11th International Research/Expert Conference
"Trends in the Development of Machinery and Associated Technology"
TMT 2007, Hammamet, Tunisia, 05-09 September, 2007.

CHARACTERIZATION OF SPUTTER CRATERS AFTER GDOES EXPERIMENT PERFORMED ON TiAlN COATINGS

Lidija Ćurković[1], Đurđica Goršćak[2], Vera Rede[1], Sanjin Mahović[1], Ivica Petrinić[1]

[1]**Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb**
Ivana Lučića 5, Zagreb
Croatia

[2]**Končar-Alati d.d.**
Fallerovo šetalište 22, 10000 Zagreb
Croatia

ABSTRACT
GDOES is a rapid depth profiling technique. This analysis is destructive and there is a formation of sputter craters on the sample surface. In this work the depth profiling method GDOES is used for determining the thickness of TiAlN coating after determining the sputtering rate, as well as the crater microstructure. The GDOES depth profiles were measured by means of spectrometer GDS 750, Leco. The obtained data were compared with the thickness values obtained from the optical microscopy and the SEM fracture cross-sections. Structure of sputter craters after GDOES experiment at different coating depths were evaluated by the optical microscopy and the scanning electron microscopy (SEM). The crater shape and depth were measured by means of Perthometer. The investigated TiAlN coatings were deposited on tool steel AISI D2 of Uddeholm Company under the trade name Sverker 21 by cathode arc evaporation technique (BAI 1200) in Balzers (Kapfenberg, Austria).
Keywords: alumina ceramics, corrosion resistance, roughness parameters.

1. INTRODUCTION

Tool steels were developed to resist wear at temperatures of forming and cutting applications. They are divided into six categories: cold work, shock resisting, hot work, high speed, mold and special-purpose tool steels. Cold work tool steels are the most important category because they are used for many types of tools and dies and other applications where high wear resistance and low cost are needed. Surface engineering such as surface treatment, coating and surface modification are employed to increase the surface hardness and minimise adhesion (reduce friction) and improve wear resistance of tool steel substrates. PVD hard coatings are well known for providing engineering surfaces with high hardness and high tribological properties. It is known that alloying of TiN coatings with additional elements could introduce marked effects on coating properties and performance [1,2,3]. Numerous studies have been carried out to manufacture multicomponent nitride coatings as alternatives to TiN coating for surface protection of cutting and forming tools. For instance, TiAlN coating is a common multicomponent system that yields superior oxidation resistance, and can significantly increase lifetime to cutting tools in comparison with conventional TiN coating [4].
In this paper, the depth qualitative composition profiles, sputtering rate and the thickness of TiAlN coating were determined by the glow discharge optical emission spectrometry (GDOES) method. The chemical compositions of each crater were determined by means of SEM with EDS detector. Structures of sputter craters after GDOES experiment at different coating depths were evaluated by the optical microscopy and the scanning electron microscopy (SEM).

Primarily, depth profiling methods using ion sputtering deliver a sputter (measuring) time scale, and not a depth scale. Converting the time scale into the true depth scale is not easy, because many different sputter effects may occur and, especially at interfaces with changing concentration gradients, stable and well defined conditions are not reached [5].

2. EXPERIMENTAL

TiAlN coatings was prepared in Balzers BAI 1200 deposition system by cathode arc evaporation technique (coating name: Lumena). Coatings were deposited onto the AISI D2 (DIN 1.2379) type tool steel substrates. The steel sample was finely ground, ultrasonically cleaned and sputter cleaned prior to coating deposition in order to obtain optimum adhesion between coating and substrate. The final macrohardness of tool steel AISI D2 after the heat treatment was 61 HRC in agreement with the tempering graphs [6]. The thickness of hard coatings was determined by the GDOES depth profiles by means of spectrometer GDS 750 (Leco) as well as from optical microscopy and SEM fracture cross-sections. The depth of spatter craters obtained from GDOES analysis is evaluated after determination sputtering rate. Sputtering rate (SR) was determined by measuring depth of crater at different time (51, 100 and 150 s) of GDS analysis. The crater shape and depth was measured by means of Perthometer S&P 4.5 (Feinprut Perthen GmbH, Germany). Structure of each sputter crater and thickness of TiAlN coating were evaluated with optical microscopy (Olympus BH) and scanning electron microscopy (TESCAN VEGA TS 5136 MM). The chemical composition of each sputter crater was evaluated by means of SEM analysis with EDS detector. Spattering rate of each sputter crater was determined according to the following equation:

$$SR = \text{depth of crater} / \text{spattering time} \ (\mu m/s).$$

After determination of average spattering rate (Table 2), the thickness of investigated TiAlN coating was determined by means of GDOES analysis.

