THE IMPACT OF NEGATIVE BIAS SUBSTRATE TO FRACTURE TOUGHNESS AND HARDNESS OF TiB_2 SPUTTERING COATINGS

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Abstract

Engineering ceramics are commonly used as coating materials in machining steel and in the non-ferrous alloys industry, mainly to increase the durability tools working in different operating conditions. Coatings based on borides, nitrides, or carbides have high hardness and good tribological properties at room and at elevated temperatures. TiB_2 is a unique material characterized by the low affinity to aluminium and its alloys. Such properties justify its successful use in the production of thin anti-wear films dedicated to increasing the durability of tools used in the non-ferrous metal processing industry. This compound is also considered to be superhard materials (hardness up to 30 GPa). Titanium diboride hardness, depending on process parameters and production methods, is in the range of 30 - 40 GPa. Unfortunately, this material has high brittleness, which create the limits of the area of its application.

The paper presents the influence of negative substrate polarization on mechanical properties (HV, E) and fracture toughness (K_{Ic}) of TiB_2 coatings. All coatings were obtained using the magnetron sputtering method on samples of SW7M steel heat treated to hardness 59 HRC. The result of the increase of negative substrate polarization is the increase in hardness and Young modulus. The fracture toughness obtained in the coatings remained at a constant level of 0.6 $MPa \cdot m^{1/2}$. This means that the coatings are very brittle. In the case of polarization U_{BIAS} = -75 V, the authors achieved a 3 times higher brittleness index K_{Ic} and an increase of hardness to 36 GPa simultaneously.

Keywords: TiB_2 coatings, fracture toughness, magnetron sputtering, PVD

1. INTRODUCTION

The non-ferrous industry is still developing, but the main fields of use of aluminium, magnesium, and titanium alloys remain the aviation and automotive industries. These trends are resulted from the European directive and explore the possibility of decreasing fuel consumption [1][2]. Light alloys, from which car and aircraft components are manufactured, are characterized by many attractive properties such as low density, a high strength to weight ratio, good formability, etc. [3-5]. In order to effectively machining non-ferrous alloys the use of special tools is required. Only then a good quality finished surface is possible to obtain. The dominant technical area for the design of material solutions in the tools industry is surface engineering [5][6]. The coating dedicated to increasing the durability of drills and tools for machining light alloys is titanium diboride because of its high hardness, high melting point, excellent wear resistance, and oxidation resistance [7-9], but the main TiB_2 advantage is the low affinity to aluminium. Despite this, titanium diboride is not a popular coating material, mainly due to its high brittleness. However, the fracture toughness may be improved by changing the parameters of the technological process [9].

2. METHODOLOGY

2.1. Experimental procedure

TiB_2 coatings were sputter-deposited to a nominal thickness of about 1 μm by the DC magnetron sputtering technique from a pure TiB_2 sintered target (purity 99.5 %) with a diameter of 100 mm and a thickness of 8 mm, adhesively bonded on a copper washer. TiB_2 coatings were deposited on polished high speed steel HS6-5-2 samples of the following composition (wt. %): 0.8 C, 4.0 Cr, 0.4 Ni, 5.0 Mo, 6.5 W, 2.0 V, which was provided in the form of discs of 25.4 mm in diameter and 6 mm in height. The steel samples were heat treated to a hardness of 59 HRC. Before deposition, the chamber was evacuated to 3×10^{-3} Pa and the samples were heated up to 300 °C. The working pressure during deposition was 0.5 Pa and the distance from the samples to the plasma source was 150 mm. During the process, the temperature was constantly monitored using a pyrometric temperature measurement system. The TiB_2 deposition was carried out under the input power of 1000 W for 60 minutes. The the negative bias of substrate was varied in the range from 0V (float voltage) to -75 V.

