Vol. 21, No. 3(2022) Mat2918
Revista Mexicana de Ingeniería Química

Evaluation of destructive and non-destructive measurement methods to determine the thickness of a titanium coating deposited by physical evaporation

Evaluación de métodos de medición destructivos y no destructivos para determinar el espesor de un recubrimiento de titanio depositado por evaporación física

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Received: August 12, 2022; Accepted: October 15, 2022

Abstract

The purpose of this research is to develop reference Ti-coatings and analyze the thicknesses evaluated via destructive (DT) and non-destructive (NDT) measurement techniques. The Ti coating was deposited on a mirror finished AISI M2 steel by the magnetron process. The nominal value of the coating thickness was 1 μm. The NDT was the X-Ray Fluorescence (XRF) method, the thickness for the portable analyzer was 1.18 ± 0.19 μm and the thickness using the spectrometer XRF was 1.17 ± 0.24 μm. For DT, thickness measurements were carried out in the cross-sections of the Ti-coatings, using the Field Emission - Scanning Electron Microscopy equipped with Energy Dispersive Spectrometer (FE-SEM-EDS). The coating thickness measurements were performed with image analysis of secondary electrons (SE), backscattering electrons (BSC), mapping (MAP) and elemental profiles (LS) using ImageJ and Aztec software. The results of the thickness measurements are SE: 1.12 ± 0.15 μm, BSC: 1.14 ± 0.13 μm, LS: 1.25 ± 0.13 μm and MAP: 1.29 ± 0.6 μm. Variations in the results were identified and estimated in the uncertainty models proposed for each method.

Keywords: coating thickness, magnetron sputtering, titanium, destructive measurement, non-destructive measurement.

Resumen

El objetivo de esta investigación es desarrollar recubrimientos de referencia de Ti y analizar sus espesores mediante técnicas de medición destructivas (TD) y no destructivas (TND). El recubrimiento de Ti se depositó sobre acero AISI M2 utilizando la técnica de pulverización física por magnetrón. El valor del espesor nominal del recubrimiento fue de 1 μm. La TND fue fluorescencia de rayos X (XRF), el espesor medido por el analizador portátil fue de 1,18 ± 0,19 μm y para el espectrómetro XRF fue de 1,17 ± 0,24 μm. Para la TD, se realizaron mediciones de espesor en las secciones transversales de los recubrimientos Ti, mediante microscopio de barrido con electrones y cañón de emisión acoplado a un espectrómetro de dispersión de energía RX (FE-SEM-EDS). Las mediciones de espesor se realizaron mediante análisis de imágenes de electrones secundarios (SE), de electrones retrodispersados (BSC), de mapeo y de perfiles elementales mediante los programas informáticos ImageJ y Aztec. Los resultados de los espesores son SE: 1,12 ± 0,15 μm, BSC: 1,14 ± 0,13 μm, LS: 1,25 ± 0,13 μm y MAP: 1,29 ± 0. 6 μm. Las variaciones en los resultados se identificaron y estimaron en los modelos de incertidumbre que se proponen para cada método.

Palabras clave: espesor de recubrimiento PVD, titanio, medición destructiva, medición no destructiva.

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https://doi.org/10.24275/rmiq/Mat2918
ISSN:1665-2738, *issn-e*: 2395-8472

1 Introduction

Currently there are several industrial processes whose main purpose is to deposit coatings (Vladimir Syasko, 2014) (Baptista *et al*., 2018). For instance, the scientific field these processes are being employed among others for the design and manufacture of new sensors (Hadis *et al*., 2015; Marques *et al*., 2015) (Moore, 2013; Xu *et al*., 2021) and implants for humans to point out some applications (Schalk *et al*., 2022) (Kourentzi K & Wilson R., 202). The essential measurand of a coating is its thickness, since its behavior will directly depend on the thickness homogeneity (Meyer, 1972) (Giurlani *et al*., 2020). According to the application, other measurands such as chemical composition, adhesion, microstructure, wear, hardness, strength, phase distribution, etc., can be added (Abiline *et al*., 2007). The demand for information requires reliable references for the coating to fulfill the application needs (Hoffmann *et al*., 2003).

Non-destructive measurement (NDT) techniques are a valuable tool for any type of process, since no sample preparation is necessary and the results are available within seconds (Coating Thickness Gauges|Helmut Fischer; Somarin *et al*.,) (Niton® XL5 portable XRF analyzer).TM XL5). Currently technologies based on miniaturization and sensors with nanomaterials are the platform for portability (Kim *et al*., 2008); (Mañoso, E., Herrera-Basurto R., Simonet B., Valcarcel M., 2013); (Sánchez-Suárez C. *et al*., 2020); (Sósol-Fernández R.E. *et al*., 2012) Portable analytical techniques can be cataloged with a lab on chip, which, was the gateway to 4.0 technology (Chemat *et al*., 2019; Moore, 2013; Xu *et al*., 2021). Now the lab is located on the production line and is not a separate space, allowing for immediate measurements and faster decision making (Rojo, 2019) (López-Arenas T. & Pulis A.. 2020)

Analytical techniques that require extensive sample preparation (e.g., cutting, grinding, coarse polishing, fine polishing and mirror finishing) demand a high consumption of resources and time. Although the invested preparation work, this is not a 100 % guarantee and the result will probably not be the most reliable (ASM International & EDFAS Organizing Committee, editors, 2012; "Ilion. TMPlanar Surface Preparation for SEM Cross Section Viewing," 2010; Ström *et al*., 2010). These so-called destructive techniques (DT) generally play a rearguard role in the analytical strategy (Valcárcel & Cárdenas, 2005). The DT are the traditional analytical techniques with the most robust measurement methods and the highest metrological hierarchy (Estill & Moody, 1965; Krishnan, 2021; Vladár, 2016).

