Metrol. Meas. Syst., Vol. XIX (2012), No. 2, pp. 365-372.



METROLOGY AND MEASUREMENT SYSTEMS

Index 330930, ISSN 0860-8229 www.metrology.pg.gda.pl



# NON-DESTRUCTIVE INSPECTION OF ANTI-CORROSION PROTECTIVE COATINGS USING OPTICAL COHERENT TOMOGRAPHY

# Paulina Antoniuk<sup>1,2)</sup>, Marcin R. Strąkowski<sup>1)</sup>, Jerzy Pluciński<sup>1)</sup>, Bogdan B. Kosmowski<sup>1)</sup>

 Gdańsk University of Technology, Faculty of Electronics, Telecommunications and Informatics, Department of Metrology and Optoelectronics, Narutowicza 11/12, 80-233 Gdańsk, Poland (marcin.strakowski@eti.pg.gda.pl, +48 58 347 1361)

 Gdańsk University of Technology, Faculty of Mechanical Engineering, Department of Materials and Welding Engineering, Narutowicza 11/12, 80-233 Gdańsk, Poland (Z pantoniuk@mech.pg.gda.pl, +48 58 348 6349)

#### Abstract

Optical Coherence Tomography (OCT) is one of the most rapidly advancing techniques. This method is capable of non-contact and non-destructive investigation of the inner structure of a broad range of materials. Compared with other methods which belong to the NDE/NDT group (Non-Destructive Evaluation/Non-Destructive Testing methods), OCT is capable of a broad range of scattering material structure visualization. Such a non-invasive and versatile method is very demanded by the industry. The authors applied the OCT method to examine the corrosion process in metal samples coated by polymer films. The main aim of the research was the evaluation of the anti-corrosion protective coatings using the OCT method. The tested samples were exposed to a harsh environment. The OCT measurements have been taken at different stages of the samples degradation. The research and tests results have been presented, as well as a brief discussion has been carried out.

Keywords: Optical Coherence Tomography, corrosion, anti-corrosion coatings inspection.

@ 2012 Polish Academy of Sciences. All rights reserved

## 1. Introduction

Presently, there is a huge demand for non-destructive testing and evaluation methods (NDE/NDT) in science and industry [1-3]. One of the promising and having a high application potential is optical coherence tomography. The Optical Coherence Tomography (OCT) is an optical measurement technique used for investigation of a wide range of scattering or semitransparent materials. The OCT enables surface and subsurface examination of different types of materials to be performed in non-contact and non-destructive way. With the aid of OCT one can visualize the depth structure of investigated materials, measure its thickness and also detect some defects and inhomogeneities with a measurement resolution of a few  $\mu$ m, high sensitivity and dynamic range [4, 5]. Nowadays, the advantages of OCT make it applied in medical diagnosis, especially in ophthalmology, dermatology, stomatology, and endoscopy [6-8], as well as in industry and science [9]. Beyond medical applications, the OCT is used for material characterization, surface and subsurface defect localization [5], strain fields mapping in polymers [10], ceramic materials examination [11], as well as art conservation [12]. The high application potential makes the OCT an interesting tool for scientists and researchers in many fields of interest.

The OCT measurements are based on selective detection of the light backscattered from particular scattering points located in the investigated object, which is needed for 2D and 3D tomography imaging. To perform such measurements, low-coherence interferometry (LCI) is applied. The LCI, also known as white-light interferometry (WLI), is an attractive measurement method offering high measurement resolution, high sensitivity, and measurement speed. Unlike classic interferometry, where ambiguity of the measurement

Article history: received on Feb. 7, 2012; accepted on Apr. 23, 2012; available online on May 18, 2012; DOI: 10.2478/v10178-012-0031-x.

## P. Antoniuk, M.R. Strąkowski, et al.: NON-DESTRUCTIVE INSPECTION OF ANTI-CORROSION PROTECTIVE COATINGS...

PAN

result often exists, LCI can provide an unambiguous (i.e. absolute) measurement result relatively easily.