2.2. Surface characterization

2.2.1. Chemical and phase composition

The chemical composition of materials that contain light elements must be investigated using a special method. One of the methods used to study of this type of materials was wavelength dispersive X-ray spectroscopy (WDS). The chemical composition of obtained coatings was analysed using a JXA-8230 device produced by the JEOL Company. Additionally, in order to confirm the formation of the titanium diboride phase, the phase composition and crystallographic orientation of the thin films were analysed by applying X-ray diffraction using Co Kα radiation with an Fe filter in XRD-7 diffractometer produced by the Seifert-FPM company.

2.2.2. Microstructure and mechanical properties

The analysis of the microstructure on brittle fractures was carried out using a scanning electron microscope (SEM) Hitachi SU-70. The results of the mechanical properties of TiB_2 coatings, including hardness and Young modulus, were carried out using a nanohardness tester from the CSM company with a Berkovich indenter. The maximum load adopted was 8 mN, which corresponded to a maximum penetration depth of about 100 nm. Maximum penetration depth was determined by the thickness of investigated coatings, because the depth of indentation must be less than 10 % of total thickness of the coating.

2.2.3. Fracture toughness

Titanium diboride is a ceramic material with a very high hardness and a high brittleness. Analysis of fracture toughness of thin films and brittle materials is limited to only few methods. One of the dedicated methods is a penetration method using a nanohardness tester with Berkovich indenter. Because indentations were very small, a scanning electron microscope (SEM) was used to measure variables to determine the brittleness index. The applied load of the indenter was selected experimentally and fixed at 200 mN. For each coating 20 indentations were made. **Figure 1** shows the method of measurement of crack length and distance between the corner to centre indentation. The measured value was used to determine fracture toughness in accordance with Equation 1.

$$K_{Ic} = Xv \; x \left(\frac{a}{l}\right)^{1/2} x \left(\frac{E}{H}\right)^{2/3} x \; \frac{P}{c^{3/2}}, \tag{1}$$

where, *Xv* - constant equal 0.016; *a* - average crack length; *l* - distance between corner to center of indentation; *E* - Young modulus; *H* - hardness; *P* - applied load; *c* - amounting a and l.

