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SELECTED POSIBILITIES OF THE DETERMINATION OF COATINGS THICKNESS

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Summary. The presented paper focus on the possibilities of the thin film thickness determination (TV). Thickness (TV) measured by Calotest method has been compared to the results achieved by SEM method on the brittle fracture. Obtained results show the inaccuracy.

Keywords: coating thickness; Calotest; confocal microscopy; SEM; GDOES

1. INTRODUCTION

Thin film thickness (TV) is a basic mechanical property of the thin film. Credible determination is essential for the checking before coated product has been delivered to customer as well as for loaded force determination during Hardness test. The indenter penetration depth should not be higher than 1/10 of thin film thickness. In the case of breaking this requirement, measured hardness and Young 'modulus will be affected by substrate hardness. Thin film thickness can be measured by many methods, for instance Calotest, contact profile roughness tester, confocal microscope, etc. Above mentioned methods are direct. On the other hand, thickness can be evaluated by indirect methods, for example from chemical composition by chemical profile determination (GDOES) or by SIMS (Secondary Ion Mass Spectrometry) method of coated surface.

The aim of presented paper is the thickness determination of evaluated thin film using different methods, the comparison of achieved results and to show the possibilities of thin film thickness measurements.

1.1 Selected methods of thin film thickness measurement

The most often used methods for thin film thickness evaluation are Calotest and SEM method of fracture surface. There can be used the chemical composition measurement by GDOES and surface roughness evaluation by: contact profile roughness tester, AFM, EFM or confocal microscope..

1.1.1 Calotest

The principle of this method is the calotte made on the thin film surface by a ball and grinding paste (Fig. 1). Thin film thickness can be calculated by circle observation and their diameters measurement (Fig. 2) using the mathematical equations for rectangular triangle.

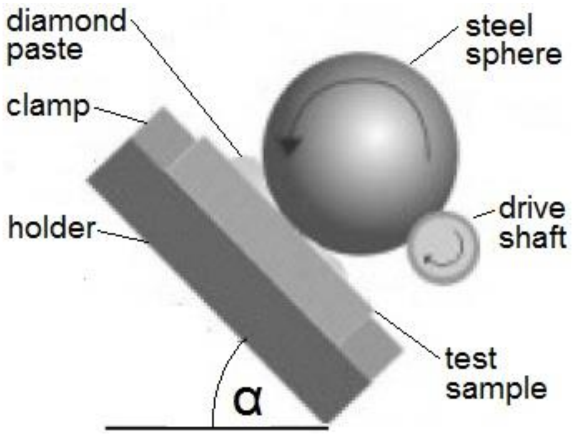

Figure 1 Calotest principle

Thin film thickness was measured in accordance with EN 1071-2 [1,3].

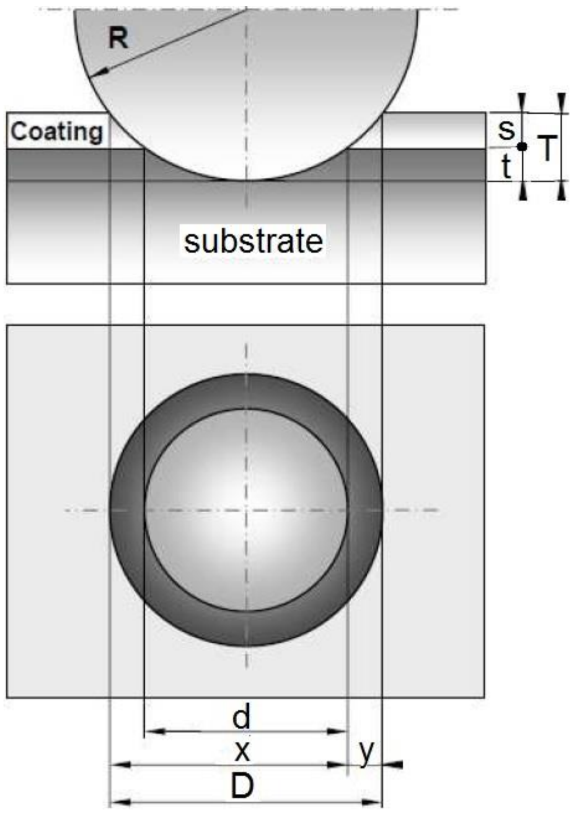

Figure 2 Calotest, Calotte dimensions [1,3].

According to Fig. 2, thickness s is calculated in accordance with equation:

$$s = T - t \quad (1)$$

where: T – total depth of penetration, t – depth of penetration in the substrate.

