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THE IMPACT OF MAGNETRON SOURCE POWER ON MECHANICAL PROPERTIES AND PHASE COMPOSITION OF TiB_2 COATINGS

Key words

Surface engineering, TiB_2 coating, magnetron sputtering, nanohardness, fracture toughness, super hard coatings.

Abstract

PVD coatings are widely used in the tool industry. They use hard materials, such as carbides, borides, and nitrides of metallic elements, because these materials have good wear resistance and stability at elevated temperatures. Titanium diboride-TiB_2 is a very interesting compound used for coatings in the tool industry. The results of the analysis of research directions in the field of coatings and layers with special operating properties (high hardness, good thermal and electrical conductivity, and good corrosion resistance) indicate that coatings and layers based on TiB_2 are one of the most effective materials that improve tool durability.

The paper presents the mechanical properties of TiB_2 coatings obtained using the magnetron sputtering method in the function of sputtering process power of TiB_2 target. The paper includes hardness and Young's modulus

measurements performed with nanoindentation method, and phase composition analysis, and the evaluation of the microstructure and topography using a Hitachi TM3000 scanning electron microscope.

Introduction

Surface engineering enables the efficient development of many technical areas [1], [2]. The tool industry is one of the dominant areas where surface engineering is the basis for the design of material solutions. Hard and wear-resistance coatings dedicated to cutting tools substantially increase the life of elements. TiB_2 is indicated in the literature as a ceramic of high melting point (3226°C), high hardness (~30 GPa), good thermal and electrical conductivity, and good corrosion resistance [3], [4], which makes it a very interesting chemical compound in many applications. The main disadvantage of this compound is high brittleness and poor adhesion of the coating to the substrate, which limits the use of the TiB_2 coating for tribological applications [5]. Thin titanium diboride coatings are obtained using PVD or CVD methods. However, the greatest potential is found with TiB_2 coatings produced by PVD because this method can used much lower temperatures of the process, which is important in the quality of tools that have been heat-treated.

The authors analysed the impact of the magnetron sputtering source power equipped with a target TiB_2 on the mechanical properties and phase composition obtained coatings. We also analysed elasticity-plasticity parameters, adhesion, and resistance to brittle fracture.

1. Experimental

TiB_2 coatings were deposited using the DC magnetron sputtering method with TiB_2 sintered target with a diameter of 100 mm and a thickness of 8 mm, adhesively bonded on a copper washer. The processes were carried out under inert gas (Ar), which aimed to control the pressure. The samples of titanium alloy Ti6Al4V were subjected to one-sided, mechanical grinding and polishing. In addition, they were skimmed in chemical washing stand UMO-50-1000 with ultrasonic bath using TRI solvent and ethyl alcohol and dried thoroughly.

Each process of deposition TiB_2 coating consisted of the following three stages:

- Heating the substrate material to a temperature of 250°C,
- Ion etching using an arc plasma source with a titanium cathode, and
- Deposition TiB_2 coatings.

The parameters of the technological process have been collated in Table 1.

Table 1. The parameters of TiB_2 deposition process

Stage 1: Heating

Heater current [A]	Temperature [°C]	Time [min]
150	250	30

Stage 2: Ion etching

I_{ARC} [A]	U_{bias} [V]	p [mbar]	q_{Ar} [ml/min]	Time [min]
60	–900	$5*10^{-4}$	1.5	15

Stage 3: Coating deposition

Magnetron power [W]	U_{bias} [V]	U_{mag} [V]	I_{mag} [A]	p [mbar]	q_{Ar} [ml/min]	Time [min]	Deposition rate [nm/min]	Power density [W/cm^2]
200	–100	403	0.49	$5*10^{-3}$	15	60	3	2.7
400	–100	434	0.92	$5*10^{-3}$	15	60	6	5.4
600	–100	496	1.20	$5*10^{-3}$	15	60	10	8
800	–100	490	1.65	$5*10^{-3}$	15	60	10	10.7
1000	–100	482	2.00	$5*10^{-3}$	15	60	21	13.4

The samples were installed in a disc holder with a diameter of 100 mm. 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.

2. Results

2.1. Mechanical properties

The analysis of mechanical properties of TiB_2 coatings, including hardness, Young modulus, and elasticity-plasticity properties, were carried out using nanohardness tester CSM with a Berkovich diamond indenter. The maximum load adopted was 2 mN, which corresponding to a maximum depth of about 50 nm. Load selection was dictated by the thickness of the obtained coatings in order to ensure that the depth of indentation is less than 10% of all coating thicknesses. The results are shown in Table 2.

Table 2. Results hardness and Young modulus measurement

Magnetron power [W]	Hardness [GPa]	Modulus [GPa]	H^3/E^2	Thickness [µm]
200	21.6±1.0	253±12	0.166	0.20
400	23.8±0.4	255±54	0.213	0.35
600	27.2±1.0	297±45	0.223	0.60
800	30.2±2.0	326±44	0.254	0.60
1000	41.8±1.0	481±91	0.320	1.25

As can be seen (Figure 1), the hardness of TiB_2 coating increases with increasing magnetron source power. Between two extreme process, i.e. 200 W and 1000 W, hardness was increase by about 2000 HV. At the same time, the increase in the elastic modulus as a function of the magnetron source power was observed.