In this study, two methods for thickness measurements of the Ti metallic coatings deposited by magnetron sputtering on an AISI M2 tool grade steel are evaluated. The thickness analysis was measured applying NDT methods (XR fluorescence) and by DT (cross-sectional preparation and analysis with scanning electron microscopy and EDS). The nondestructive tests provide reliable results on thickness, these were confirmed by the DT measurement methods). These results allow the generation of robust analytical methodologies to reliably measure thicknesses of thin coatings obtained by PVD. The method may allow saving material resources and time, which strengthens decision making in these processes.

2 Materials and methods

2.1 Coating deposition method

A semi-industrial OERLIKON Domino Mini model was used for Ti deposition. A batch of three steel discs was coated by the metal sputtering method (magnetron sputtering). The discs were placed in 3 positions of the 2-axis rotating planetary system. This device rotates the parts inside the chamber of the PVD equipment. The positions in the planetary system [top (T), middle (M) and bottom (B)] of each steel discs. The discs were weighed and measured prior the coating process. For the deposition of Ti, a commercial Ti bar of 456 mm long × 81 mm wide × 10 mm thick with 99.5 % purity from PLANSEE was used. The first step is constant vacuum until reaching 1×10^{-5} mbar and simultaneously heating the samples up to 450 °C for 2 h. Then, the surface of the steel samples was activated using Ar^+ ions at 1×10^{-2} mbar by applying the AEGD process (Vetter *et al*., 1993), applying -50 V DC, 20 kHz polarization for 40 min. The deposition was performed by gradually increasing both the electrical potential (0 to 150 V) and the electrical power (0 to 7 kW) over 9 min. Then a constant power of 7 kW with an electrical bias potential of 150 V were applied for 35 min. These processes were carried out by keeping a constant Ar-flow of 110 cm^3/min. The last stage consisted of cooling for 1 h with constant vacuum 1×10^{-5} mbar, to finally extract the coated samples.

Table 1. Procedure to obtain a mirror finished surface of AISI M2 steel.

Stage	Sub-stage	Abrasive material	Time (min)	Rotational speed (rpm)
Coarse grinding	1	Abrasive paper FEPA # 180	12	250
	2	FEPA Abrasive Paper # 220	4	250
	3	FEPA Abrasive Paper # 320	4	250
	4	FEPA abrasive paper # 500	4	250
Cleaning process soap solution, rinse with plenty of water, dry with pressurized air.				
Fine grinding	5	FEPA # 800 abrasive paper	4	300
	6	FEPA Abrasive Paper # 1200	5	300
	7	FEPA Abrasive Paper # 2000	6	350
Cleaning process with soap solution, rinse with plenty of water, dry with pressurized air.				
Polishing	8	Cotton cloth + monocrystalline diamond suspension (6 μm)	5	300
	9	Cotton cloth + monocrystalline diamond suspension (1 μm)	5	300
Cleaning process with soap solution, rinse with plenty of water, dry with pressurized air, clean the surface with cotton or absorbent paper impregnated with methanol, dry with pressurized air, clean the surface with cotton or absorbent paper impregnated with acetone, dry with dry air.				
Final polishing	10	Cotton cloth + alumina nanoparticles suspension (0.05 μm)	5	350
Cleaning process with soap solution, rinsing with abundant water, drying with pressurized air, ultrasonic cleaning of the specimen with methanol, drying with pressurized air, ultrasonic cleaning in acetone, drying with dry air.				

2.2 *Preparation of the base metal (substrate)*

The AISI M2 tool grade steel was selected as base metal due to its high compatibility with metallic Ti coatings. The steel was sectioned into discs of Ø = 2.5 cm and 7 mm thickness. The discs were subjected to the preparation process described in Table 1. This table shows the parameters needed at each preparation stage to obtain the mirror-finished surfaces reported in this investigation. It should be noted that the prepared surfaces were evaluated for average roughness, and surface defects such as second phases, scratches, pores, and cracks. These coating defects were identified and quantified.

2.3 *Analysis techniques*

Most thickness measurements in the industry are performed by XRF, since it is a simple, fast, non-destructive and robust process, therefore, standards have been developed to perform measurements under quality criteria and under conformity, including ISO 3497 (ISO & BIS) and ASTM B568 (ASTM).

Nondestructive testing (NDT) was performed by two techniques: (a) One by portable modality using a Thermo Scientific XL 5 portable analyzer, Niton Connect software Version: 2.2.0.1306. Then, thickness measurements were performed with a Fischerscope X-ray XRF spectrometer XAN 219, which is a floor mode spectrometer. The portable spectrometer analyses were performed by configuring the equipment for thickness measurements: 30 s, 50 kV, filters and SSD detector. The setup requires chemical composition data and the densities of Ti and AISI M2 steel. The chemical composition of the coating was considered to be the same 99.5% pure Ti target. For M2 steel the information was obtained from matweb (Online Materials Information Resource - MatWeb). The thicknesses measured with the soil spectrometer were obtained following the method described in the standards. The experimental parameters were 40 s, 25 kV and 128 eV resolution.