3. RESULTS AND DISCUSSION

The chemical composition of the investigated tool steel is presented in Table 1.

Table 1. Chemical composition of the tool steel in wt %.

Steel type	Mass concentration of elements (%)					
	C	Mn	Si	Cr	V	Mo
AISI D2	1.55	0.40	0.30	11.80	0.80	0.80

The result shown in Figure 1 indicate that the sputtering was not uniform, because crater bottoms are not quite flat. Also, it means that removal of coating material is not uniform (Figure 2, Figure 3, Table 3). This non-uniform removal of coating material is the main reasons for the shape of sputter craters and the curves produced by GDS analysis (Figure 2).

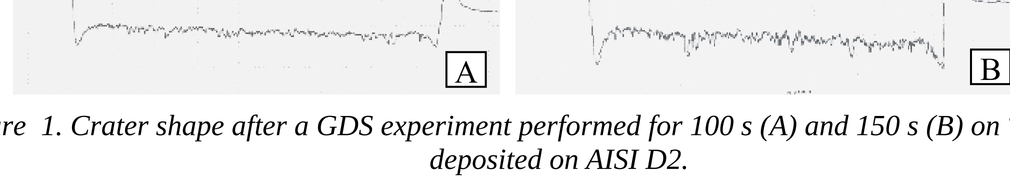

Figure 1. Crater shape after a GDS experiment performed for 100 s (A) and 150 s (B) on TiAlN deposited on AISI D2.

The average TiAlN coating sputtering rate was 45.3 nm/s, as shown in Table 2.

Table 2. The calculate average values of spattering rate of TiAlN coating.

t, s	depth, μm	spattering rate (*SR*), nm/s	average *SR*, nm/s
51	2.535	49.7	45.3
100	4.380	43.8	
150	6.360	42.4	

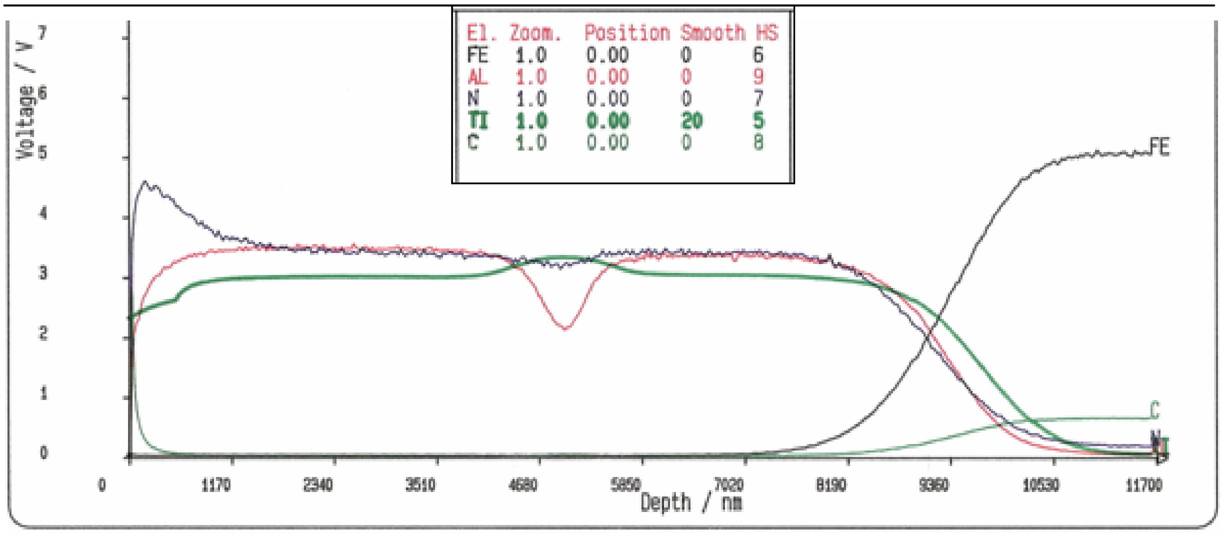

Figure 2. Dept profiles for TiAlN coating.

