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GROWTH AND STRUCTURE OF SPUTTERED TIB_2 THIN FILMS

*Deposition processes for thin hard layers are known for several decades, but still new inventions of layer and their combinations are appearing. The latest research shows the perspective usage of coated materials of the types **Tffiz, TiAl-TiB, TiB-Ti,** which have the special properties, very interesting for some technological applications. The study of the first basic properties ofTiB_2 research and detailed study of the texture and microhardness are presented in this paper. TiB_2 coatings were deposited on the Si (001) samples and mirror polished stainless steel substrates by DC magnetron sputtering method.*

Introduction. Titanium diboride (TiB_2) is a ceramic compound with hexagonal structure in which the boron atoms form a covalently bonded network within metallic Ti matrix (Fig. l). Specify resistance (6.4-9. IQjicm) is low, temperature coefficient of dilatability is (4,6*10-6 K-l). These properties, chemical stability, high heat resistance of the ceramic materials with high hardness (30 GPa), high wear and corrosion resistance fulfil high demands on modern progressive cutting tools. Prepared diboride coatings show a high potential for tribological applications. TiB_2 coatings can be prepared by different CVD and PVD methods. Magnetron sputtering in inert atmosphere appears to be the most suitable method, due to low deposition temperature and relatively high deposition rate. This deposition process also allows producing coatings on substrates with complicated geometry. Another advantage is absence of toxic and explosion gases during the deposition.

Experimental works. TiB_2 coatings were deposited by DC magnetron sputtering from stoichiometric TiB_2 target with diameter 120 mm and thickness 6 mm in Ar atmosphere. As substrate materials were used Si (001) samples and mirrors polished stainless steel.

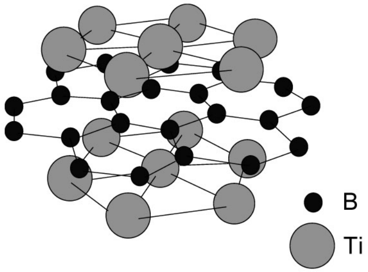

Fig. 1. Illustration of the hexagonal TiB_2 crystal structure

Before deposition, the substrates were cleaned in an ultrasonically cleaning bath. The substrates were positioned stationary 5 cm from target. As a process gas was used an argon with purity of 99,994%. Starting vacuum was 2 x 10-3 Pa, the substrates were cleaned *in-situ* by argon-ion etched for 15 min using substrate bias voltage -500 V and an Ar pressure of 2 Pa. The deposition time was 15 min. The pressure was changing from deposition to deposition in range 0.2 – 1 Pa by the 0.2 Pa step. Magnetron current was 1.4 A and corresponding magnetron voltage had been changing in range 350 V – 390 V. Preferred orientation and phase composition were observed by X – Ray Diffraction (XRD) analysis in Bragg – Brentano geometry, using HZG4 equipment with CuKa monochromator (wave length – 0,015418 nm). The Auger Electron Spectrometry (AES) was applied for control of coating's stoichiometry. The roughness profile was analysed by Atomic Force Microscope (AFM) – Solver P47. Coatings morphology and thickness were examined by SEM (JEOL) microscopy. Stainless steel substrates were used for Vicker's microhardness measurement (INDENTEC) and adhesion was measurement by the Scratch Tester.

Results and discussion. AES analysis indicated Ti/B ratio approximately 1/2. Presence of carbon and oxygen was also observed. AES determined this composition of the surface layers of a sample – 56,2 % Boron, 33,1 % Titanium, 4,9 % Carbon and 5,7 % Oxygen (Fig. 2). It is an assumption that coatings contamination by oxygen and carbon resulted into too high initial pressure and in attendance of particles from oil rotation and diffusion pumps.

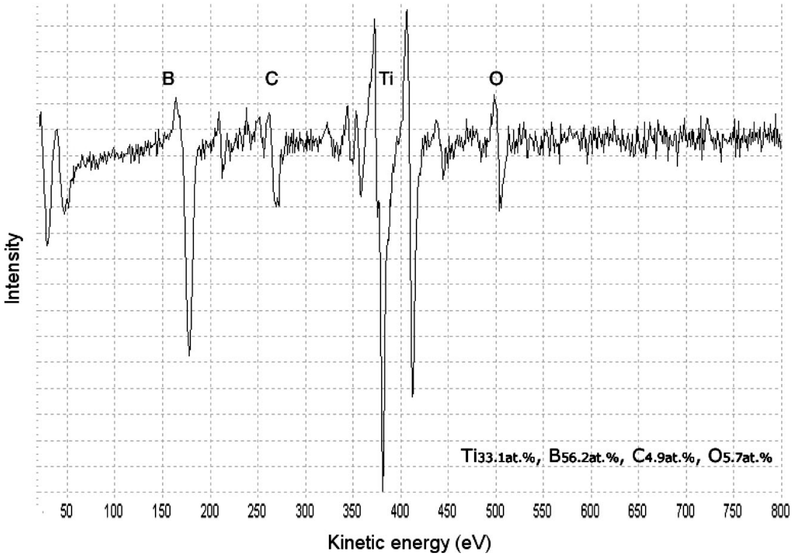

Fig. 2. AES spectrum of TiB_2 coating deposited at 0.2 Pa

Dependence of the surface roughness on the deposition pressure was observed by AFM method. Mean roughness values R_a and maximal roughness R_{max} values are showed in the Tab. 1.