The main classification of OCT systems is performed by measurement signal processing. Till now there are three generations of OCT systems. The first one is the time-domain OCT (TD-OCT). The measurement signal is processed in the time domain. Therefore, the TD-OCT systems need some mechanical movable parts for tuning the main interferometer. This causes a low imaging frame rate, which increases measurement time. The next generation belongs to spectral domain OCT systems. The interference signal is recorded by an optical spectrometer, which usually is made of an optical diffraction grating and an array of detectors. The signal processing is performed in the frequency domain, which brings all information about scattering points placed in the light beam direction inside the tested devices in one shot. The SD-OCT does not need any mechanical parts, which have to be used in TD-OCT. Therefore, the SD-OCT system is very fast and enables the changes of material structure to be visualized in real time [6, 12]. The latest generation of OCT is swept-source OCT systems (SS-OCT). Measurement signal processing is performed in the frequency domain. However, an ultra-fast tunable laser and single detector are used instead of a broadband light source and a matrix of detectors, respectively. Relating to the SD-OCT, it increases the imaging frame rate, signal to noise ratio and also the measurement dynamic level.

The standard OCT system delivers only intensity images, which show the arrangement of scattering points inside the tested device. A common problem is the recognition of particular parts of OCT images in order to identify the specific layers composing the investigated structure. For this purpose, the OCT systems with additional functions have been developed. The main extensions enable the OCT Doppler analysis (D-OCT) [13], polarization sensitive (PS-OCT) [14-20] or expand the OCT measurements by wavelength dependent analysis of the device scattering or absorption features (SOCT) [21]. The other functional OCT systems which cannot be omitted, are Coherent Anti-Stokes Raman Scattering OCT (CARS-OCT) [22, 23] and Coherent Stokes Raman Scattering OCT (CSRS-OCT) [23], Second Harmonic Generation OCT (SHG-OCT) [23], and Third Harmonic Generation OCT (THG-OCT) [23].

Our research interests are concentrated on polarization-sensitive OCT systems. In contrast to conventional OCT, the PS-OCT is capable of analyzing the state of polarization of the light backscattered from the investigated object [14-20]. Applying the polarization-sensitive analysis in OCT allows to characterize the polarization properties of the particular parts of the tested object. It enables the recognition of different layers composing the tested object, as well as investigation the phenomena occurred inside the structure (e.g. strain mapping). Moreover, polarization-sensitive analysis (PS) brings also a unique benefit to OCT measurements of optically isotropic devices. The PS analysis improves the OCT imaging by increasing the visualization contrast [9, 14].

The features of the OCT method and promising benefits encouraged us to use it for anticorrosion protective coatings evaluation. Most widely used methods, which give full information about the investigated layer, are invasive and also need special preparation of the tested sample. Those, which belong to NDE-NDT domain, do not give complex data about surface and subsurface degradation of the protective coating. One of the most challenging problems is to detect any degradation fields made by pitting corrosion. According to our studies there is lack of contactless, non-destructive, and easy-to-use methods for complex evaluation of anti-corrosion protective coatings. During our research, samples with different anti-corrosion protective paints have been measured by OCT. The measurements have been performed for the samples in different stages of the corrosion progress. Also, the protective layers and metal surface quality have been evaluated. The tests and obtained results have been presented in Section 3. The measurement system has been described in Section 2 as well.

## 2. OCT measurement system

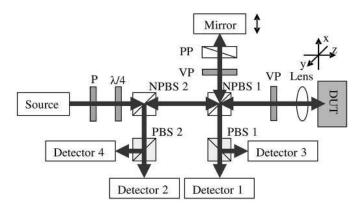


Fig. 1. The PS-OCT setup; PBS  $1\div 2$  – polarization beamsplitter, NPBS  $1\div 2$  – non-polarization beamsplitter, PP – dispersion compensation prism, P – polarization plate,  $\lambda/4$  – quarter-wave plate, VP – variable wave plate; DUT – device under test.