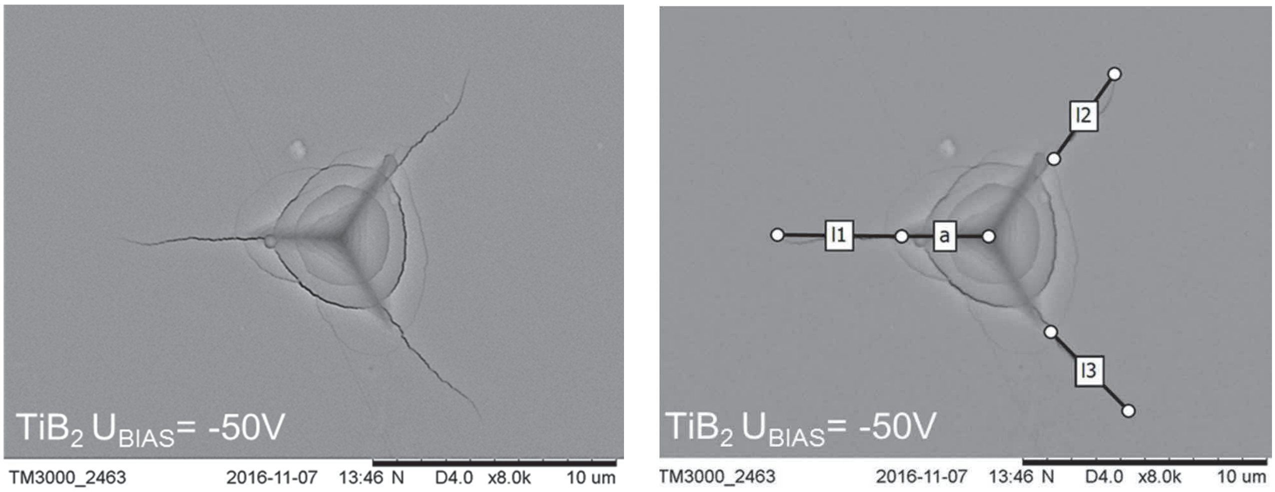

Figure 1 The method of measurement of fracture toughness of TiB_2 coating

2.2.4. Coefficient of friction

Investigations of the coefficient of friction were carried out using the scratch method on a Revetester. Due to the fact that TiB_2 is dedicated as a coating for drills and tools for machining non-ferrous alloys, 3 different non-ferrous alloys were selected as counterparts: PA6 aluminium alloy, Mo58 brass, and Ti6Al4V titanium alloy. Additionally, bearing steel 100CrMo73 was chosen.

3. RESULTS

Electron probe microanalysis using WDS spectrometry is the dedicated method for the investigation light elements. It is the preferred technique because of its higher spectral resolution in comparison to EDS spectrometry. Coatings have a following composition (at. %): 67 B, 33 Ti. The B / Ti atomic ratio is similar to the ratio of stoichiometric TiB_2 composition. The analysis of phase composition confirmed that the coatings have a hexagonal TiB_2 structure. XRD has also revealed that the films show a strong crystallographic texture with (0001) planes of most crystallites being parallel to the film surface.

The hardness of the obtained TiB_2 coatings is about 30 GPa (**Table 1**). This means that the films can be classified as super-hard materials. The results can be divided into two groups with similar properties: float potential and -25 V bias have a hardness of 30 GPa and modulus of 400 GPa, and -50 V bias and -75 V bias have a hardness of about 35 GPa but with different modulus. The H^3 / E^2 index (plasticity deformation index) increased with increasing negative bias polarization. This means that, in the case of the coating with a high value of index, crack propagation will be more difficult unlike coating with a lower H^3 / E^2 value. This difference was observed in the microstructure of brittle fractures of TiB_2 coatings (**Figures 2a, b**).

All coatings have very fine columnar microstructure. They are characterized by good adhesion to the substrate. In the case of TiB_2 coating obtained using float potential, the fragile nature of the fracture was observed. The use of polarization -75 V resulted in creating a significant change in crack creation. In **Figure 2b**, white dashed lines show the differences in the direction of crack propagations.

The results of fracture toughness analysis of TiB_2 coatings are shown in **Table 1**. Coatings from the range of 0 to -50 V have a similar index of brittleness. The authors observed that the distance between corner to centre of indentation remains constant.