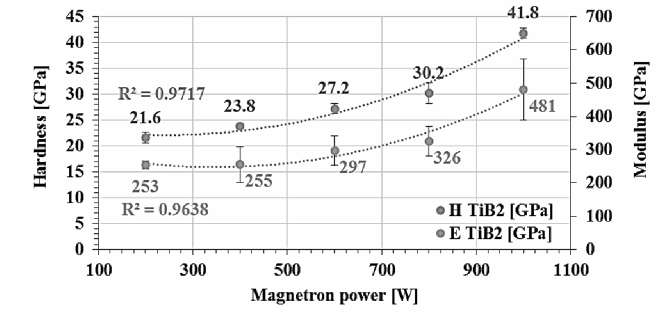

Fig. 1. Hardness and modulus TiB_2 coating obtained with different magnetron power

The high hardness of the coating may suggest a high brittleness. The authors performed the analysis of the plasticity deformation index H^3/E^2 with reference to the literature [6], [7], [8].

A high value of the H^3/E^2 index means a low susceptibility to plastic deformation with the result that in the material will be performed faster crack propagation leading to the degradation of the coating. The results can suggest that increasing the magnetron source power causes increased brittleness of the obtained coatings.

2.2. Chemical and phase composition

X-ray microanalysis was carried out using a microanalyser JXA-8230 JEOL with a WDS spectrometer. The WDS spectrometry is one of the few methods allowing the quantitative analysis of the chemical composition of coatings with the participation of light elements, including boron. The stoichiometric TiB_2 compound should contain about 68% titanium and 32% boron. The chemical composition of obtained coatings is shown in Table 3.

Table 3. Chemical composition TiB_2 coatings

Magnetron power [W]	Weight %				Atomic %
	B	Ti	Al	V	B/Ti
200	21	73	3	3	1.26
400	28	68	2	2	1.77
600	30	68	1	1	1.96
800	31	68	0.5	0.5	2.09
1000	31	68	0.5	0.5	2.09

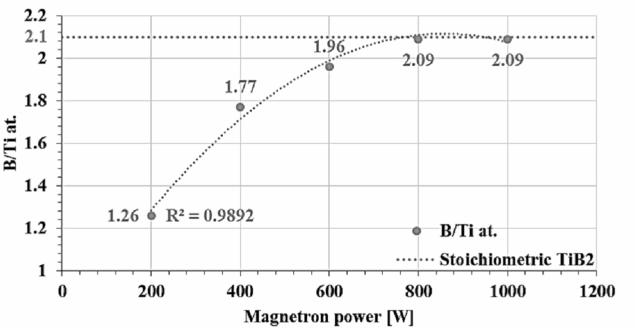

Fig. 2. The B/Ti weight % ratio TiB_2 coating obtained with different magnetron power

The results of the chemical composition show that the increase of magnetron source power created excellent conditions to enable the formation of stoichiometric TiB_2. As shown in Figure 2, the value of the ratio B/Ti for the low magnetron source power strongly deviate from the stoichiometric TiB_2.

The research on phase structure was carried out on a D8 DISCOVER Bruker diffractometer. The analysis of the literature [3], [9] shows that the main growth directions of the hard TiB_2 phase is [001] and [101].

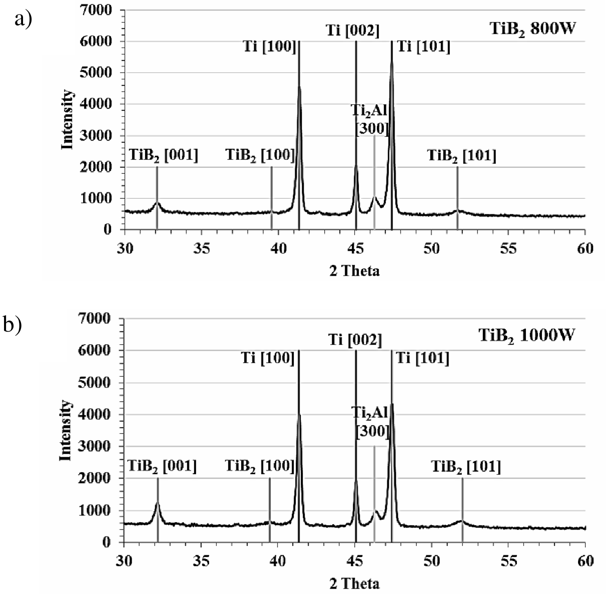

Fig. 3. The diffractograms TiB_2 coating obtained with different magnetron power: a) 800 W; b) 1000 W

Diffraction studies showed the presence of a TiB_2 phase in the coating with the growth directions of [001] [100] [101] (Figure 3). The intensity of the phase signal is considerably higher for the coating evaporated using a 1000 W magnetron source power. This may indicate the presence of a more stoichiometric and hard TiB_2 phase in the coating structure compared to the lower power.

