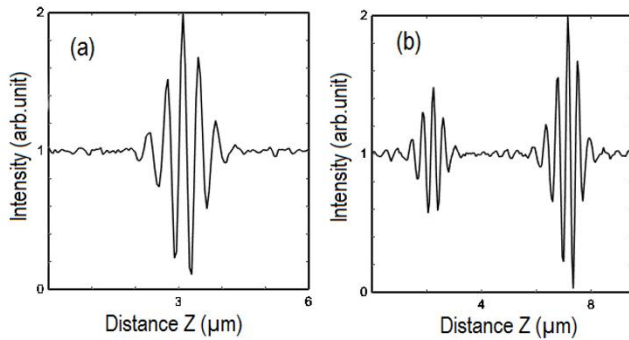
**Figure 1** Schematic diagram of the optical system. L1, aspheric lenses; L2, imaging lens  $f = 200$  mm; A.D, aperture diaphragm; F.D, field diaphragm; MO, 50x microscope objectives; RM, reference mirror; PZT, piezoelectric device.



**Figure 2** Interferometric image acquired by the camera and construction of the XYZ image stack.

The illumination arm (from the source to the third lens) is a Köhler illuminator allowing homogeneous illumination of the sample as well as the ability to control the spatial coherence of the source using an aperture diaphragm. The image acquired by the camera consists in the image of the sample with white light fringes superimposed on its surface (Fig. 2). These interference patterns result from the superposition of the light coming back from the sample and the reference mirror. The sample is investigated by scanning these fringes over depth (Z-axis) using a piezoelectric table (PIFOC from PI) located under the sample that changes the distance between the surface of the sample and the reference mirror. The piezo actuator is controlled in a closed loop with a capacitive position sensor, having a sensitivity of 1 nm. At each displacement step, an XY image is acquired with the camera, leading to a stack of XYZ images as illustrated in Fig. 2. The image acquisition is performed with a Photonfocus monochrome camera having 1024x1024 pixels and a Giga Ethernet connection. To obtain a sufficient sampling of the interferometric signal, the displacement step is adjusted to 50 nm. The whole system is controlled using a program developed in LabVIEW 2014 with the ImaqVision module.

**2.2 Processing** The interferometric signal from which is extracted the reflectance of the sample, is obtained by looking at the variation of the intensity in each image in the stack at a given pixel. Two typical interferometric signals are given in Fig. 3 which were extracted from the image stack of a very reflective material (Fig. 3(a)) and a transparent layer (Fig. 3(b)). The Fourier transform of these signals is proportional to several parameters including the spectral reflectance of the structure reflecting the light. In the case of a highly reflective material, only one interferogram appears in the interferometric signal, in which case only one step is required to extract the spectral reflectance, consisting of the calibration of the optical system. This calibration step is essential and consists in measuring a sample with a known spectral reflectance (previously measured using a spectrometer for example) in order to determine the spectral signature of the system for the given experimental / surrounding conditions. For a simple transparent material, two interferograms appear in the signal, (one from the surface and one from the rear interface). This makes possible a depth-resolved measurement i.e., the determination of the spectral



**Figure 3** Interferometric signals from (a) silicon substrate, (b) PDMS layer deposited on silicon.

reflectance of the rear interface, by processing each of the interferograms separately. In this case, an additional step to that of the calibration is necessary to obtain the depth-resolved spectral reflectance.

The two following formulas express, respectively for cases (a) and (b), the relationships between the desired spectral reflectance and the Fourier transform of the interferograms.

$$R_S(\lambda) = \frac{\left| \frac{FT[I(\delta)]_S}{FT[I(\delta)]_{cal}} \right|^2}{R_{cal}(\lambda)} \quad (1)$$

$$R_{S_2}(\lambda) = \frac{\left| \frac{FT[I(\delta)]_{S_2}}{FT[I(\delta)]_{cal}} \right|^2}{R_{cal}(\lambda)} \frac{R_{cal}(\lambda)}{(1 - R_{S_1}(\lambda))^2} \quad (2)$$