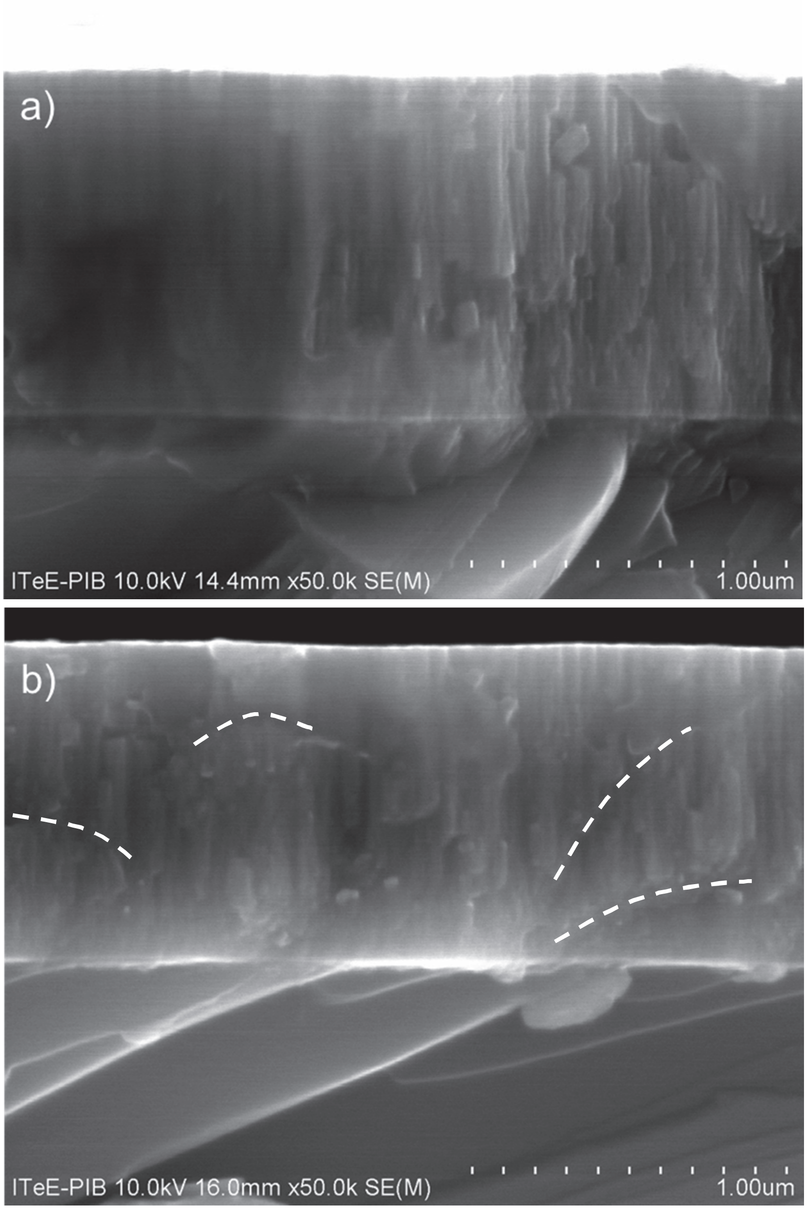

Figure 2 The microstructure of a brittle fracture of TiB_2 coating obtained with different bias polarization: a) 0 V (float polarization), b) -75 V bias

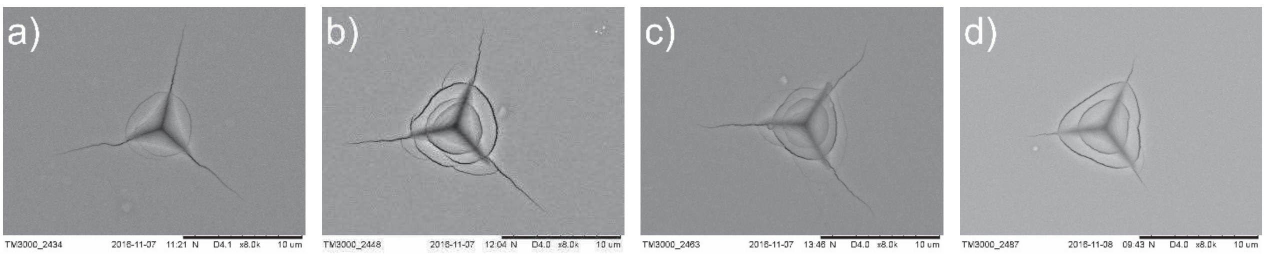

Figure 3 Scanning electron micrographs of indentation on TiB_2 coatings: a) 0 V (float polarization), b) -25 V bias, c) -50 V bias, d) -75 V bias

The thin film obtained using -75 V bias polarization has a 3 times higher K_{Ic} index. There was no significant difference in the distance between corner to centre of indentation in comparison with other layers, but crack length is smaller (**Figure 3d**). At the same time, the hardness of TiB_2 U_{BIAS}= -75 V coating is higher.

Table 1 Results of measurement of fracture toughness, hardness and Young modulus of TiB_2 coatings

Bias substrate (V)	Hardness (GPa)	Modulus (GPa)	H^3 / E^2	*a* (μm)	*l* (μm)	*c* (μm)	K_{Ic} (MPa·$m^{1/2}$)
0 (float voltage)	29.5 ± 0.5	395 ± 10	0.17	5.25	3.36	8.61	0.56 ± 0.05
-25	31.5 ± 0.5	400 ± 10	0.19	5.36	3.96	9.32	0.52 ± 0.06
-50	34.5 ± 0.5	405 ± 10	0.25	4.33	3.80	8.13	0.66 ± 0.08
-75	36.5 ± 0.5	430 ± 10	0.27	1.73	3.96	5.69	1.81 ± 0.30

The results of measurements of the coefficient of friction (**Figure 4**) show that all coatings have a much smaller coefficient of friction in comparison to the uncoated sample.