Total depth of penetration *T* is calculated in accordance to equation:

$$T = \mathrm{R} - (\tfrac{1}{2}\sqrt{4R^2 - D^2}) \tag{2}$$

where: R – ball diameter, D –external circle diameter of calotte.
Subsequently, depth of penetration in the substrate *t* is calculated in accordance with equation:

$$T = R - (\tfrac{1}{2}\sqrt{4R^2 - d^2}) \tag{3}$$

Afterwards equation (1) will be:

$$s = \frac{1}{2}\sqrt{4R^2 - d^2} - \sqrt{4R^2 - D^2} \tag{4}$$

For thin film, the penetration depth is small in comparison to radius *R* of the ball. Therefore, the equation is simplified to:

$$s = \frac{D^2 - d^2}{8\,R} \tag{5}$$

or, by substituting $D = x + y$ and $d = x - y$. Then equation (5) is:

$$s = \frac{\mathrm{x\,y}}{2\,R} \tag{6}$$

1.1.2. SEM analysis

Scanning electron microscopy analysis is used for evaluation of relief surface. Before observation, the evaluated surface has to be cleaned in dissolvent using ultrasonic waves and dried by hot air in order to get rid of organic substances. Prepared sample is located in the chamber where after locking the vacuum is created. Afterwards, the evaluated surface observation is realised with particular magnification (Fig. 3).

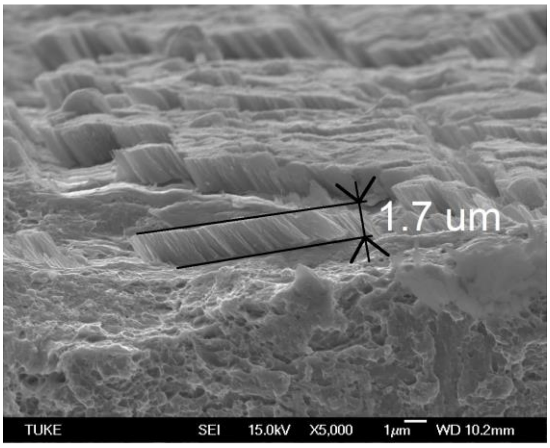

Figure 3 Fracture of Ti thin film, thickness 1.7 µm, SEM.

1.1.3. GDOES analysis

The chemical composition determination through evaluated surface cross-section can be obtained by GDOE method (Fig. 4). Its principle is based on chemical composition evaluation of de-dusting surface by Ar ions (Fig. 5). De-dusted material from evaluated surface is analysed in small volume in vacuum using the spectrometer. Particular chemical element volumes are evaluated in dependence on distance of de-dusted material from surface (Fig. 6). Measurement is accurate. Particular evaluated chemical elements plot curves and their proximity to 0 % presents a place where the coating is connected with substrate (Fig. 6). According to GDOES analysis the coating thickness is 1.7 μm.

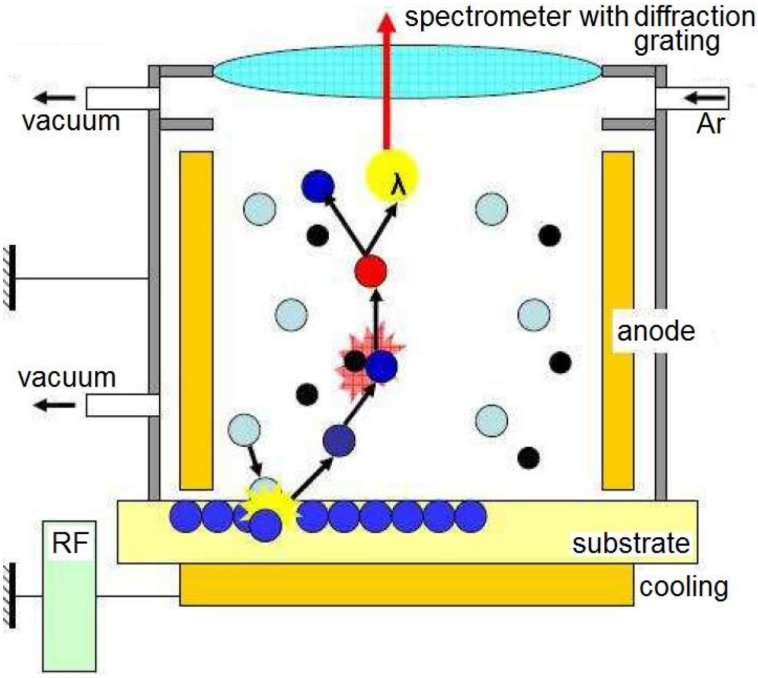

Figure 4 GDOES schematically [1,3].

