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Effect of the auxiliary cathode on the thickness of the HiPIMS TiAlN coating deposited on the inner surface of the tube

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Abstract: The paper is focused on influencing the thickness of the TiAlN layer deposited using the high power pulse magnetron sputtering (HiPIMS) method on the inner surface of the tube. The HiPIMS method makes it possible to deposit layers at temperatures up to 400°C. An auxiliary cathode placed in the axis of the coated tube was used. The auxiliary cathode made it possible to deposit an AlTiN layer with a thickness in the center of the coated tube up to 45% higher compared to the thickness of the layer deposited without an auxiliary cathode.The layer thickness was measured to be from 1.5 µm to 2.2 µm for the layer deposited without an auxiliary cathode and 1.05 µm to 1.95 µm forthe layer deposited with the wire as the cathode in the axis of the coated tube. Adhesion of the evaluated layers showed the degree of HF2 at the measured thickness points.

1 Introduction

Coatings deposited on the functional surface of the component enable the desired properties of the functional surface to be achieved. Currently, the HiPIMS (High Power Impulse Magnetron Sputtering) method belongs to the improved MS (Magnetron Sputtering) methods. The HiPIMS method uses very short pulses with a power density on the target surface (during the pulse) that exceeds the typical DC power density by approximately two orders of magnitude (on the order of kW/cm2). This increases the ionization of the sputtered material and creates a metal-based plasma as opposed to a gas plasma for conventional sputtering. The duty cycle is small (<10%), the heating of the target can be regulated. The initially high negative bias that is used leads to an increase in the kinetic energy of the charged particles that bombard the surface of the deposited TV [1,2]. TVs deposited by the HiPIMS method have a denser, less columnar structure, where renucleation usually occurs [1-3]. Compared to the MS method, the HiPIMS method allows the target material to be dedusted in short pulses ranging from 50 µs to 200 µs.

TiN layers are among the basic coatings for improving the wear resistance of FP. By adding Al to the dedusted target, TV TiAlN is created, which additionally has a higher resistance to oxidation and good tribological properties, such as, above all, a low coefficient of friction and wear [4].

Coating the inner surface of the tube, where the tube rotates around an axis passing through the center of the tube perpendicular to its axis - (see Fig. 2), with vacuum methods also has a disadvantage: the thickness of the deposited TV is not constant but decreases from the edge of the tube to the center, where it is the thinnest [5].

In the article, the authors focused on influencing the thickness unevenness of the TiAlN coating deposited by the progressive HiPIMS method into a tube with a diameter of 70 mm and a length of 100 mm using the above-mentioned auxiliary cathode. The adhesion and chemical composition of the TiAlN coating at the point of thickness measurement was also evaluated.

2 Methodology

The TiAlN coating was deposited from a magnetron target in the ratio Ti/Al=50:50 (dimensions approx. 180x500 mm) on samples of C45 steel also steel 12050 (STN 412050) in the shape of a disc with a diameter of 22 mm and a thickness of 4 mm. The chemical composition of steel (wt.%) is 0.42 ÷ 0.50% C, 0.40% Si, 0.50 ÷ 0.80%

Mn, 0.40% Cr, 0.10% Mo, 0.40 % Ni and 0.035% P [6]. The functional surfaces of the samples were gradually polished using diamond pastes with a grain size of 15 µm, 9 µm, 3 µm and 1 µm. A surface roughness (Ra) of approx. 12 nm. After that, the samples were cleaned in acetone by ultrasound for 10 minutes and later dried with warm air for 5 minutes. Finally, the samples were placed using magnets on the inner surface of the tube located in the vacuum chamber on the cathode (Figure 1,2). The dimensions of the tube were length 100 mm and diameter 100 mm. Because the fixing magnets had a length of approx. 25 mm and the thickness of the coated samples was approx. 4 mm, the pipe diameter can be reduced to 70 mm. The bombardment of the surface of the Ar samples (99.999%) took place in the DC (direct current) mode using two magnetrons located opposite each other around the perimeter at a pressure of 1.6 mPa, where the bias voltage of the holder (bias Ub) was -200 V, the magneton power was 1.6 kW and the cleaning time of the tube surface before the deposition process by Ar ion bombardment (etching) was 60 minutes. The Ar flow rate in the vacuum chamber was 300 cm3×min-1.