The presented device (Fig. 1) is the polarization sensitive optical coherent tomography system (PS-OCT), which has been designed and developed in the Department of Metrology and Optoelectronics (Faculty of ETI) at Gdańsk University of Technology. The PS-OCT system gives intensity cross-sectional or volumetric images of the sample inner structure, as well as complete information about local changes of the birefringent features. The applied polarization-sensitive analysis increases the range of measured parameters and improves the visualization contrast of the images [24].

The designed system is based on the Michelson interferometer where a movable mirror driven by a piezo actuator is placed in the reference arm. Therefore, depth scanning, also known as A-scan, can be performed very fast. Combined with transversal scanning, the A-scan provides data for cross-sectional imaging. Referring to Fig. 1, the light from the ultrabroadband source is guided to the measurement interferometer. The backscattered light from the sample (DUT) and light reflected from the mirror are recombined and subsequently separated by the polarization beam splitter (PBS 1, PBS 2) into orthogonal components. They are recorded by two pairs of photodetectors (Fig. 1: Detector 1 and 3, Detector 2 and 4). In order to increase the signal to noise ratio, low noise balanced photodetectors have been used [14]. The OCT system features have been summarized in Table 1.

Item	Value
Lateral scanning resolution	10 µm
Depth scanning resolution	4 μm
Lateral scanning velocity	200 µm/s
Light source type	Photonic crystal fibre source (MenloSystems TB-1550)
Central wavelength	1550 nm
Optical spectral width	400 nm
Emitted power of light	42 mW

For the experiments, only the intensity OCT images were acquired. However, the range of measurements has been good enough to assess the usefulness of OCT method for the evaluation of anti-corrosion protective layers.

www.journals.pan.pl

P. Antoniuk, M.R. Strąkowski, et al.: NON-DESTRUCTIVE INSPECTION OF ANTI-CORROSION PROTECTIVE COATINGS...

#### 3. Tests and measurements

During the research a series of tests were carried out with the aid of the OCT system. The main aim of the research was to assess the usefulness of the OCT method for evaluation of anti-corrosion protective paints. These tests included: (i) clean metal surface inspection, (ii) the protective coatings evaluation, (iii) and monitoring of the corrosion process. As tested protective coatings, acrylic and Hammerite paints were used. The experiment conditions and the tests results are shown in the next subsections.

## 3.1. Materials surface inspection

The tests have been done in order to evaluate the state of the unprotected metal surface using OCT. A group of three steel samples with different stage of surface degradation were prepared. The measurements were carried out for samples with clean surface and with some surface corrosion products, as well as a sample with deep corrosion pits. The test results have been shown in Fig. 2, Fig. 3, and Fig. 4.

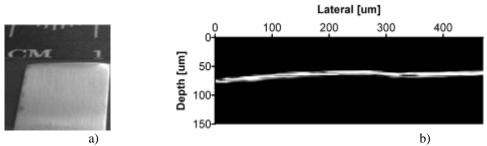


Fig. 2. Steel sample with clean surface; a) sample image, b) OCT cross-sectional image.

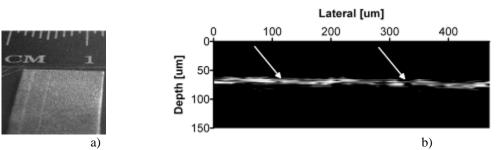


Fig. 3. Steel sample with surface eroded by corrosion process; a) sample image, b) OCT cross-sectional image.

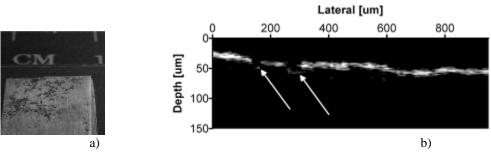


Fig. 4. Steel sample with corrosion pits; a) sample image, b) OCT cross-sectional image.

For the clean sample a smooth profile of the sample surface is shown in the OCT image (Fig. 2b). However, for the rest of investigated samples, we can observe some roughness and pits appear at surfaces (Fig. 3b and Fig. 4b). The biggest corrosion pits were indicated by arrows. According to OCT measurements the biggest pits had a depth of 15  $\mu$ m.