An initial estimation of the chemical composition or thickness of a coating by XRF is performed with software that generally has poor accuracy (Ager, F.J. *et al.*, 2017) Therefore, it is common that measurements with higher accuracy are performed with a fundamental parameter (FP) method, which is an advantageous method, when problems also arise

due to insufficient certified reference materials (Kolbe, M. *et al*., 2005).

Firstly, the equipment was setting up and a performance inspection of the Thermo XL 5 was carried out using the HHZN400FE999 Zn/Fe 400 μin or 10 μm reference material. Then portable XRF thickness measurements were performed on 5 different areas of the Ti coated surface. The analysis area is 5 mm^2 approximate.

In each zone at least 20 thickness measurements were performed to obtain an estimation of the homogeneity of the coating. Then the coated discs were also subjected to thickness measurements using a floor standing XRF spectrometer, using the manual method for thickness measurements. For this, linear analyses were performed, and each line had 25 points with an analysis area of 3 mm^2. The results are averages of 10 lines that covered the entire area of the coated sample.

Thickness measurements through cross section or DT was performed on a JEOL JSM 7200 F FE-SEM scanning electron microscope with field emission gun and equipped with an Oxford Ultim Max X-ray scattering analyzer. The magnification scale was inspected using Applied microspheres Micro Standard No. 11010 polystyrene spheres. The preparation of the cross section was a modified method based on the ASTM E3. The modified method was developed for mirror polishing of the steel specimen prior to the Ti-coating. The cross-section preparation method includes cutting the section of interest with a diamond disk, mounting in conductive bakelite, roughing, polishing and chemical etching with 5 % nital. Additionally, the specimen was metallized with gold to increase the contrast and brightness of the images. This Au-coating was performed with a JEOL Smart Coater. The DT analysis includes images obtained by secondary and backscattering electrons, mapping and elemental profiles. Micrographs were obtained at 2 500 to 20 000 X. An acceleration potential of 15 kV, working distance of 10 mm and a beam current of 13 mA were used. For mapping and elemental profiling, it was ensured that the dead time was in the range of 30 to 45 %.

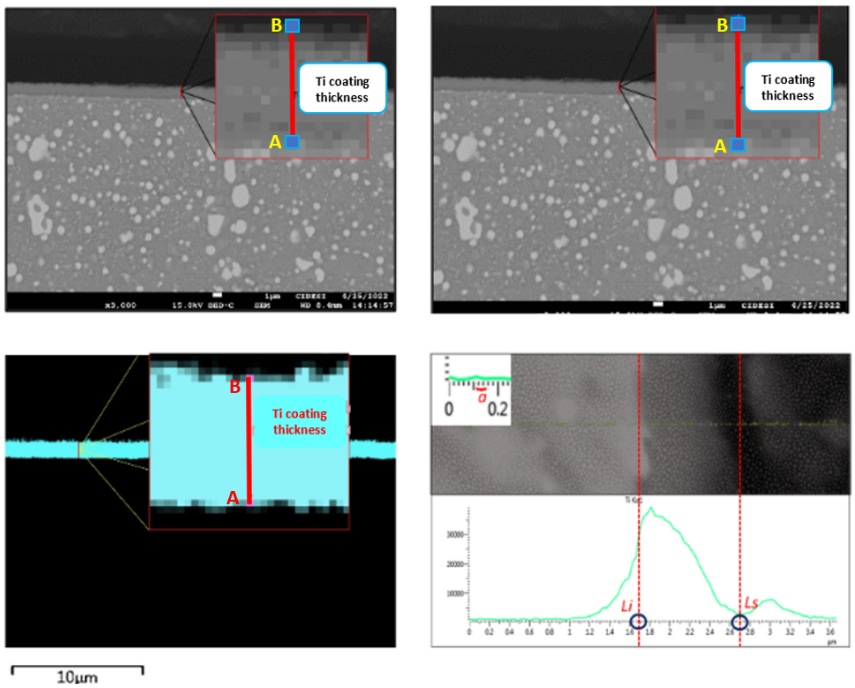

Fig 1. Criteria for thickness measurements of Ti coatings obtained by microphotographs using secondary (SE) and backscattered electrons (BSE), mapping (MAP) and elemental profiles (LS) is similar. The measurement area must be chosen and find the pixels aligned and to be linked (marked in blue), then select the top (A) or bottom of the pixel chosen as the start and draw a straight line from that point to the top (B) or bottom of the pixel where the measurement ends. For LS, a supported measurement of the image, where the profile was taken, is made and lines are drawn from the spectrum of interest, then the intersection points are found.

The free ImageJ software was used to perform the measurements on the selected images. The measurement criteria applied was to start the line at the last edge of the best-defined pixel in the image and ended at the edge of the corresponding pixel in the final section. Figure 1 shows the measurement criteria used in each measurement method. In the measurement criterion it is very important to identify the start and end of the thickness measurement. Matching between the start and end in the corresponding area in the right pixel must be ensured. The line connecting the start and end point must be straight, avoiding cuts or inclinations. It is not recommended to have as reference points the middle of a pixel.

