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# Mechanical and tribological properties of the $TiB_2$ coating deposited by HiPIMS method

**Lukas Rusinko**
Alexander Dubček University of Trenčín, Faculty of Special Technology, Department of Mechanical Technologies and Materials, Ku Kyselke 469, 911 06 Trenčín, Slovak Republic, EU, lukas.rusinko@tnuni.sk
**Patrik Kluciar**
Alexander Dubček University of Trenčín, Faculty of Special Technology, Department of Mechanical Technologies and Materials, Ku Kyselke 469, 911 06 Trenčín, Slovak Republic, EU, patrik.kluciar@tnuni.sk
**Jozef Trebuna**
Alexander Dubček University of Trenčín, Faculty of Special Technology, Department of Mechanical Technologies and Materials, Ku Kyselke 469, 911 06 Trenčín, Slovak Republic, EU, jozef.trebuna@tnuni.sk
**Andrej Dubec**
Alexander Dubček University of Trenčín, Faculty of Industrial Technologies in Puchov, Ivana Krasku 491/30, 020 01 Púchov, Slovak Republic, EU, andrej.dubec@tnuni.sk
**Daniel Kottfer**
Alexander Dubček University of Trenčín, Faculty of Special Technology, Department of Mechanical Technologies and Materials, Ku Kyselke 469, 911 06 Trenčín, Slovak Republic, EU, daniel.kottfer@tnuni.sk (corresponding author)

***Keywords:*** HiPIMS, $TiB_2$ coating, hardness, Young´s modulus, coefficient of friction.
***Abstract:*** In the article, the authors briefly describe the high power impulse magnetron sputtering (HiPIMS) method. This method is briefly characterized in the article. Authors researched the mechanical and tribological properties of the $TiB_2$ coating deposited by the HiPIMS method on a steel substrate on an industrial machine. They were measured: hardness 25 GPa, Young's modulus 240 GPa, adhesion HF1, thickness 4.1 µm and coefficient of friction 0.7.

## 1 Introduction

Titanium is used as a transition thin coating to ensure the desired adhesion [1]. By adding nitrogen, its hardness increases and is used as a protective TiN thin coating of cutting tools [2]. By adding boron to Ti, we obtain a $TiB_2$ coating whose hardness is higher than that of the TiN coating [3-7]. The $TiB_2$ coating can be deposited by CVD method [3,4], PVD method by evaporation [5-7], RF sputtering [8-17], HiPIMS methods [18-20]. Often, the properties of $TiB_2$ coatings are doped with elements [21-22].

JI CHENG DING et al. [21] deposited a HiPIMS $TiB_2$ coating of approx. 1.5 µm thick at a frequency of 80 Hz, a pressure of 0.7 Pa, the deposition time was 120 min., the deposition temperature was 200 °C They reached a hardness of 26 GPa and a Young's modulus of 198 GPa.

ZHANG T.F. et al. [18] deposited a HiPIMS $TiB_2$ coating for 120 min. at a pressure in the vacuum chamber of 0.4 Pa, a temperature of 300 °C and 300 °C, a bias of 0 to 200 V and a distance from the magnetron of 12 cm. They achieved the following properties: the thickness of the coating was approx. 2 µm at both deposition temperatures, the hardness was from 20 GPa to 53 GPa (300 °C) and 17.5 GPa to 46 GPa (200 °C).

The HiPIMS method is an improved PVD sputtering method. It consists in the fact that the DC voltage during magnetron sputtering is rectified by interruption (Figure 1) [19]. The DC generator charges the capacitor bank of the pulse unit. The energy stored in the capacitors is dissipated into the plasma in pulses of precisely defined width and frequency using ultra-fast switches. The charging voltage of a capacitor bank typically ranges from several hundred V to several kV. The accumulated energy is released in pulses of a defined width and frequency in the range of several µs using transistors located between the capacitors and the cathode with switching capability, which enables the deposition of a coating with a denser structure, with higher hardness and resistance to wear.