## 3.2. Investigation of anti-corrosion painted coatings

The next task of the research was to evaluate the quality of the anti-corrosion protective painted coatings based on OCT measurements. For the tests the three steel samples with well prepared surfaces were covered by different protective coating. The first one was made by putting the acrylic paint, the second was covered by Hammerite paint. The last one was an example of multilayer coating made of lacquer. Such prepared samples were measured using the OCT system. The measurement results have been presented in Fig. 5, Fig. 6, and Fig. 7.

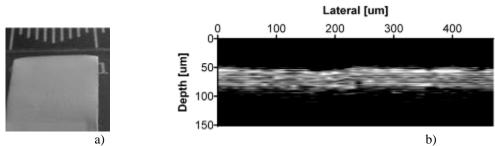


Fig. 5. Sample with acrylic paint coating; a) sample image, b) OCT cross-sectional image.

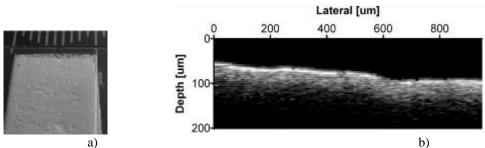


Fig. 6. Sample protected by Hammerite anti-corrosion coating after treatment by aggressive environment; a) sample image, b) OCT cross-sectional image.

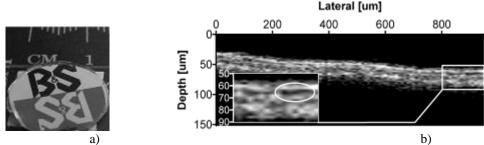


Fig. 7. Sample with multiple coating; a) sample image, b) OCT cross-sectional image.

By analyzing the obtained results it is possible to determine the protective layers thickness, as well as detect the defects and inhomogeneities of the coatings. For example, taking under consideration the first sample (Fig. 5 – a steel sample covered by acrylic paint) the measured thickness is about 40  $\mu$ m. Its value is constant over whole measured area. Moreover, the protective coating is homogeneous. Comparing with the next sample (Fig. 6 – steel covered by Hammerite paint) it is definitely better performed. The Hammerite painted layer was deposited by using a paintbrush. Therefore, the layer thickness is not constant over the measured area.

The OCT measurements results obtained for the last sample have been presented in Fig. 7. This is the sample with the multilayer protective coating. In Fig. 7b one can recognize the particular lacquer layers. Even a defect like delamination can be seen. One of the areas, in which the delamination occurs, has been indicated in the Fig. 7b.

www.journals.pan.pl

P. Antoniuk, M.R. Strąkowski, et al.: NON-DESTRUCTIVE INSPECTION OF ANTI-CORROSION PROTECTIVE COATINGS...

#### 3.3. Corrosion monitoring

The progress of the corrosion process was investigated by acquiring the OCT images for the sample with different stage of degradation. As the tested sample, the steel sample covered by acrylic protective coatings was used. In order to accelerate the corrosion rate the samples were put into concentrated nitric acid and concentrated hydrochloric acid (aqua regia), and then into sulfuric acid vapours.

Fig. 8 presents the sample before putting it into an aggressive environment. The OCT image (Fig. 8b) shows that there are no significant defects in surface and subsurface areas of the coatings. Under the influence of concentrated nitric acid and concentrated hydrochloric acid the structure of the coating was defected. Obtained results of cross-sectional OCT imaging are presented in Fig. 9b. Analyzing the protective coatings structure one can find same corrosion pits on the layer surface and also defects inside the investigated coatings. The biggest changes in the structure and defects have been highlighted in Fig. 9b. The last two pictures (Fig. 10) were made for a sample seriously damaged by the sulfuric acid vapours. The OCT image (Fig. 10b) expresses the range of corrosion damages, as well as its structure and shape.

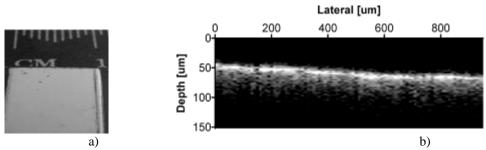


Fig. 8. Sample with anti-corrosion coating; a) sample image, b) OCT cross-sectional image.