3 Results and discussion

3.1 Preparation of mirror polished surfaces

There are reports indicating that coating adhesion depends on ~70 % of the surface preparation prior deposition. The development of a procedure to obtain mirror finish surfaces can be a routine job, however, it involves several steps that must be reproduced to guarantee the coating quality. Detailed reports on surface preparation methods were not found in the literature, often, when metallic substrates are applied, the authors simply refer to a metallographic preparation. In our case, the ASTM E 3, served as a reference to develop the procedure reported here. The conditions for roughing (grinding), polishing, cleaning and characterization stages are carefully described and documented. The procedure to obtain a mirror finish surface is crucial in the stages of cleaning during roughing, polishing and final finishing. The same preparation method was applied to measure the cross-sections of the ~ 1 μm of Ti-based metallic coatings.

3.2 Non-destructive techniques (NDT) for thickness measurement

The X-ray fluorescence spectra shown in Figure 2 are obtained by portable and floor XRF spectrometry. The representative spectrum shown in Figure 2a is obtained using the portable XRF spectrometer. According to the configuration and the applied program for the thickness measurement, the equipment performs the adjustment with an internal standard and delivers the result in micrometers with a variability measured

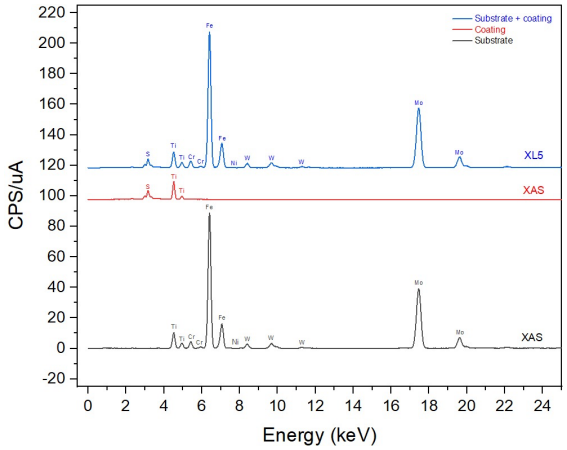

Fig 2. Spectra obtained using the Thermo Scientific XL 5 portable analyzer (a) and spectrum obtained by the Fischerscope X rays XAN 219 (b) for thickness measurement of the Ti coatings + base metal.

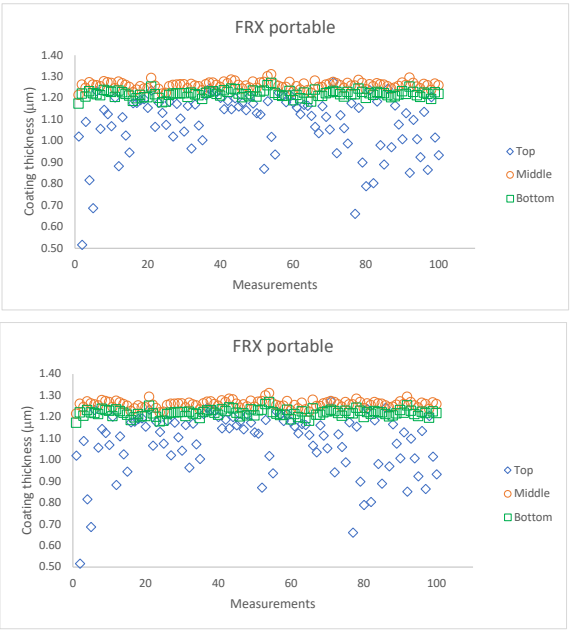

Fig 3. Coating thicknesses obtained from the two NDT measurement methods, (a) results obtained by Thermo Scientific XL 5, and (b): results obtained by Fischerscope X rays XAN 219.

in 2 standard deviations. The XRF-spectrum shown in Figure 2b side is obtained with the floor XRF spectrometer. For this measurement, the method consists of obtaining independent spectra of the Ti coating+steel and the spectra of the bare steel. The analysis is performed using an external or internal standard and this measurement is converted into a

Table 2. Basic statistics and percentage coefficient of variation for each of the samples (M015, M028 and M029).

Stadistics	FRX portable			FRX floor spectrometer		
	Top	Middle	Bottom	Top	Middle	Bottom
Coating thickness average (μm)	1.08	1.26	1.22	1.04	1.25	1.22
Standard deviation of measurements (μm)	0.14	0.02	0.02	0.19	0.02	0.01
Coefficient of variation (dimensionless)	12.91	1.31	1.35	17.04	1.42	1.19