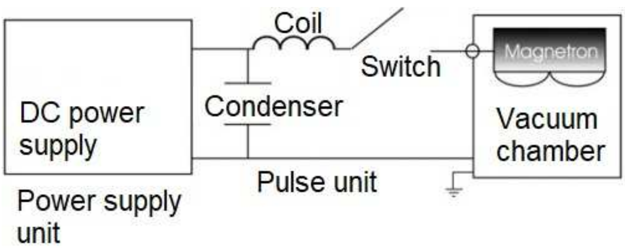

*Figure 1 HiPIMS power supply architecture diagram [19]*

The article is focused on the research of the mechanical and tribological properties of the $TiB_2$ coating deposited by the HiPIMS method and their comparison with published results in foreign scientific journals. The achieved results will serve to select the frequency and impulse width for further research on the $TiB_2$ HiPIMS coating to obtain higher hardness and a lower coefficient of friction.

## 2 Methodology

To evaluate the thickness of the coating, adhesion and coefficient of friction and wear, steel samples were used, which had the shape of a cylinder with dimensions d=22 mm and h=4 mm (Fig. 2), the material of the samples is steel (according to STN EN 19 830), whose chemical composition is (at.%): C - 0.8%; Mn - 0.45%; Si - 0.45%; Cr - 4.15%; W - 6.60%; Mo - 4.95% and V - 1.9%.

A TiB2 coating deposited by High Power Impulse Magnetron Sputtering (HiPIMS) on a CC800 device from Camecon, SRN (Figure 3) was used. Four Ti40B60 targets were used, which had dimensions of approx. 500x180 mm. The vacuum chamber of the device is 720 mm wide, 940 mm high and 920 mm deep, its volume is approx. 600 $dm^3$. Deposition of the $TiB_2$ coating took 3 hours and 55 minutes. The working gas Ar was used with a flow rate from 380 to 570 $cm^3$/min. During the deposition process, four Ti40B60 targets (magnetrons 1, 2 and 5, 6 - Figure 2) were used, which were arranged around the perimeter of the vacuum chamber opposite each other. In the center of the vacuum chamber, a table rotated with a planetary movement of the samples (Figure 3). The power of each HiPIMS magnetron was 4.5 kW, voltage 550 V and current 21 A. The sample table rotated at 0.5 revolutions per minute, the bias was -60 V.

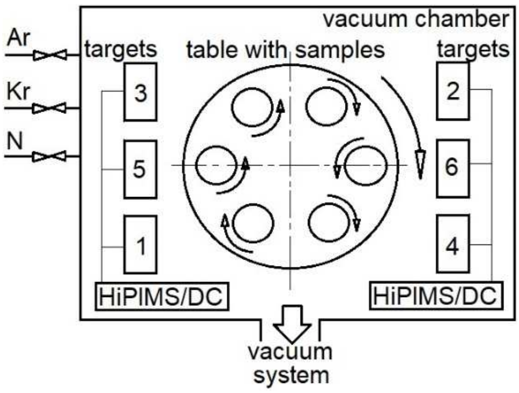


*Figure 2 CC800 HiPIMS device, schematic*

*Figure 3 Samples in the preparation in a vacuum chamber*

The properties were evaluated:

• *Thickness* – using the Kalotest method. The Swiss Calotest device from CSM Instruments was used. The parameters of the test were: the steel ball had a diameter of 15 mm, the grinding time of the calota was 300 s, and the speed of rotation of the ball was 900 revolutions/min. Struers sanding paste with a sanding grain diameter of 0.5 to 1.0 µm was used. A Japanese OLYMPUS - MX51 optical microscope was used to measure the diameters of the skull.

• *Adhesion* – was evaluated using the Mercedes test method according to the VDI 3198 standard. The puncture was made with a diamond Rockwell indenter in the shape of a cone with an angle of 120°, which gripped the top of the indenter. The injection evaluation was performed using a Japanese OLYMPUS - MX51 optical microscope.

• *Hardness and Young's modulus* – They were performed by the indentation method on a Bruker HYSITRON TI 950 TriboIndenter, with a maximum loading force of 10,000 µN. The puncture was performed with a diamond Berkovich indenter in the shape of a triangular pyramid. The maximum loading force was less than 2 N.

• *Friction coefficient* – a Pin-on-disk test was used with a linear path of the counter piece (steel ball with a diameter of 4 mm), length of the path 10 mm, speed of the counter piece 10 mm/s; test duration 7200 s; the counterweight loading force was 5 N.

## 3 Results and discussion

The thickness of the $TiB_2$ coating was measured to be 4.1 µm (Figure 4) (frequency 800 Hz, pulse width 50 µs), which is 100% more than that measured by ZHANG T.F. et al. [18], who measured the thickness of the $TiB_2$ coating approx. 2 µm and 300% more than THORNBERG J. et al. [23] measured a thickness of only 1 µm.