The deposition process was performed without and with the cathode located in the axis of the coated tube (Figure 1,2). The deposition lasted 210 minutes with the following parameters: 4 magnetrons placed opposite each other around the perimeter of the vacuum chamber were used, each magnetron having a power of 12.5 kW, cathode bias voltage -60 V, N2 flow rate (99.999%) 230 cm3xmin-1, Ar flow rate (99.999 %) 300 cm^3xmin^{-1} and the pressure in the vacuum chamber 4.7 mPa. The temperature of the coated samples did not exceed 300°C. The sample table rotated at a speed of 0.5 revolutions per minute. A CEMECON CC800 HiPIMS device was used.

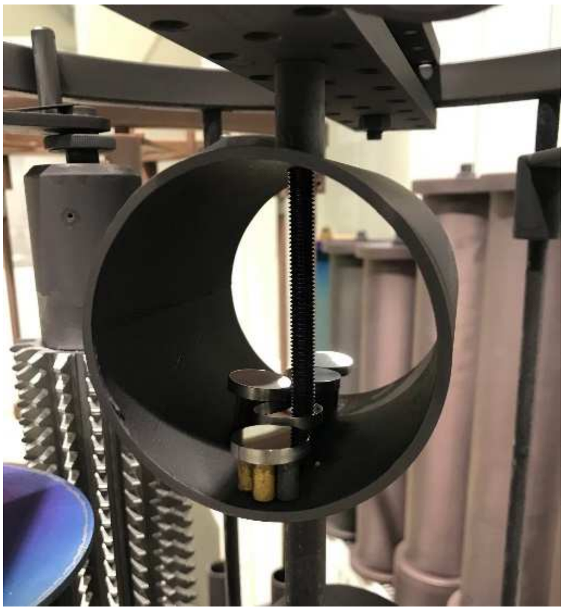

Figure 1 Placement of samples in a tube-shaped fixture (L=100) without a cathode in the tube axis in a vacuum chamber

The thickness of the TiAlN layer was evaluated by the Kalotest method with CSM Instruments. In order not to cut the coated pipe, the steel samples described above were used, placed in five places on the inner surface of the tube (Figure 1,2). A steel ball with a diameter of 15 mm and an abrasive paste with a diamond grain size from 0.5 to 1.0 µm were used for 300 s at a ball speed of 900 per minute. Figure 3 shows the dome from a thickness measurement at a point 10 mm from the edge of the tube with a TiAlN coating deposited by a wire as the cathode in the axis of the coated tube (Figure 2).

Adhesion was evaluated using the Mercedes test method, where the indentation of the edge of a diamond cone (Rockwell indenter) into the surface of the coated sample with a loading force of 1500 N was evaluated. The scale for evaluating adhesion is shown in Figure 3. The indentation edge was observed and evaluated with an Olympus 5000 optical microscope.

Figure 2 Placement of samples in a tube-shaped fixture (L=100 mm) with the cathode in the axis of the tube in a vacuum chamber

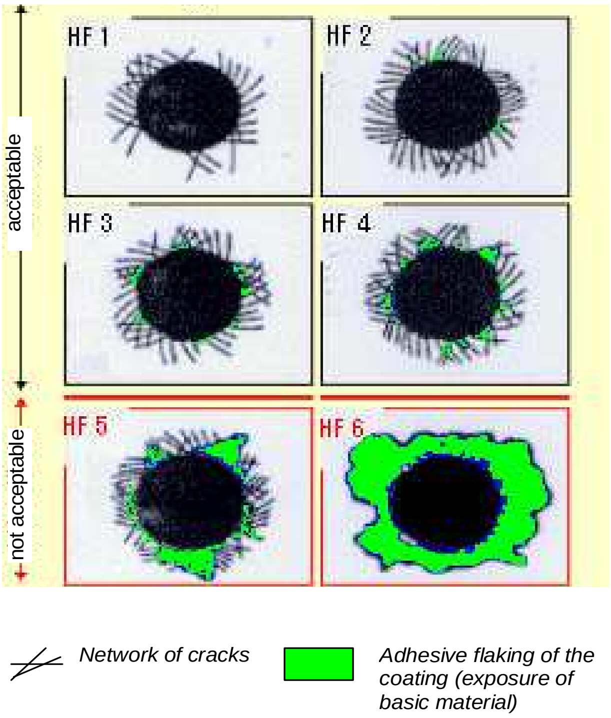

Figure 3 Mercedes test scale" HF1-HF 4 - satisfactory, HF4-HF6 - unsatisfactory adhesion [7]

The surface of the TiAlN layer was observed using the VEGA 3 TESCAN thermal emission electron scanning microscope scanning device. The chemical composition was evaluated using an EDX analyzer x-act Oxford Instruments as an area analysis of the surface of the evaluated coating.