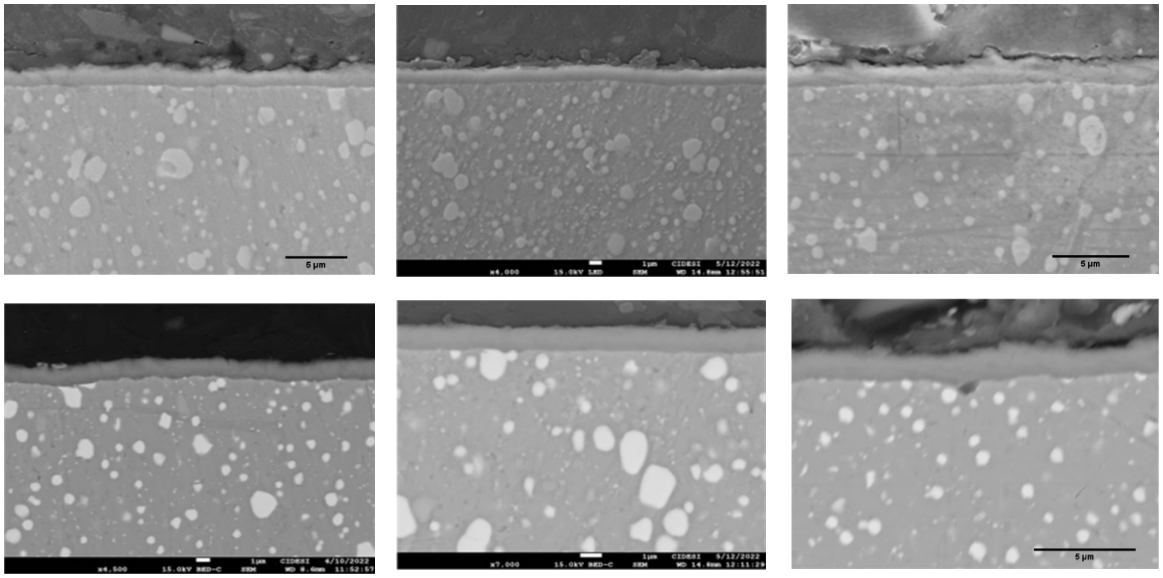

Fig 4. The top FE-SEM micrographs are SE-images and the micrographs at the bottom are BSE-images, of the Ti coatings located at the Top, Middle and Bottom positions.

mass per area signal. Finally, the density of the material of interest (internal or external standard) is converted to a measurement corresponding to the thickness of the Ti coating.

Figure 3 shows the thicknesses of the Ti coatings according to their position in the planetary system, using the NDT. The graph on the up shows the thickness measurements obtained with the portable spectrometer and on the bottom the measurements obtained with the floor spectrometer. The measurement range for the portable spectrometer is 1.02 to 1.31 μm and for the floor spectrometer is 1.05 to 1.31 μm.

Table 2 shows the basic statistics and the percentage coefficient of variation for each Ti-coating. The sample M reported the largest thickness (position in the center of the planetary system), while the sample T (top position in the planetary system) has the smallest thickness. The, sample B (placed at the bottom position of the planetary system) is within the range of the B and T samples. According to the coefficient of variation, the dispersion of values from highest to lowest was B, M and T for portable FRX, and B, M and T for floor FRX. An ANOVA was also performed with the results obtained from each measurement method. In both cases it was concluded that there is a significant difference in the thickness measurements with a confidence level of 95 %.

These NDT are widely used in the industrial sector; therefore, it is important to establish maintenance, inspection and calibration programs that allow the equipment to be in optimal condition. The nature of the measurements of these techniques are indirect, therefore, it is required to have certified reference materials with a high level of similarity with the unknown sample. In the case of the XL 5 analyzer, it was designed as a positive material identifier (PMI), which limits the configuration of the analysis. In this case, it is also relevant to identify that the computer program implemented to perform the analyses is the correct one, since these analyzers have specific software for each application. In the case of the floor spectrometer, the implementation of the measurement method is very important and requires

appropriate measurement standards. The applied spectrometer allows changing analysis parameters such as; potential difference, analysis time and area size. These techniques speed up the results by avoiding sample preparation, but the results have a greater variation with respect to direct measurement techniques using high resolution methods such as FE SEM-EDS.

3.3 Destructive technique (DT) for thickness measurement

Figure 4 shows images obtained by scanning microscopy using secondary electrons (SE) and backscattered electrons (BE) to collect complementary information for image analysis and measure with the best possible precision. Micrographs were obtained from different areas of the samples and at different magnifications (2 500 to 20 000 X) to check the homogeneity of the coating thickness. Secondary electron imaging was performed to obtain morphological information of coating including the interface between the base metal and the Ti coating. Backscattered electron imaging provides information on the differences in chemical composition among the materials of the coating-substrate system by taking advantage of their atomic mass (Z) as a criterion in the hue of each region in the image.

The bright areas correspond to chemical elements with larger Z and dark areas are for elements with smaller Z. In the case of Ti (coating) Z = 47.90 g / gmol and Fe associated with (substrate) Z = 55.85 g / gmol, it presents sufficient contrast to delimit the regions corresponding to each element of the coating-interphase-substrate system. The SE images display details of the cross section preparation (polishing and chemical attack of each of the components of the steel), which is revealing the coating relief. This is because the chemical attack did not generate a substantial change in both the precipitates of the steel and the Ti coating. On the other hand, the BSE images display flat surfaces which facilitates image analysis and thickness measurements. This surface finishing reduces the errors that could be generated in the measurements due to differences in heights.

Figure 5 shows the images obtained by each microscopy method and the image analysis to perform the thickness measurements. In the case of SE, BSE and elemental mapping, the analysis is similar where a series of filters and adjustments in contrast, brightness and focus are used in the images to define the edges of the coating and avoid measurements outside these limits. The elemental profile analysis requires an image analysis that involves the SE or BSE image, the image and profile analysis with the Aztek software.