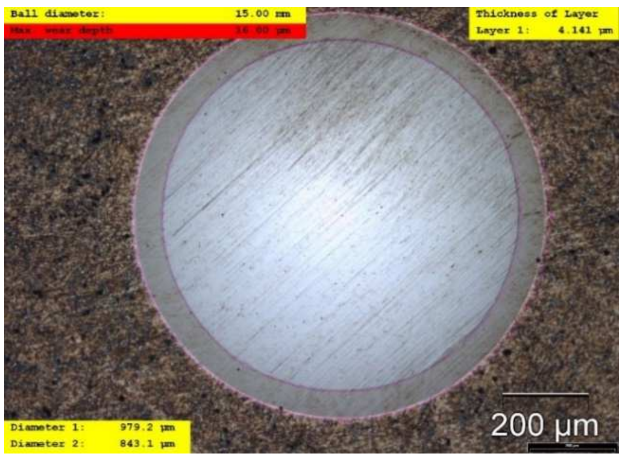


*Figure 2 Calota $TiB_2$ coating deposited at a frequency of 800 Hz and a pulse width of 50*

A view of the surface of the evaluated coating (Figure 5) shows a columnar structure with a primer of grains from 1 µm to 5 µm and gaps (voids) of 1 µm size are visible. The chemical composition of the $TiB_2$ coating is shown in Figure 6.

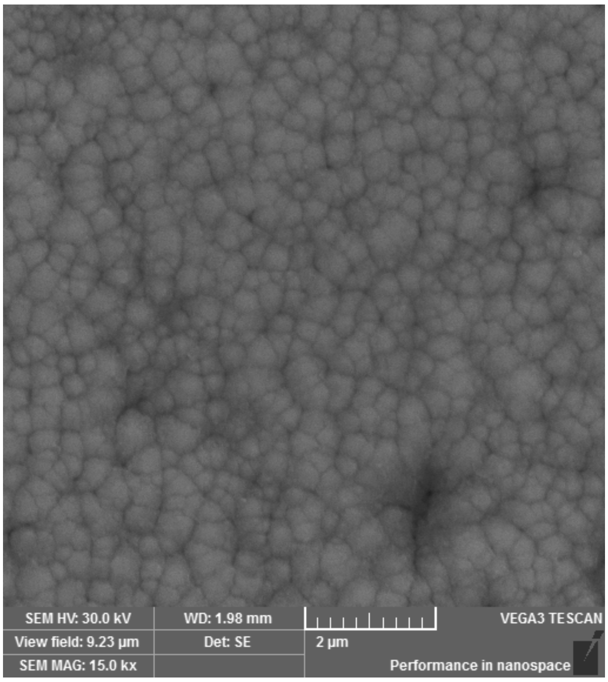


*Figure 3 View of the TiB2 coating surface, SEM*

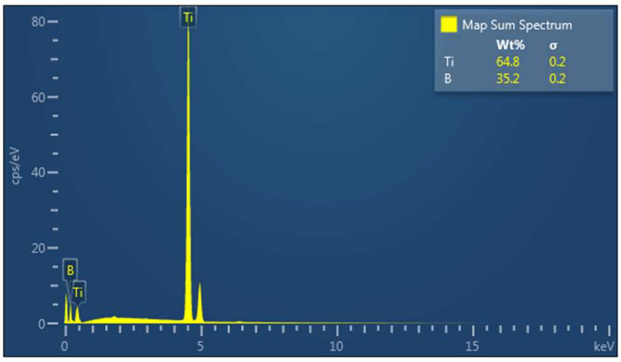


*Figure 4 Chemical composition of TiB2 coating, EDS*

A hardness of 25 GPa was measured, which is 20% less than ZHANG et al. [18], (29 GPa at 300 °C, bias 50 V). Young's modulus was approx. 240 GPa, which is comparable to Nedfors et al. [24] (approx. 480 GPa, 600 Hz and 1000 Hz, bias -60 V) by 40% less. Adhesion evaluated by the Mercedes test according to the VDI scale (Figure 7) reached grade HF 1 (Figure 8).