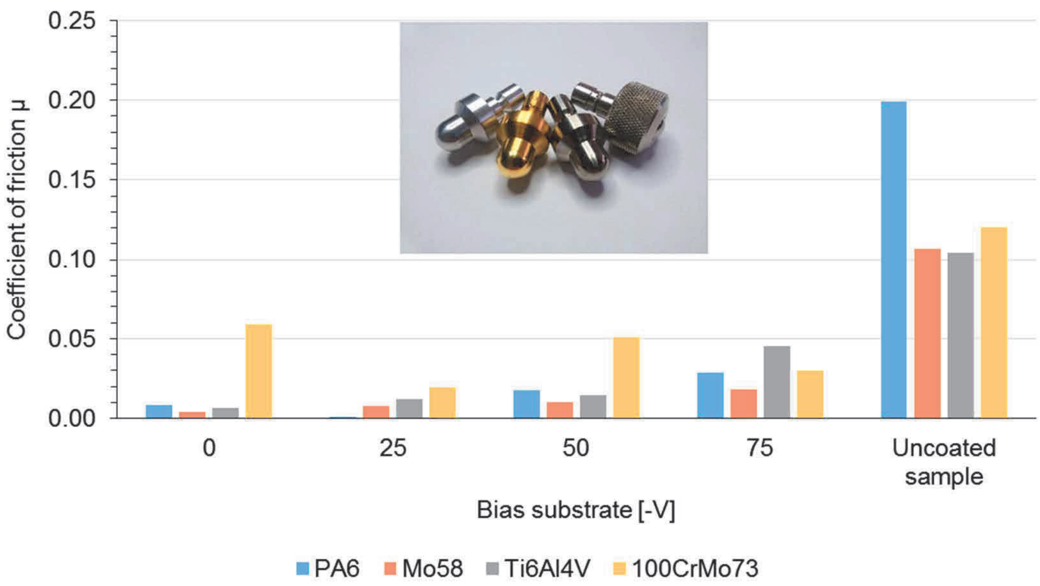

Figure 4 The coefficient of friction of the TiB_2 coating in contact with chosen non-ferrous alloys and bearing steel

The low coefficient of friction can be due to the low affinity of titanium diboride to non-ferrous alloys. The authors also observed that coatings had a decreased coefficient of friction in contact with steel. This means that TiB_2 coating may be both a good surface material for machining non-ferrous alloy but also for machining steel.

4. CONCLUSION

Titanium diboride is a recommended coating material for machining non-ferrous alloys, mainly aluminium and aluminium alloys. The major problem for the application of this compound is its very high brittleness. One of the methods of improving the fracture toughness is changing the parameters of the technological process. The use of substrate polarization U_{BIAS}= -75 V resulted in a three-fold increase of the K_{Ic} fracture index. At the same time, an increase in hardness of about 5 GPa was observed. Variation in material's properties (hardness, Young modulus, fracture toughness) were caused by the differences in microstructure. In the case of U_{BIAS} = -75 V, the authors observed many areas where the direction of crack creation was changing Unfortunately, values of the index of fracture toughness of about 1.8 MPa·m$^{1/2}$ are suitable for brittle materials. However, the level of this value is similar to the K_{Ic} values of commercially used coatings on the tools for machining the following: TiN (1.1 - 2.5 MPa·m$^{1/2}$), CrN (1.5 - 3.5 MPa·m$^{1/2}$) [10], and TiN / CrN (1.3 - 2.0 MPa·m$^{1/2}$) [11].

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