2.3. Fracture toughness

In order to obtain a clear answer providing the brittleness of the obtained coatings, the authors conducted tests of fracture toughness. Indentations were made using a Vickers hardness tester FV-7 Future Tech, and then a scanning electron microscope (Hitachi TM3000) was used to measure crack length from the corners, which was used to determine fracture toughness in accordance with Equation 1 [10] as follows:

$$K_{Ic} = \frac{0.035 HV\sqrt{a}}{\Phi\left[\frac{HV}{E\Phi}\right]^{0.4}\sqrt{l/a}} \tag{1}$$

where

HV – hardness,
E – Young modulus,
a – half of the diagonal,
l – average length of the cracks,
Φ – constant be equal 3.

The force applied to the indenter was selected experimentally and amounted to 0.5 N. For each coating, three indentations were made in order to develop statistics. A micrograph of the coating after indentation is shown in Figure 4.

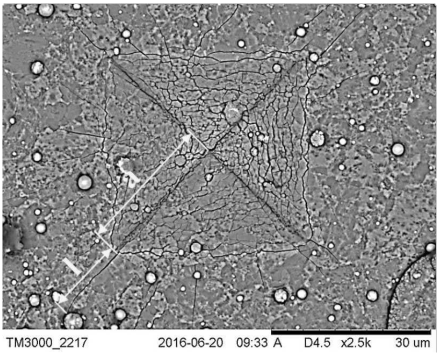

Fig. 4. The micrograph of Vickers indentation on the TiB_2 coating obtained used 1000 W magnetron source power

Based on the literature [11], the ratio of the fracture toughness to the hardness of the TiB_2 coating was calculated. The results are shown in Table 4.

Table 4. Results of fracture toughness measurements

Magnetron power [W]	K_{Ic}/HV
200	0.74
400	0.65
600	0.43
800	0.41
1000	0.39

The authors observed that the ratio K_{Ic}/HV is smallest for the coating that is characterized by the greatest hardness. In addition, this index shows the brittleness of the tested coatings.

Discussion and conclusions

The magnetron sputtering method is a method that allows the effective TiB_2 coating deposition. The authors found that a magnetron sputtering power in the range of 200–1000 W has a significant effect on the chemical composition and mechanical properties of TiB_2 coating. Increasing the magnetron source power can cause a significant increase the stoichiometry of the TiB_2 phase. In the power range of 600–1000 W, deposited TiB_2 coatings were fully stoichiometric in composition. The increase in amount of TiB_2 phase can cause an increase in the brittleness of the coating, as evidenced by the change fragility index K_{Ic}/HV. The results of phase composition showed that increasing the power range of 800–1000 W causes an increase in the intensity of the diffraction peaks for the TiB_2 phase for crystal plane [001] and about 10% for planes [100] and [101].

The obtained results for the TiB_2 coating deposited using magnetron sputtering method raises the following conclusions:

- The magnetron source power has a large influence on the stoichiometric composition and mechanical properties of TiB_2 coatings.
- We can control the brittleness and hardness of the obtained TiB_2 coatings by changing the magnetron source power. Adjustment of these parameters is dependent on the application area. In the case of components exposed to dynamic loads, high brittleness is avoided, as opposed to coatings exposed to static loads.

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Wpływ mocy rozpylania magnetronowego na właściwości oraz skład fazowy powłok TiB_2

Słowa kluczowe

Inżynieria powierzchni, powłoki TiB_2, rozpylanie magnetronowe, nanotwardość, odporność na pękanie, powłoki supertwarde.

Streszczenie

Powłoki PVD znajdują powszechne zastosowanie w przemyśle narzędziowym. Do ich wytwarzania stosuje się twarde związki takie jak: węgliki, borki, azotki metali, które charakteryzują się dobrą odpornością tribologiczną oraz stabilnością w podwyższonej temperaturze. Dwuborek tytanu – TiB_2 jest bardzo interesującym materiałem powłokowym, cieszącym się dużym zainteresowaniem w przemyśle narzędziowym. Analiza literatury w zakresie powłok funkcjonalnych (powłok o dużej twardości, dobrym przewodnictwie cieplnym i elektrycznym, dobrej odporności korozyjnej) wskazuje TiB_2 jako jeden z najbardziej efektywnych materiałów zwiększających trwałość narzędzi przeznaczonych do obróbki powierzchniowej stopów metali nieżelaznych.

W artykule przedstawiono właściwości mechaniczne powłok TiB_2 otrzymanych z wykorzystaniem metody rozpylania magnetronowego w funkcji mocy rozpylania targetu TiB_2. Artykuł zawiera pomiary twardości oraz modułu Younga, analizę struktury fazowej, badania mikrostruktury oraz badania odporności na pękanie cienkich powłok.