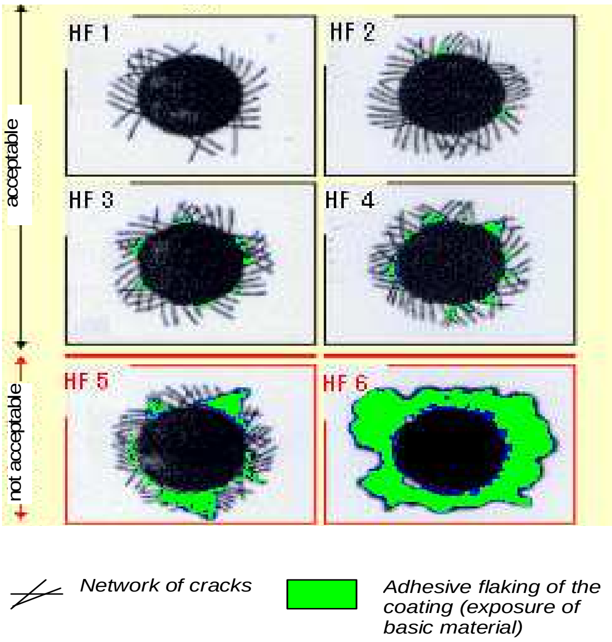


*Figure 7 Mercedes test scale" HF1-HF 4 - satisfactory, HF4-HF6 - unsatisfactory adhesion [25]*

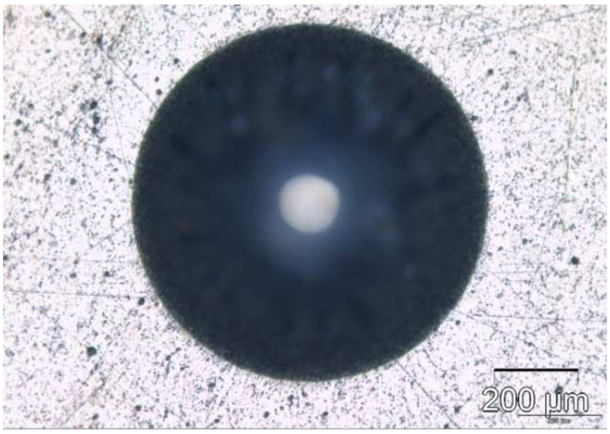


*Figure 8 Adhesion of $TiB_2$ coating deposited at a freqency of 4000 Hz and a pulse length of 50 µs, grade HF1*

The coefficient of friction after 2 hours reached a value of 0.7 (Figure 9), which is standard for the hardness of the evaluated coating of 25 GPa and the hardness of the counter piece (steel ball), because it was worn gradually, which increased the contact area. The rapid wear of the ball occurred after the first tens of seconds, when the coefficient of friction increased sharply to a value of 0.65 and then gradually increased to a value of 0.4. After a period of 5000 s, it rose to a final value of 0.7, which did not change until the end of the test (7200 s).

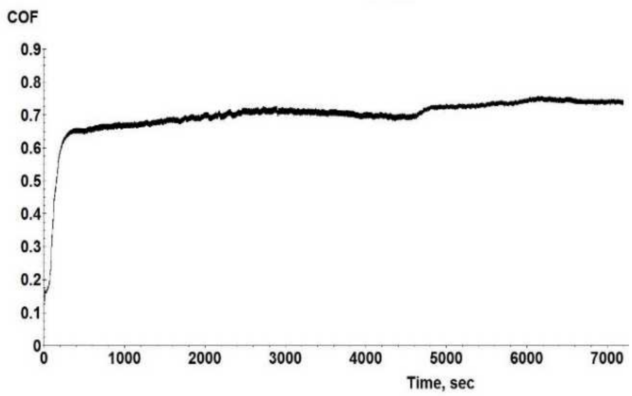


*Figure 9 Dependence of the friction coefficient on time, $TiB_2$ coating deposited at a frequency of 4000 Hz and a pulse width of 50 μs*

## 4 Conclusions

A $TiB_2$ layer was deposited on the steel substrate, where:

- a thickness of 4.1 μm was measured,
- the evaluated coating has a dense columnar structure, which corresponds to a hardness of 25 GPa and a Young's modulus of 240 GPa,
- adhesion reached grade HF1,
- the coefficient of friction was 0.7.

Based on the above, it can be concluded that the evaluated coating is suitable for industrial use for coating cutting tools.

Further research will focus on the influence of technological parameters such as frequency and width of impulse on the thickness, hardness, Young´s modulus and coefficient of friction of the $TiB_2$ coating deposited using progressive HiPIMS method.

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