Tab. 1

Dependence of roughness R_{fl} andR_{max} on the working Ar pressure

Pressure [Pa]	0.2	0.6	1
R_a[nm]	0,2	0,3	1,2
R_{max}[nm]	1,2	4	12

It can be seen on the Fig. 3. that surface roughness of TiB_2 layers prepared by the various deposition pressures had increasing tendency together with increase of working pressure.

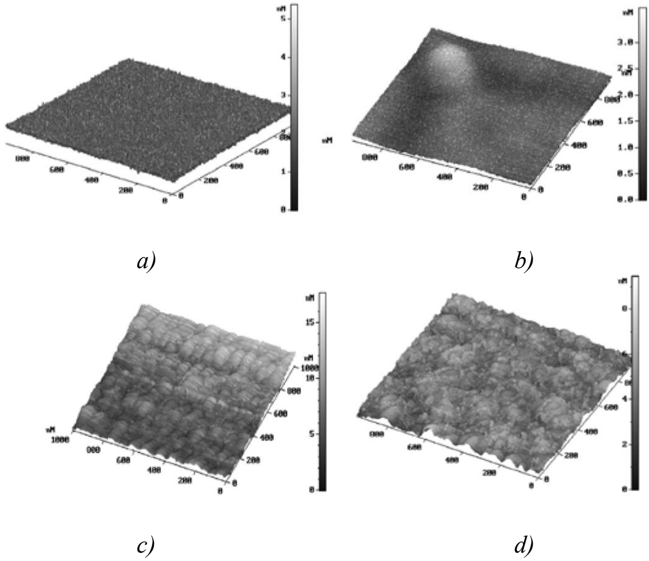

Fig. 3. AFM analysis of TiB_2 surface: deposition Ar pressure a) 0,2 Pa; b) 0,6 Pa; c) 0,8 Pa; d) lPa

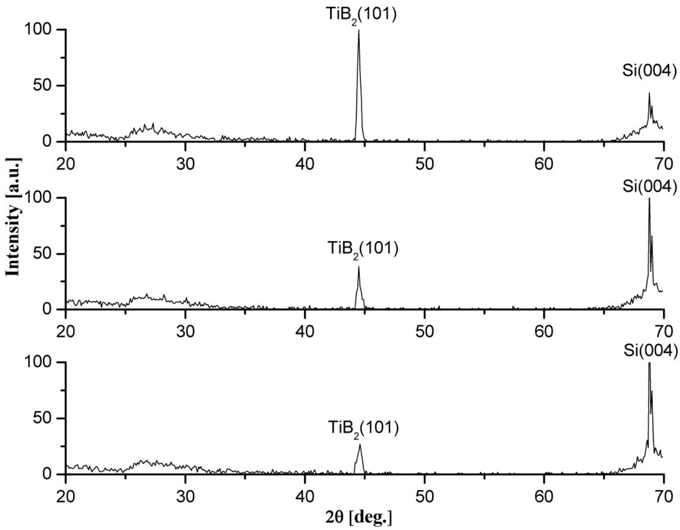

Fig. 4. TypicalXRDpatterns of TiB2 coatings deposited on the Si(OOl) substrates at the a) 0,2Pa; b) 0,6 Pa c) Ipa

A texture of TiB_2 coatings was observed, as is shown in XRD patterns in Fig. 4. The only sharp diffraction peak except of the Si (004) substrate peak, can be attributed to (101) diffraction of TiB_2 phase in 20 = 44.5°.

Typical SEM image of fractured coating format deposited on silicon substrates is shown in Fig.5. The coatings thickness was 2, 5 um. The microhardness measurements exhibit microhardness of TiB_2 coatings higher than 5000 $HV_{0;0}$5 and sufficient adhesion for all deposition samples. All deposited coatings showed a metallic grey and brilliant mirror surface.

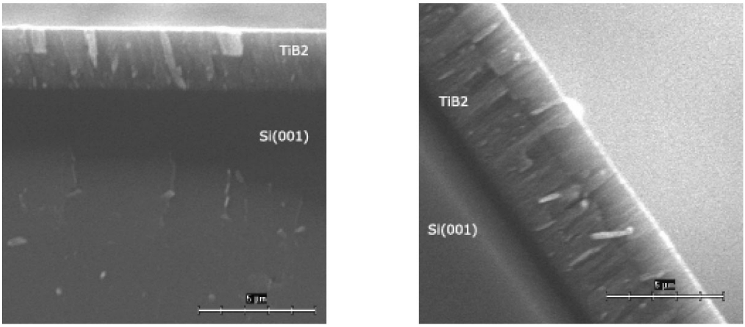

Fig. 5. Cross-sectional SEM images ofTiB_2 coatings deposited on the Si (001) substrates

Conclusion. This interesting new-coated material on the TiB_2 base is the object of our research project with the title "Research of the properties of new super-hard coatings on base TiB_2 on the cutting tools". Within this work, the possibility of DC magnetron sputtering deposition of the TiB_2 coatings has been shown. TiB_2 coatings microhardness was higher than 5000 $HV_{0>}$os with stable reproducibly . The roughness (R_a) changed with deposition pressure. A significant texture was observed. With respect to the microhardness and the surface roughness, the best TiB_2 coatings were reached at working pressure of 0.2 Pa. It can be concluded that TiB_2 coatings prepared by DC magnetron sputtering exhibit the high microhardness and small roughness. Therefore they are very promising for practical applications. The presented study is still in progress.

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