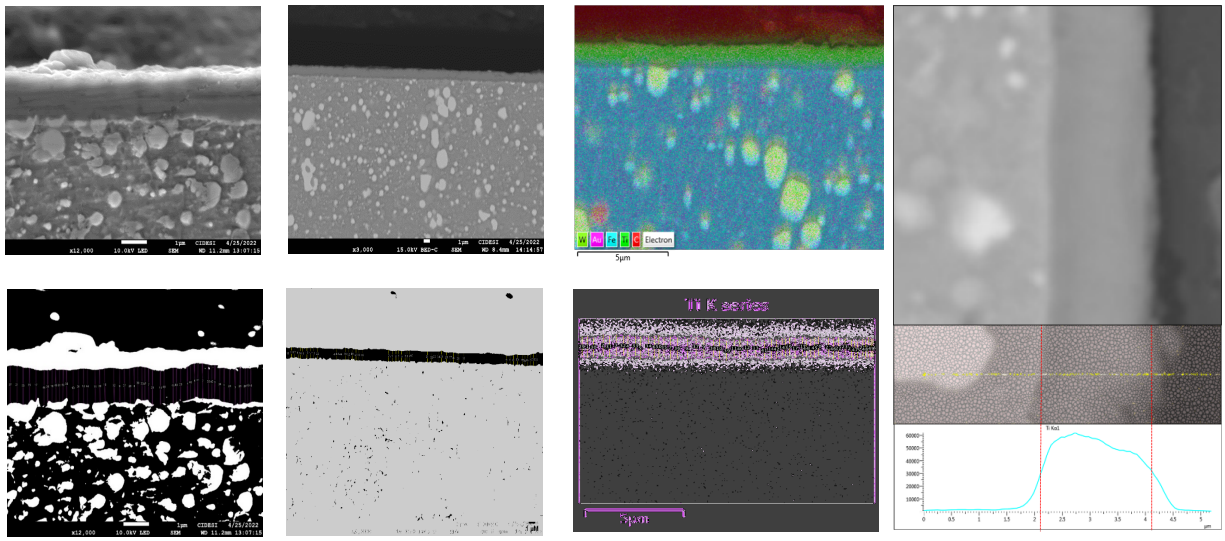

Fig 5. Image analysis sequence to determine the coating thickness. The original image and the final image after being subjected to filters and adjustments to best determine the edges of the thickness are presented.

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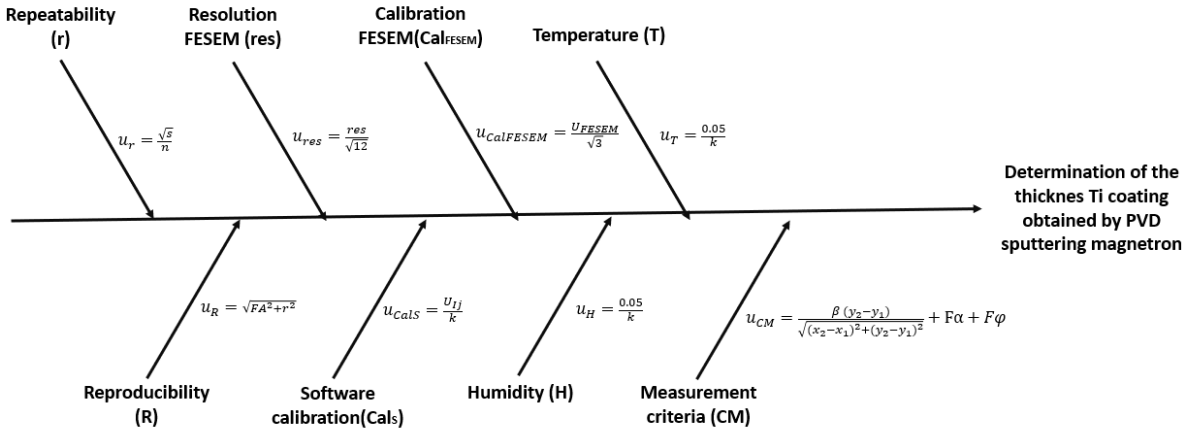

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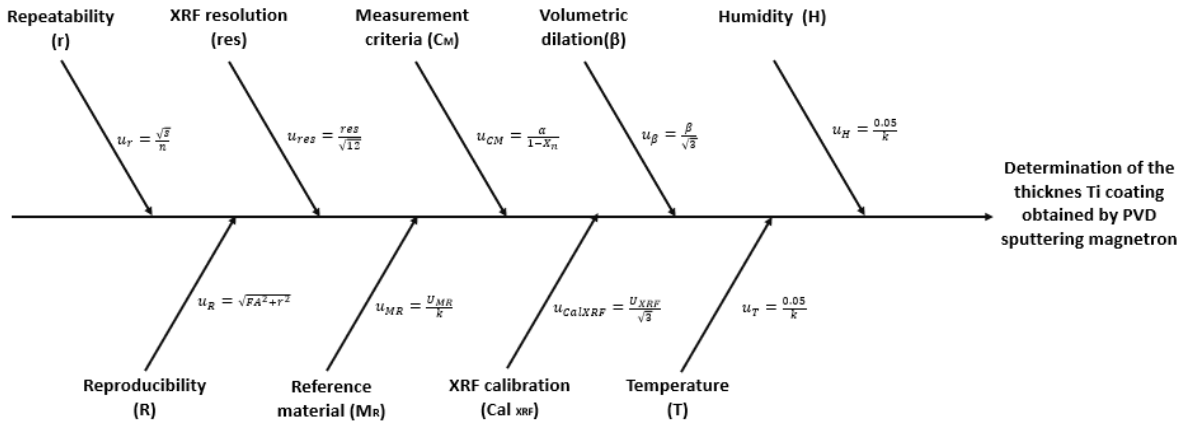