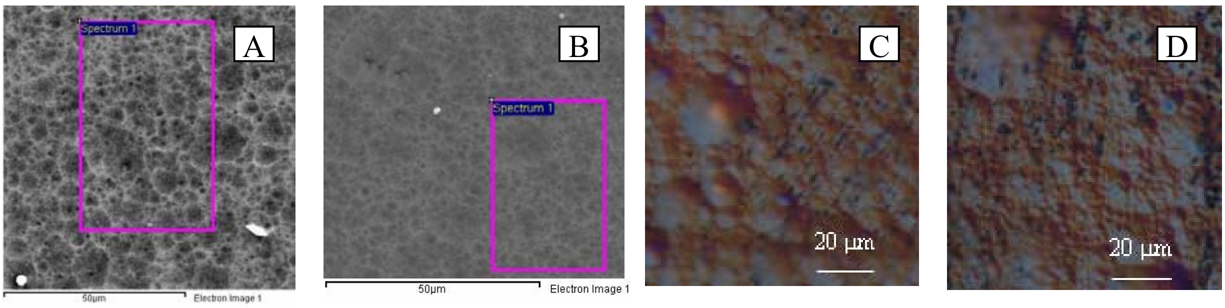

Figure 3. Microstructure of sputter craters evaluated by optical microscopy after 100 s (A) and 150 s (B) of GDS analysis. Microstructure of sputter craters evaluated by SEM after 100 s (C) and 150 s (D) of GDS analysis.

Typical SEM micrographs illustrating the cross-section views of TiAlN coating deposited by cathode arc evaporation technique (coating name: Lumena) are shown in Fig. 4 B, C. The interface between the coating and the substrate is sharp and without irregularities (Fig. 4). The microstructure is slightly columnar, but it is not clearly expressed. Coating thickness of Lumena was 9.0 μm. Similar results were obtained by measuring thickness by means of the GDOES (9.1 μm) and optical microscopy (8.9 μm). This means that the GDOES method can be used for thickness determination of TiAlN coating after determination of sputtering rate.

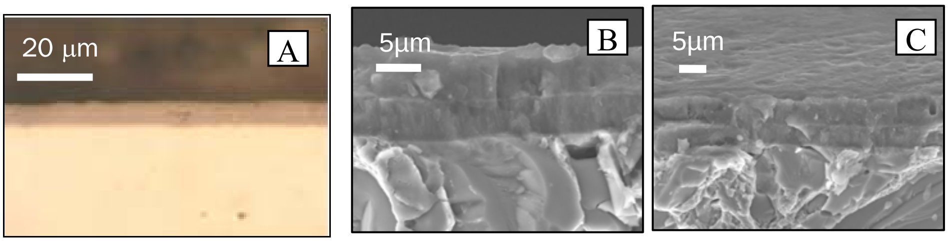

Figure 4. Optical microscopy (A) and SEM fracture cross-sections of TiAlN coating(B, C).

Table 3. Chemical composition of sputter crater after 100 and 150 s analysis by GDOES. Results are obtained from SEM analysis using EDS detector.

t, s	Ti, wt. %	Al, wt. %	N, wt. %
100	54.37	16.77	28.86
150	51.13	23.26	25.62

4. CONCLUSION

The obtained result of measuring the thickness of TiAlN coating using the GDOES method comply with the results obtained by optical microscopy and scanning electron microscopy. This GDOES method is fast, and doesn´t need any special sample treatment. The result indicates that it is possible to convert the time scale into the true depth scale. Removal of coating material is not uniform because changing concentration of coating elements across the layer was noted.

5. ACKNOWLEDGMENTS

The presented research results were achieved within the scientific project "Structure and properties of engineering ceramics and ceramic coatings" (Agreement No 120-1201833-1789; 10106-797300-1) supported by the Croatian Ministry of Science, Education and Sport.

6. REFERENCES

[1] Jehn Herman A., Rother B.: Homogenity of Multi-Component PVD Hard Coatings, Surf. Coat. Tech., 112 (1999) 103-107.

[2] Goršćak Đ., Panjan P., Ćurković L., Kapudžija D.: Mechanical Properties and Application of Various PVD Hard Coatings in Cold Work Tool, Proceedings of 7th International Tooling Conference, ed. M. Rosso, M. Actis Grande, D. Ugues, Torino (2006) 473-480.

[3] Goršćak Đ., Panjan P., Čekada M., Ćurković L.: Comparison of Mechanical Properties of Various PVD Hard Coatings for Forming Tools., 1st Interantional Conference on Heat Treatment and Surface Engineering of Tools And Dies, Ed. Božo Smoljan, Pula, CSHTSE (2005) 211-216.

[4] Jindal P.C., Santhanam A.T., Schleinkofer U., Shuster A.F.: Performance of PVD TiN, TiCN, and TiAlN Coated Cemnted Carbide Tools in Turning, Int. J. Refract. Met. Hard Mater., 17 (1999) 163-170.

[5] Oswald, S., Baunack, S.: Comparison of depth profiling techniques using ion sputtering from the practical point of view, Thin Solid Films, 425 (2003) 9-19.

[6] Sverker 21, cold work tool steel. Tool steel Facts, Uddeholm Company.