3 Results and discussion

A view of the surface and chemical composition of the TiAlN layer deposited without an auxiliary cathode from the edge of a 100 mm long tube is in Figures 4 and 5. The surface of the evaluated coating deposited with an auxiliary cathode is in Figure 6, the chemical composition is in Figure 7. Area chemical analysis showed that at by using an auxiliary cathode, the nitrogen content in the layer decreased and, on the other hand, the content of Al and Ti slightly increased (Figure 5 and 7). This may be due to the auxiliary cathode, which caused a denser field of dedusted Ti and Al particles in the inner space of the tube.

A view at the surface of AlTiN layers (Figs. 4 and 6) shows the occurrence of voids (surface without layer) with a diameter of approx. 20 nm to 50 nm. This indicates a similar structure and mechanical properties of the evaluated coatings.

Other properties of AlTiN coatings were not evaluated.

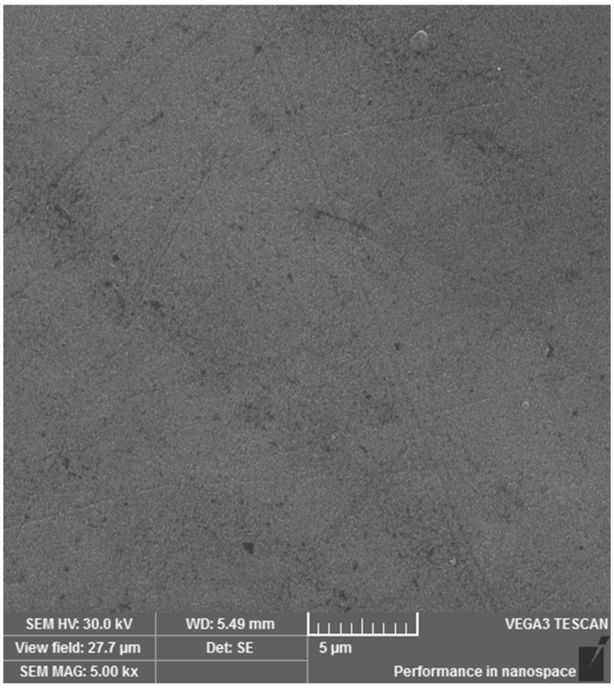

Figure 4 View of the surface of the AlTiN layer (tube length 100 mm) deposited without an auxiliary cathode, SEM

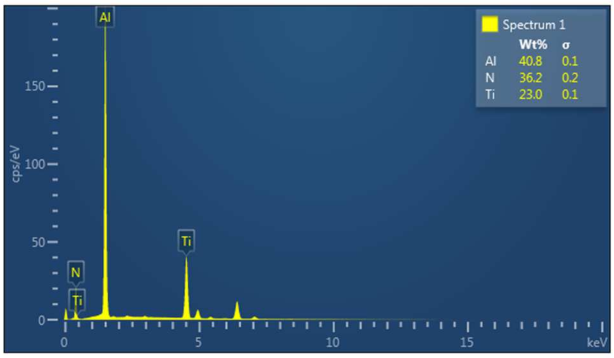

Figure 5 Chemical composition of the AlTiN layer (tube length 100 mm) deposited without an auxiliary cathode, EDX