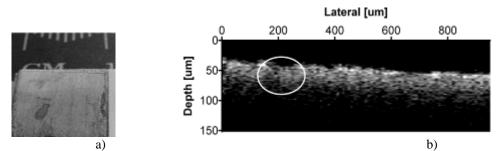


Fig. 9. Sample with anti-corrosion coating under the influence of aggressive environment; a) sample image, b) OCT cross-sectional image.

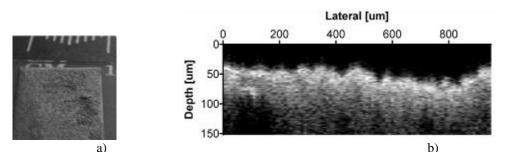


Fig. 10. Corrosive sample destruction; a) sample image, b) OCT cross-sectional image.

# 4. Conclusions

The experiment was carried out in order to assess the OCT ability for inspection of painted anti-corrosion protective coatings and corrosion progress monitoring. The tests were prepared for different anti-corrosion coatings. Obtained results show the usefulness of PS-OCT system for surface and subsurface defects examination of highly scattering materials like painted anti-corrosion protective coatings. By the use of OCT, the surface profile of tested devices can be measured, as well as surface defects and pits can be determined, and also the inner structure of the object can be examined. Moreover, the measurements can be carried out in situ without special preparation of tested devices. Unique features of OCT, like non-destructive and non-contact measurements, high measurement resolution, high application potential, can make this system an interesting tool for inspection of anti-corrosion coatings and corrosion monitoring.

## Acknowledgements

The authors would like to thank Mr. Marek Sienkiewicz from Gdańsk University of Technology for the technological support and valuable technical consultations.

This study was partially supported by the Polish Ministry of Science and Higher Education under the grant No. N N515 335636, and DS programs of the Faculty of Electronics, Telecommunications and Informatics, Gdańsk University of Technology.

## References

- [1] Weckenmann, A., Bernstein, J. (2010). Optical multi-sensor metrology for extruded profiles. *Metrol. Meas. Syst.*, 17(1), 47-54.
- [2] Zawada-Tomkiewicz, A., Ściegienka, R. (2011). Monitoring of a micro-smoothing process with the use of machined surface images. *Metrol. Meas. Syst.*, 18(3), 419-428.
- [3] Zawada-Tomkiewicz, A. (2011). Estimation of surface roughness parameter based on machined surface image. *Metrol. Meas. Syst.*, 17(3), 493-504.
- [4] Fercher, A.F., Drexler, W., Hitzenberg, C.K. (2003). Optical coherence tomography-principles and applications. *Rep. Prog. Phys.*, 66, 239-303.
- [5] Wiesauer, K., Pircher, M., Götzinger, E. (2005). En-face scanning optical coherence tomography with ultra-high resolution for material investigation. *Opt. Express*, 13(3), 1015-1024.
- [6] Wojtkowski, M., Leitgeb, R., Kowalczyk, A., Bajraszewski, T., Fercher, A.F. (2002). In vivo human retinal imaging by Fourier domain optical coherence tomography. *J. Biomed. Opt.*, 7(3), 457.
- [7] Fujimoto, J.G., Pitris, C., Boppart, S.A., Brezinski, M.E. (2000). Optical coherence tomography: an emerging technology for biomedical imaging and optical biopsy. *Neoplasia*, 2(1-2), 9-25.
- [8] Fujimoto, J.G., Brezinski, M.E., Tearney, G.J., Boppart, S.A., Bouma, B., Hee, M.R., Southern, J.F., Swanson, E.A. (1995). Optical biopsy and imaging using optical coherence tomography. *Nature Med.*, 1(9), 970-972.
- [9] Stifter, D. (2007). Beyond biomedicine: a review of alternative applications and developments for optical coherence tomography. *Appl. Phys. B*, 88(3), 337-357.
- [10] Wiesauer, K., Pirchen, M., Gotzinger, E., Hitzenberger, C.K. (2007). Investigation of glass-fibre reinforced polymers by polarisation-sensitive, ultra-high resolution optical coherence tomography: Internal structures, defects and stress. *Composites Science and Technology*, 67(15-16), 3051-3058.
- [11] Bashkansky, M., Duncan, M.D., Kahn, M., Lewis III, D., Reintjes, J. (1997). Subsurface defect detection in ceramics by high-speed high-resolution optical coherent tomography. *Opt. Lett.*, 22(1), 61-63.
- [12] Targowski, P., Rouba, B., Góra, M., Tymańska-Widmer, L., Marczak, J., Kowalczyk, A. (2008). Optical Coherence Tomography in Art Diagnostics and Restoration. *Appl. Phys. A*, 92(1), 1-9.