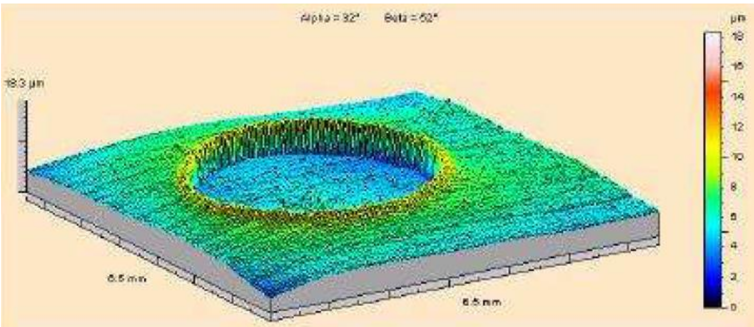

Figure 5 Crater after de-dusting of evaluated surface, confocal microscope [1,3].

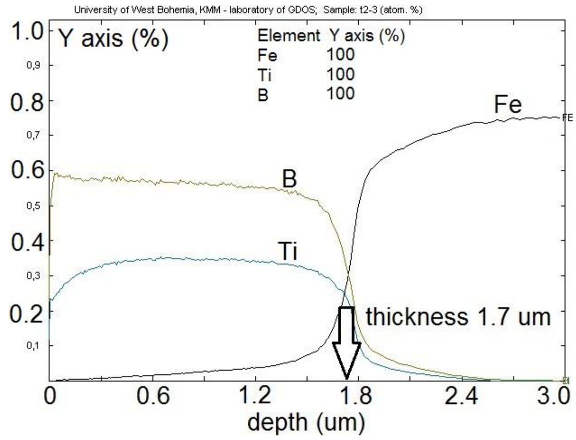

Figure 6 GDOES of chemical composition of TiB coating, thickness of the layer 1.7 μm, confocal microscope.

1.1.4. Surface roughness evaluation

Surface roughness can be evaluated using profile roughness tester, atomic force microscopy (AFM), Electromagnetic force microscopy (EFM) or confocal microscopy. Observed surface profile (roughness) was carried out on the interface surface – coating and surface without coating. Difference between those surfaces can be considered as a evaluated coating thickness, see Fig. 7, Al coating.

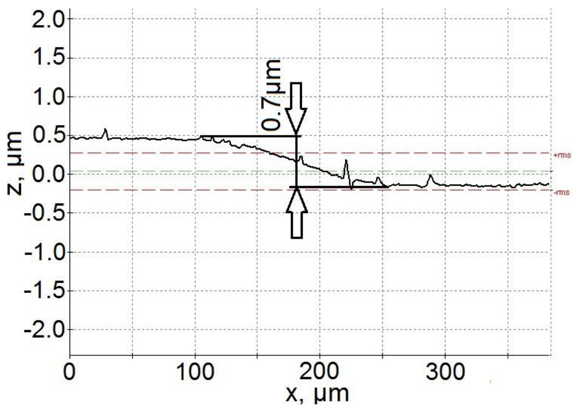

Figure 7 Profile curve of surface with Al coating, confocal microscope [2].

2. Experimental part

Sample with WC/C coating was used for thin film thickness evaluation. The coating was deposited on steel 12050 substrate by PECVD. Thin film thickness was evaluated by Calotest. Sample was cleaned by ultrasonic in acetone within 20 min. Subsequently, the sample was located in Calotest device and there was created the calotte using diamond grinding paste and the ball with diameter 25.4 mm. Grinding time was 5 min. Subsequently, the sample was located into optical microscope and the circle (Calot´s) diameters were measured. Evaluated coating thickness was calculated by MS Excel using equation (1). Measurement of thickness was carried out using SEM Jeol 7000F by cross-section fracture measurement. The sample was cooled by fluid nitrogen for 5 min. Subsequently, the sample was broken in order the coating was loaded by pull. For SEM observation, the sample was cleaned by ultrasonic waves for 20 min and dried by hot air. Afterwards, the sample was located in the chamber and vacuum was created. Measurement was realised on the particular place in order to see whole cross section of coating. Results achieved by measurement and calculation were compared. Finally, the coating thickness was determined by dependence of depth chemical concentrate profile by GDOES method.

3. Results and discussion

Coating thickness measured by Calotest achieved 1.14 µm. There were 3 calottes, external circle diameters were from 708.8 µm to 716.8 µm (Fig. 8). Internal circle diameters were from 595.4 µm (Fig. 8) to 644.0 µm. Evaluated thicknesses were 1.51 µm, 0.98 µm and 0.92 µm. Final value was calculated as an average of measured dimensions of grinded calottes.