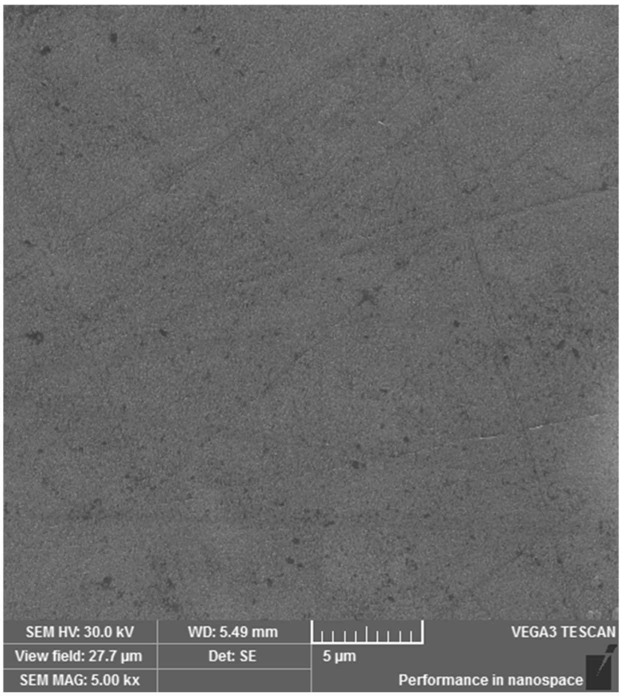

Figure 6 View of the surface of the AlTiN layer (tube length 100 mm) deposited with an auxiliary cathode, SEM

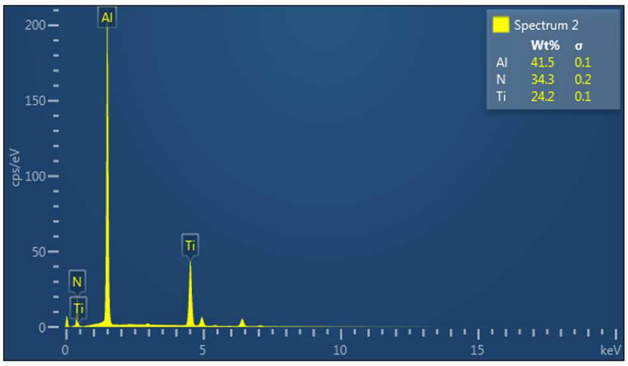

Figure 7 Chemical composition of the AlTiN layer (tube length 100 mm) deposited with an auxiliary cathode, EDX

The thickness of the coating deposited on the inner surface of a tube with a diameter of 70 mm and a length of 100 mm was evaluated.

In the case of a tube L=100 mm, the measured values were 1.5 to 2.2 µm in the case of a coating deposited with a wire as a cathode in the axis of the coated pipe (Figure 8). The thickness of the layer is one third lower in the middle of the tube compared to the thickness value at the ends of the tube (Figure 4). The diameter of the thickness measured at the edge of the tube (L=100mm) is shown in Figure 9.

In the case of the measured values of the thickness of TiAlN layers deposited without wire (L=100 mm) as an auxiliary cathode in the axis of the tube, the measured values were from 1.05 µm (in the center) to 1.95 µm (10 mm from the edge), which is approximately 10% less compared to the thickness of the layer deposited with the wire measured at a point 10 mm from the edge of the tube. The thickness of the evaluated layer measured in the center of the tube wall is up to 30% lower compared to the layer deposited with the wire in the tube axis (Figure 8). It can also be stated that the thickness in the center of the tube has decreased to 55%, which is significantly more than in the case of the deposited layer without wire in the axis of the tube. This may be due to the lower plasma density in the tube space [2,8].

The adhesion of the evaluated coatings showed a grade of HF 2 (Figure 10).

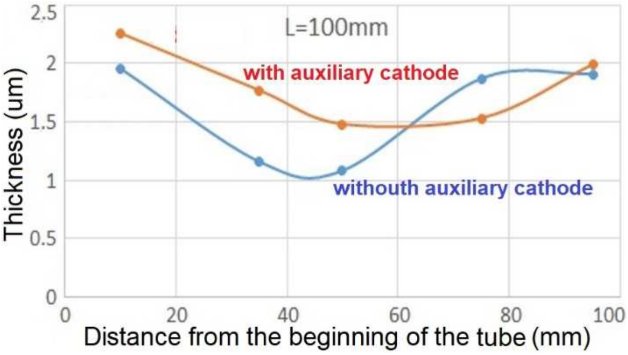

Figure 8 Dependence of the TiAlN layer thickness on the measurement position in the tube, L=100 mm