P. Antoniuk, M.R. Strąkowski, et al.: NON-DESTRUCTIVE INSPECTION OF ANTI-CORROSION PROTECTIVE COATINGS...

- [13] Makita, S., Fabritius, T., Yasuno, Y. (2008). Quantitative retinal-blood flow measurement with threedimensional vessel geometry determination using ultrahigh-resolution Doppler optical coherence angiography. *Opt. Lett.*, 33(8), 836-838.
- [14] Strąkowski, M., Pluciński, J., *et al.* (2008). Polarization sensitive optical coherence tomography for technical materials investigation. *Sensors and Actuators A*, 142(1), 104-110.
- [15] de Boer, J.F., Milner, T.E. (2002). Review of polarization sensitive optical coherence tomography and Stokes vector determination. *J. Biomed. Opt.*, 7(3), 359-371.
- [16] Goetzinger, E., Pircher, M., Fercher, A.F., Hitzenberger, C.K. (2004). Polarization-sensitive optical coherence tomography: a comparison of methods. In *Proc. SPIE*, 5316, 365.
- [17] Hitzenberger, C.K., Götzinger, E., Sticker, M., Pircher, M., Fercher, A.F. (2001). Measurement and imaging of birefringence and optic axis orientation by phase resolved polarization sensitive optical coherence tomography. *Opt. Express*, 9(13), 780-790.
- [18] Stifter, D., Sanchis Dufau A.D., Breuer, E., Wiesauer, K., Burgholzer, P., Höglinger, O., Götzinger E., Pircher M., Hitzenberger C. K. (2005). Polarisation-sensitive optical coherence tomography for material characterisation and testing. *Insight*, 47(4), 209-212.
- [19] Jiao, S., Yao, G., Wang, L.V. (2000). Depth-Resolved Two-Dimensional Stokes Vectors of Backscattered Light and Mueller Matrices of Biological Tissue Measured with Optical Coherence Tomography. *Appl. Opt.*, 39(34), 6318-6324.
- [20] Jiao, S., Wang, L.V. (2002). Jones-matrix imaging of biological tissues with quadruple-channel optical coherence tomography. *J. Biomed. Opt.*,7(3), 350-8.
- [21] Kasseck, C., Jaedicke, V., Gerhardt, N.C., Welp, H., Hofmann, M.R. (2010). Frequency domain optical coherence tomography with subsequent depth resolved spectroscopic image analysis. In *Proc. SPIE*, 7554, 75542T-1-5.
- [22] Marksb, D.L., Bredfeldt, J., Hambir, S., Dlott, D., Kitchell, B., Gruebele, M., Boppart, S.A. (2003). Molecular Species Sensitive Optical Coherence Tomography using Coherent Anti-Stokes Raman Scattering Spectroscopy. In *Proc. SPIE*, 4956, 9-13.
- [23] Vinegoni, C., Bredfeldt, J.S., Marks, D.L., Boppart, S.A. (2004). Nonlinear optical contrast enhancement for optical coherence tomography. *Opt. Express*, 12(2), 331-341.
- [24] Wierzba, P. (2008). Stability of an optical displacement sensor using a two-beam polarization interferometer. *Metrol. Meas. Syst.*, 15(2), 205-213.