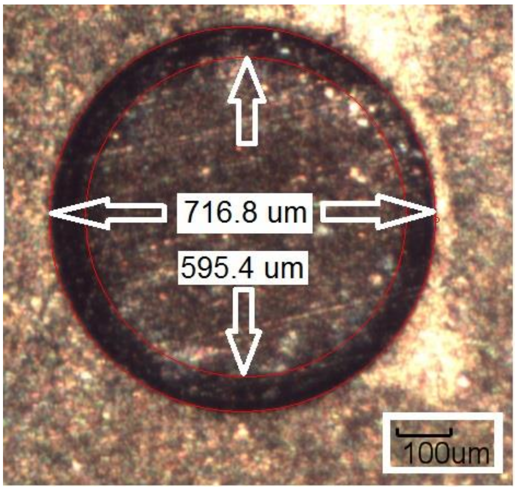

Figure 8 Calotte with circle dimensions created on surface with WC/C coating, thickness 0.98 µm, optical microscope.

Coating thickness measured by chemical concentrate profile method GDOES achieved 1.23 µm. It is obvious, that WC/C coating thickness determined by GDOES method was 7 % lower in comparison to thickness measured by cross section fracture method.

Coating thickness measured by cross section fracture of WC/C coating (Fig. 10) achieved 1.3 µm. That value is considered as the most accurate value and can be used as a standard in comparison to other methods. Based on used methods, the closest value was obtained by GDOES method.

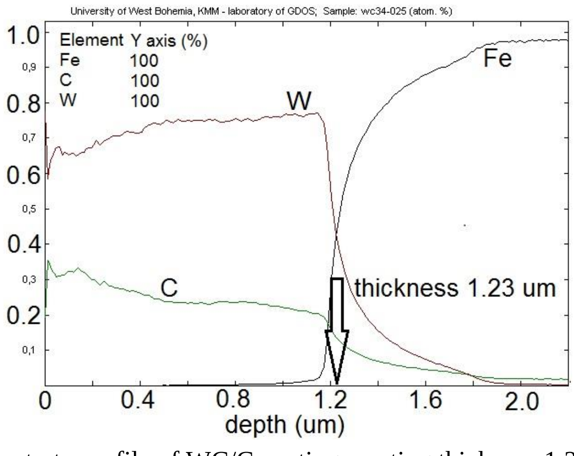

Figure 9 Chemical concentrate profile of WC/C coating, coating thickness 1.23 µm, GDOES analysis.

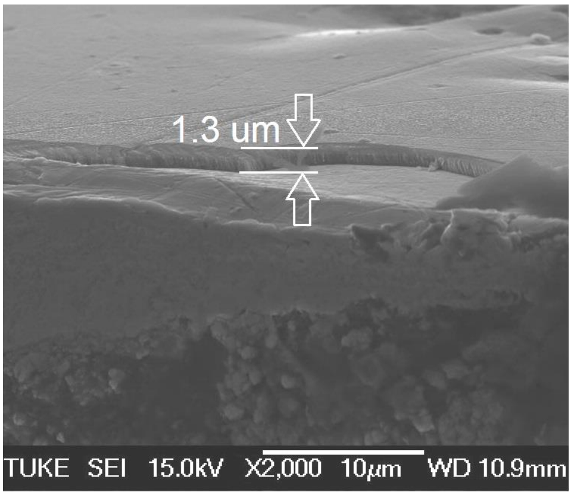

Figure 10 Cross section fracture of WC/C coating, coating thickness 1.3 μm, SEM.

4. CONCLUSIONS

Based on the measured results and cited literature, it can be stated that:

- WC/C coating thickness was measured using 3 different methods,
- Measured results were compared to thickness obtained by cross section fracture method,
- Coating thickness measured by SEM achieved 1.3 μm,
- Coating thickness measured by Calotest achieved 1.14 μm,
- Coating thickness measured by GDOES method achieved 1.23 μm,
- The closest value to standard was coating thickness measured by GDOES method.

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References

Journals:

[1] Kupczyk, M., Cieskowski, P., Libuda, P., Jakrzewski, D.: Opracowanie procedury przygotowania próbek do precyzyjnych pomiarów grubosci cienkich powlok wytworzonych metoda lukow-plazmowa: Zeszyty Naukowe Politechniki Poznanskiej, 2005, No. 2. P. 33-41.

Conference Proceedings:

[2] Kottfer, D. - Gajdoš, I. - Kaščák, Ľ. – Rehák F. Evaluation of mechanical properties of evaporated Al coating deposited on plastic substrate, In: *International Scientific Conference “The Modern Technologies of Polymer Materials Obtaining and Processes”,* Book of abstracts, Lviv, Septembre 21 – 23, 2016

Web sites:

[3] *Niektóre metody badaň wlasciwosci warstw TiN 2011.* Available at: http://vttkoszalin.pl/files/Metody%20badan%20wlasciwosci%20twardych%20warstw.PDF