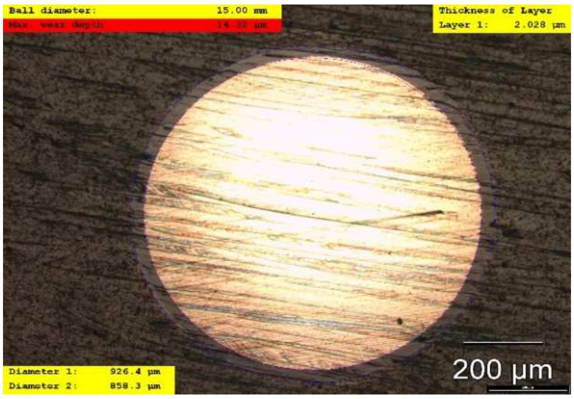

Figure 9 TiAlN layer calote, measured thickness 2.028 µm

Figure 10 Puncture after Mercedes test HF level 2

4 Conclusions

A TiAlN layer was deposited on the inner surface of the tube with and without an auxiliary cathode located in the axis of the tube, where:

- thicknesses of the assessed layer from 1.05 µm to 2.2 µm (tube L=100 mm).
- a significantly lower decrease in thickness was measured in the case of using an auxiliary cathode in the pipe axis L=100 mm (approx. 30%) compared to the deposition of a layer without an auxiliary cathode (45%). This was caused by a better distribution of the plasma in the space of the tube, achieved by placing the auxiliary cathode in the axis of the tube.
- adhesion of the TiAlN layer in all property's evaluation points was satisfactory, HF 2.

Further research will focus on the influence of technological parameters on the growth of the thickness of the AlTiN layer deposited on the inner surface of the tube by the HiPIMS method.

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References

[1] SARAKINOS, K., ALAMI, J., KONSTANTINIDIS, S.: High power impulz magnetron sputtering: A review on scientific andengineering state of the art, *Surface and Coating Technology*, Vol. 204, pp. 1661-1684, 2010. https://doi:10.1016/j.surfcoat.2009.11.013

[2] LI, C., TIAN, X., GONG, C., XU, J., LIU, S.: Synergistic enhancement effect between external electric and magnetic fields during high power impulse magnetron sputtering discharge, *Vacuum*, Vol. 143, pp. 119-128, 2017. https://doi.org/10.1016/j.vacuum.2017.06.007

[3] GRECZINSKI, G., LU, J., BOLZ, S., KOELKER, W., SCHIFFERS, CH., LEMMER, O., PETROV, I., GREENE, J.E., HULMAN, L.: Novel strategy for low-temperature, high-rate growth of dense, hard, and stress-free refractory ceramic thin films, *Journal of Vacuum Science & Technology A.*, Vol. 32, No. 4, 041515, 2014. https://doi.org/10.1116/1.4884575

[4] DENG, Y., TAN, C., WANG, Y., CHEN, L., CAI, P., KUANG, T., LEI, S., ZHOU, K.: Effects of tailored nitriding layers on comprehensive properties of duplex plasma-treated AlTiN coatings, *Ceramics International*, Vol. 43, No. 12, pp. 8721-8729, 2017. https://doi.org/10.1016/j.ceramint.2017.03.209

[5] YANHUI, Z., CHAOQIAN, G., WENJIN, Y., YUQIU, CH., BAOHAI, Y.: TiN films deposition inside stainless-steel tubes using magnetic field-enhanced arc ion plating, *Vacuum*, Vol. 112, pp. 46-54, 2015. https://doi.org/10.1016/j.vacuum.2014.11.014

[6] STN 412050, Steel 132050, [Online], Available: http://ust.fme.vutbr.cz/tvareni/img/databaze/ocel_12050.3.pdf [4 May 2023]. (Original in Czech)

[7] VDI 3198, ASTM C1624 - 05(2010). Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point Scratch Testing, West Conshohocken, PA, 2010, [Online], Available: http://www.astm.org/Standards/C1624.htm [4 May 2023], 2010.

[8] LI, C., TIAN, X., GONG, C., XU, J.: The improvement of high power impulse magnetron sputtering performance by an external unbalanced magnetic field, *Vacuum*, Vol. 133, pp. 98-104, 2016. https://doi.org/10.1016/j.vacuum.2016.08.020

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