Fig 6. Sup. Cause-effect diagram to identify the input quantities of the uncertainty model used in thickness determination with floor spectrometer XRF. Inf. Cause-effect diagram for uncertainty components in thickness determination by ES image analysis.

3.4 Measurement uncertainty models

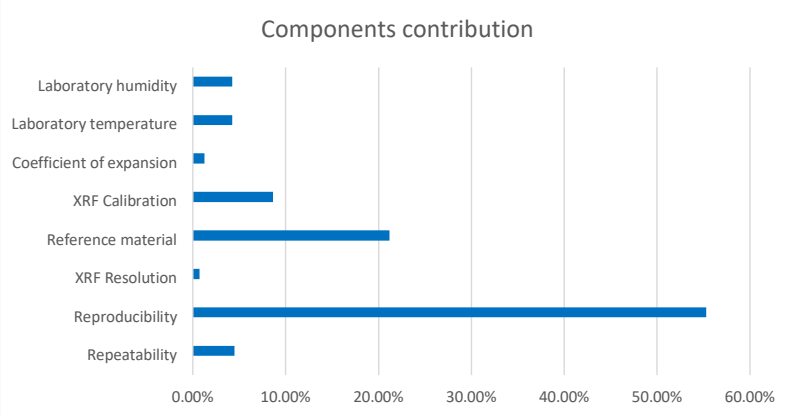
The definition of the uncertainty allows us to know the quality of the thickness measurements. A lower uncertainty shows that the processes are generally under control. Uncertainty is also a key element to prove the metrological traceability of measurements. Uncertainty estimation was performed according to method reported in the Guide to the expression of uncertainty in measurement (Mathew 2017). In the present investigation, methodologies were developed for NDT and DT thickness measurements. The measurand for X-ray fluorescence analysis can be defined with equation (1) and the corresponding uncertainty is shown in equation (2).

$$t = -\alpha \ln(1 - X_n) \tag{1}$$

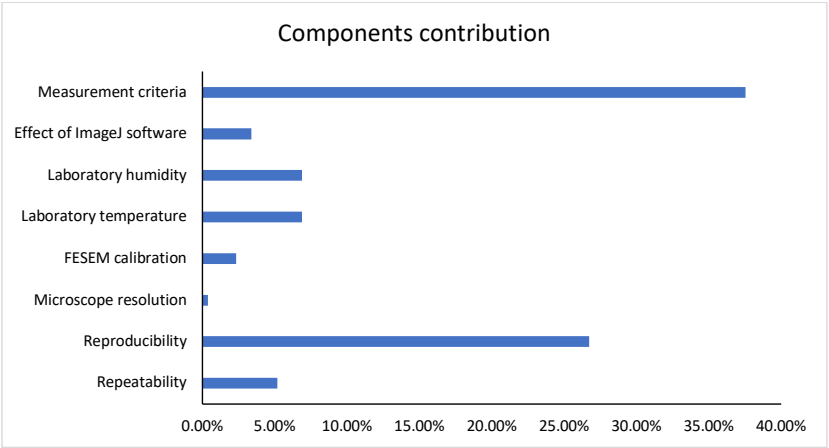
$$u_{tXRF} = \frac{\alpha}{1 - X_n} \tag{2}$$

Table 3. Examples of the uncertainty budgets for measurements obtained by floor XRF and image analysis with ES.

Measurement of the thickness of Ti coating obtained by PVD sputtering magnetron by XRF floor spectrometer					
Source of uncertainty	Average value	Source variability	Unit	Distribution type	Standard uncertainty
Coating length	1.17253	-	µm	-	-
Repeatability	-	0.14446	µm	Type A	0.00527
Reproducibility	-	3.17874	µm	Type A	0.06532
XRF Resolution	-	0.00300	µm	Type B	0.00087
Reference material	-	0.02000	µm	Type B	0.02500
XRF Calibration	-	0.05000	Hr	Type B	0.01020
Coefficient of expansion	-	0.05000	µm	Type B	0.00150
Laboratory temperature	-	0.01000	µm	Type B	0.00500
Laboratory humidity	-	0.01000	µm	Type B	0.00500



Measurement of the thickness of Ti coating obtained by PVD sputtering magnetron by ES					
Source of uncertainty	Average value	Source variability	Unit	Distribution type	Standard uncertainty
Thickness length	1.11944	-	µm	-	-
Repeatability	-	0.21065	µm	Type A	0.00384
Reproducibility	-	0.01942	µm	Type A	0.01980
Microscope resolution	-	0.0010	µm	Type B	0.00029
FESEM calibration	-	0.0030	µm	Type B	0.00173
Laboratory temperature	-	0.01000	˚C	Type B	0.00510
Laboratory humidity	-	0.01000	Hr	Type B	0.00510
Effect of ImageJ software	-	0.00500	µm	Type B	0.00250
Measurement criteria	-	0.027769	µm	Type B	0.02779