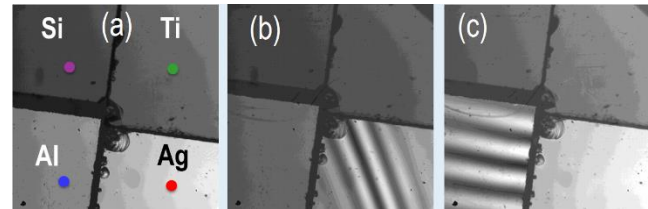
In the case (a) (Eq. (1)),  $R_S$  is the reflectance spectrum of the material surface,  $R_{cal}$  is the reflectance spectrum of the sample used for the calibration step,  $FT[I(\delta)]_S$  and  $FT[I(\delta)]_{cal}$  are the Fourier transforms of the interferometric signals from the material studied and the calibration sample. In the case (b) (Eq. (2)),  $R_{S_2}$  is the reflectance spectrum of the rear interface of the transparent layer,  $FT[I(\delta)]_{S_2}$  is the Fourier transform of the second interferogram, and  $R_{S_1}$  is the reflectance of the surface, which was found from applying Eq. (1).

Once the interferometric image stack is built, the processing is only based on Fourier transforms and is therefore not at all onerous. Because the interferometric signal is usually extracted from 3x3 binning pixel, the measurement is very well spatially resolved. Some characteristics are presented in table 1 in section 3.3.

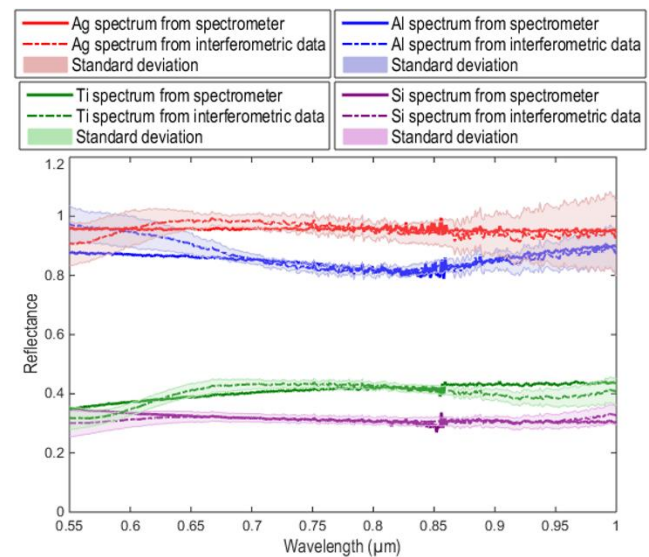
**3 Experimental results** This section reports spectral reflectance measurements made on surfaces of highly reflective materials and through a transparent layer of PDMS deposited on a substrate of silicon with some silver dots. For all the results presented, a substrate of silicon is used as reference for the calibration step, its reflectance spectrum being measured independently with a spectrometer. In the last section, several performance criteria, such as acquisition and processing times, and the spatial resolutions, are presented.

**3.1 Highly reflective materials** The sample investigated in this section consists in four different materials (Ti, Al, Ag, Si) evaporated on a silicon substrate and placed side by side so that they can be measured simultaneously (Fig. 4). For the titanium, aluminium, and silver materials, the thin film deposits are 1  $\mu\text{m}$  thick. The silicon sample consisted directly of the solid material. However to compensate the loss of thickness, 1  $\mu\text{m}$  of aluminium was deposited on the rear face. The layers were deposited using an electron gun based bell jar type evaporator from TEMESCAL with planetary substrate holder for good thickness uniformity. The n-type silicon substrate consists of an ultrapure FZ wafer with a (111) orientation and a resistivity greater than 4000 ohm.cm.

The purpose of the exercise was to study the possibility of the identification of different materials in a given field of view through their individual spectral response. To this end, the four samples were cut into squares and glued together so that the upper faces were nearly in the same plane and the four materials visible in the same field of view at the center as shown in Fig. 4. Two interferometric images are also represented for two vertical positions of the sample (Fig. 4(b) and (c)).



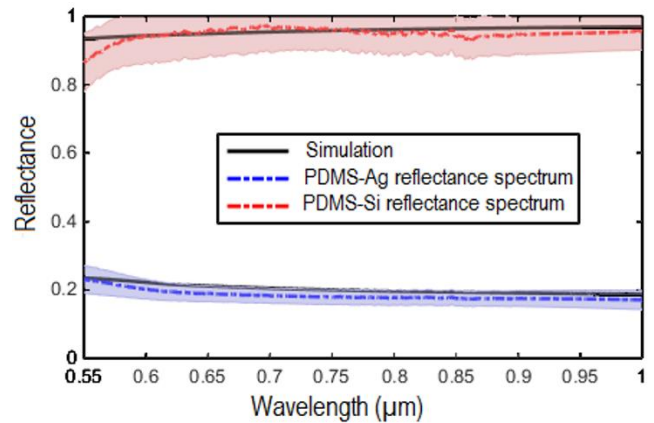
**Figure 4** (a) Direct optical image of the sample consisting of the four different materials. (b)-(c) Interferometric images at two different Z-positions of the sample extracted from the stack.



**Figure 5** Reflectance spectra of the silver, aluminium, titanium, and silicon materials obtained from the local interferometric method (dashed lines). The spectra are compared with those obtained from an optical UV-VIS-IF spectrometer (solid lines).

For the spectrometer measurements, the fast oscillations of the response at 850 nm are due to the change of the light emission lamp. The four materials were measured 50 times on 50 different binning pixels. The interferometric signals were extracted from areas close to each other and approximately located around the purple, green, blue, and red points in Fig. 4(a). The mean value and the standard deviation of these 50 reflectance measurements are plotted in Fig. 5 and are compared to the reflectance spectra obtained from an optical spectrometer model Lambda19 UV-VIS-NIR from Perkin Elmer. A very good match is obtained between the results; the largest discrepancies observed in the 550-600 nm and 950-1000 nm ranges are related to the lower signal to noise ratio of the Fourier transform of the interferometric signals [8] in the extremities of the spectrum. With these optical system parameters, the lateral resolution is measured to be  $0.5 \mu\text{m}$ , leading to a size of the measurement point of  $0.79 \mu\text{m}^2$ .

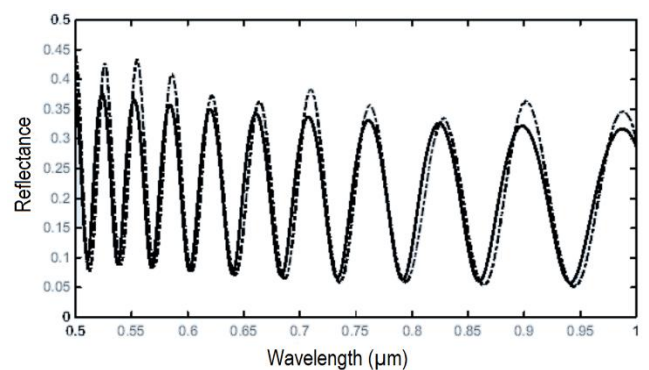
**3.2 Transparent samples** One of the advantages of using interferometric data is the possibility of making spectral measurements localized in both the transverse and the axial directions. We demonstrate this ability by measuring the reflectance spectrum of buried interfaces. The sample investigated is a  $9.3 \mu\text{m}$  thick layer of PDMS deposited on a silicon substrate having a series of silver dots. The PDMS was deposited by spin coating for 10 s at a speed of 500 rpm to stretch the viscous film followed by 30 s at 10000 rpm to reach the thickness of  $9.3 \mu\text{m}$ . The results are compared with those obtained from a simulation program. The program is based on an electromagnetic matrix method for stratified media, and enables the simulation of a multilayer structure surrounded by two infinite media [9]. The program allows the extraction of either the total reflectance spectrum, or the local reflectance spectrum between two layers. The parameters adjusted in the program are the thickness and the complex refractive indices of the PDMS, silicon and silver that were taken from the literature [10]–[12]. We have previously demonstrated that for depth-resolved spectral measurements, it is necessary to decrease the effective numerical aperture of the optical system, in this case by closing the aperture diaphragm, in order to reduce the errors that distort the results [13]. Consequently, the lateral resolution slightly decreases to  $0.85 \mu\text{m}$ . For this kind of measurements the notion of axial resolution is added. This corresponds to the minimal distance along Z between two structures that can be measured independently from each other. In the optical configuration used, this was measured to be  $1.01 \mu\text{m}$ . In the following figure, the reflectance spectra of both the PDMS-silicon and PDMS-silver interfaces are plotted. Because both materials are visible in the same field of view, the acquisition were made at the same time.