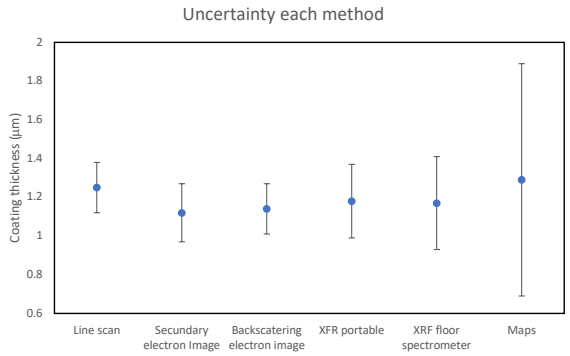


Fig 7. Comparison between methods used to determine Ti coating thickness.

Where:

t - coating thickness.

α - calibration constant.

X_n - Normalized intensity.

The segment between two points in a plane was defined as a measurand for thickness measurement through the cross-sectional analysis. The points are defined by coordinates as follows: A (x_1, y_1), B (x_2, y_2) and β is conversion factor. The measurand for cross-sectional analysis defined as the union of two points in a plane is shown in equations (3) and (4). The measurand is described by eq (3) and its uncertainty is shown in eq (4):

$$t = \overline{\beta AB} = \beta\sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2} \tag{3}$$

$$u_{tFESEM} = \frac{\beta(y_2 - y_1)}{\sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2}} \tag{4}$$

Figure 6 shows the cause-effect diagram proved to determine the uncertainties involved in the XRF and FESEM-EDS thickness measurements.

Table 3 presents the uncertainty budgets for XRF measurements and for measurements obtained by cross-sectional analysis using a FESEM-EDS.

Figure 7 shows the uncertainties obtained for each technique and method used for thickness measurement. In all methods, the thickness variations, related to the sample position in the planetary, are the most important characteristic. The coating thickness variation is correlated to the position of the base substrate metal in the planetary. The top-position produced the most heterogeneous coating thickness causing significant differences compared to the other coatings in the batch. For imaging methods, the effect of the measurement criterion is added. In the case of the elemental mapping there is a relevant factor due to the coordination of the measurement systems involved in the process. The coordination between measurement systems in FESEM can be considered a systematic error, derived from some mismatch between the analysis routines, so, by finding its origin it can be minimized.

Conclusions

A deposition process was designed for Ti coatings with a nominal thickness of 1 μm using the magnetron sputtering technique that allowed obtaining thicknesses with high levels of uniformity, homogeneity, and stability. An analysis of variables identified the effect of the position of the disc (substrate) in the planetary, as one of the main variables that are directly reflected in the reproducibility of the process, since, in the upper position the coating obtained has a more heterogeneous thickness than in the central and lower positions.

The characterization with NDT showed that it saves time and resources; however, the variation of the thickness measurements is higher than with DT. The XL 5 analyzer is preconfigured, so its uncertainty model is limited to these conditions. The floor spectrometer has a higher resolution and is more flexible because it can be configured ad hoc with the required chemical analysis. This allows a more detailed analysis of the components of the thickness variations, this study allowed observing that the uncertainty estimated for this technique is more affected by the reproducibility factor.

In the case of TD, thickness measurements were performed with the combination of point imaging and chemical microanalysis methods available at FESEM-EDS. Direct thickness measurements were also performed by image analysis. In this process it was identified that an important factor of variation is the measurement criteria. Thickness results with mapping and elemental profile require parameters and proper synchronization of the FESEM and the microanalysis system to achieve reliable measurements, these conditions generate long analysis times.

The uncertainty obtained for the elemental mapping technique was the highest and is attributed not only to reproducibility but also to the effect of harmonization between the measurement systems involved. For the mappings, it was not possible to make corrections by applying image analysis. For the elemental profile, it was possible to make corrections

by applying image analysis, which resulted in a lower uncertainty. The uncertainty estimation has allowed us to include this parameter as a quality criterion in the coating design. The results are encouraging to propose these metallic coatings deposited by magnetron sputtering as candidates for measurement standards.

Acknowledgments

All authors thanks to your institutions for support to realize of this project.

Access to the CENAPROT coatings laboratory CIDESI headquarters and to the FORDECYT/247/2018 297265 (Strengthening of the Research, Development and Innovation Capacities of CIDESI to Meet the Scientific-Technological Needs in Additive Manufacturing of the Industry in the Central-North Region of Mexico), FORDECYT 296384 (CONACyT consortium operation in additive manufacturing) funding projects are gratefully acknowledged.

Thanks to the program 2015-02-1077 (Study and Understanding of the Degradation and Stability of Lubricating Hard Coating Systems for Medium and High Temperature Applications) Frontiers of Science managed by CONACYT.

G.C. Mondragón-Rodríguez also thanks the Researchers for Mexico program (formerly Cátedras) sponsored by CONACYT. R. Herrera-Basurto is grateful for the support granted through the postdoctoral fellowship program and the SNI, both from CONACYT. A. Domínguez García, thanks to TMIC for the support.

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