**Figure 6** Depth-resolved reflectance spectra of the PDMS-Ag and PDMS-Si buried interfaces. The experimental results are compared to simulations (black lines).

Once again, it can be noted from Fig. 6 that there is a very good match between the experimental and theoretical results. The slight differences can be related to the values used for the refractive indices of the materials in the program that may be slightly different from the real ones.

Instead of processing one interferogram after the other, the Fourier transform can be applied to the whole interferometric signal which leads to the measurement of the total reflectance spectrum as illustrated in Fig. 7. The sample used was a  $3.3 \mu\text{m}$  thick PMMA layer deposited on a silicon substrate. The spectrum is consistent with the expected one. These spectra are often used since their modulation enables the measurement of the thickness of very thin films with high accuracy [14].



**Figure 7** Total reflectance spectrum of a  $3.3 \mu\text{m}$  thick PMMA layer deposited on a silicon substrate. The experimental spectrum (dashed line) is compared to that obtained from the simulation program (solid line).

**3.3 Performance** This section summarizes some performance criteria for spectral measurements localized either on surfaces or within the depth of a transparent layer. The acquisition and processing times were measured for the acquisition of image stacks having a size of  $128 \times 113 \times 8 \mu\text{m}^3$

and  $128 \times 113 \times 15 \mu\text{m}^3$  (X x Y x Z) for respectively the cases of surface and depth-resolved measurements.

**Table 1** Performance criteria of the spectral analysis.

	Surface measurements	Depth-resolved measurements
Maximum FOV*	$236 \times 236 \mu\text{m}^2$	$236 \times 236 \mu\text{m}^2$
Acquisition time	22 min 35 s	33 min 30 s
Processing time	30 s	1 min 30 s
Wavelength range	550 – 1000 nm	550 – 1000 nm
Lateral resolution	$0.5 \mu\text{m}$	$0.85 \mu\text{m}$
Meas point size	$0.79 \mu\text{m}^2$	$2.27 \mu\text{m}^2$
Axial resolution	-	$1.01 \mu\text{m}$
Spectral resolution	Depends on the length of the signal	

\* Field of view.

While the first seven parameters only depend on the optical parameters of the system, the achievable spectral resolution depends on the length of the processed signal. Indeed, an increase in the spectral resolution is obtained from the processing of the signal with the maximum of sampled points. For transparent samples, the length is fixed by the distance between each interferogram (see Fig. 3(b)). Usually, the signal is selected using a window with a length which is approximately the same as that of the interferogram [13]. This gives a spectral resolution of 30 nm at a wavelength of 650 nm for both cases. The measurable wavelength range is set by both the illumination spectrum of the source and the spectral response of the camera. This is therefore a parameter that can be adapted as a function of the range that is desired to be measured. For the optical system used, the wavelength range was 550-1000 nm.

**3 Conclusion** This work reports a recent technique based on the analysis and processing of white light interference fringes for the local reflectance spectra determination of both reflective and transparent materials. The measurements are spatially resolved in both the lateral and the axial directions. In the transverse direction, a resolution of  $0.5 \mu\text{m}$  is achieved for surface measurements and slightly decreases to  $0.85 \mu\text{m}$  for depth-resolved measurements because of the necessity to reduce the effective NA. We have shown that the results of the measured reflectance spectra are consistent with those obtained from an optical spectrometer and simulations. Being a far field technique and due to the use of a white light source, the spectral information is obtained over large fields of view, dependant on the magnification and camera target size. By means of a single scan along the optical axis, local results are obtained at the same time and for all the wavelength included in the effective source spectrum (product of the source spectrum by the camera spectral response). This makes the method a very convenient tool compared to other techniques such as SNOM or hyperspectral imaging, which require a sequential recording as a function

of either the wavelength or position. Although the acquisition time may increase for the investigation of thick materials, the processing time will remain very low, so enabling a very fast spectral analysis of the sample. In addition, new advances in real time coherence scanning interferometry and FF-OCT could improve the acquisition and processing times for full volume characterization [15].

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