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# DEVELOPMENT OF TITANIUM DIBORIDE COATINGS DEPOSITED BY DC MAGNETRON SPUTTERING

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## 1. Introduction

The proper appliance of protective coatings on cutting and forming tools, seals, gears, bearings and other tribological components can extend component lifetimes. Protective coatings with high hardness provide better wear resistance of coated steel tools and hard carbide against abrasion at high contact pressures. The thin hard coatings and their favourable properties are well known for several decades, but still are coming new inventions of layers and their combinations. Although it appears, that improvement possibilities of the coatings properties have been depleted is not true. The latest research focuses on the perspective usage the coated materials of the types $TiB_2$, *TiAl-TiB, TiB-Ti,* which fulfil the special requirements on the modern progressive cutting tools and on the tribological applications [4].

*Titanium diboride* ($TiB_2$) is ceramic compound with hexagonal structure in which boron atoms form a covalently bonded network within metallic Ti matrix. $TiB_2$ is well known for its high hardness (reported hardness values of up to 6700 HV), high chemical stability at higher temperature, wear and corrosion resistance and good electric conductivity. Deposition of $TiB_2$ based coatings is usually carried out by PVD or CVD techniques. Magnetron sputtering appears to be the most suitable, because deposition temperature can be reached with relatively high deposition speed. However, application of $TiB_2$ coatings is complicated because of poor coatings adhesion caused by high residual stress level. This problem can be solved with substrate heating and

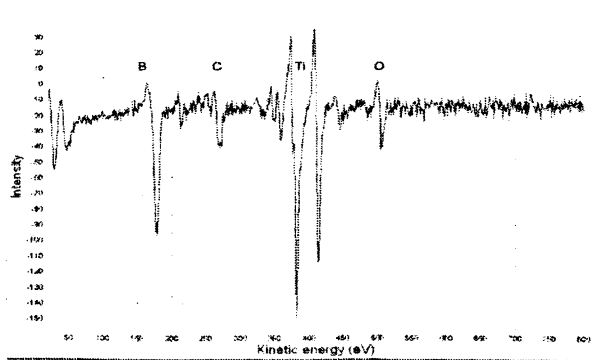
## 2. Experimental works

As substrate materials were used Si (001) samples and mirror polished stainless steel. Before deposition, the substrates were cleaned in an ultrasonically cleaning bath. The coatings were deposited by usage of DC magnetron sputtering. Used $TiB_2$ target has diameter of 40 mm and thickness 6 mm. The substrates are 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 ware observed by X – Ray Diffraction (XRD) analysis in Bragg – Brentano geometry, using HZG4 equipment with CuK$\alpha$ monochromator (wave length – 0,015418 nm). Auger Electron Spectrometry (AES) was applied for control of coating's stoichiometry. Surface quality and roughness were analysed by Atomic Force Microscope (AFM) – Solver P47. Coatings morphology and thickness were examined by SEM (JEOL) microscopy. Parameters of sputtering process were optimised onto Si (001) substrates. Stainless steel substrates were used for microhardness measurement (INDENTEC).

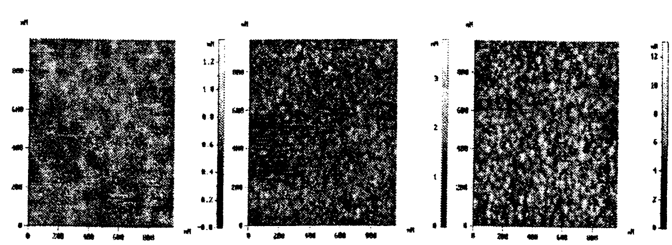
### 3. Results and discussion

Compositional depth profile was determined by Auger Electron Spectrometry analysis, which indicated Ti/B ratio approximately 1/2. It was observed the presence



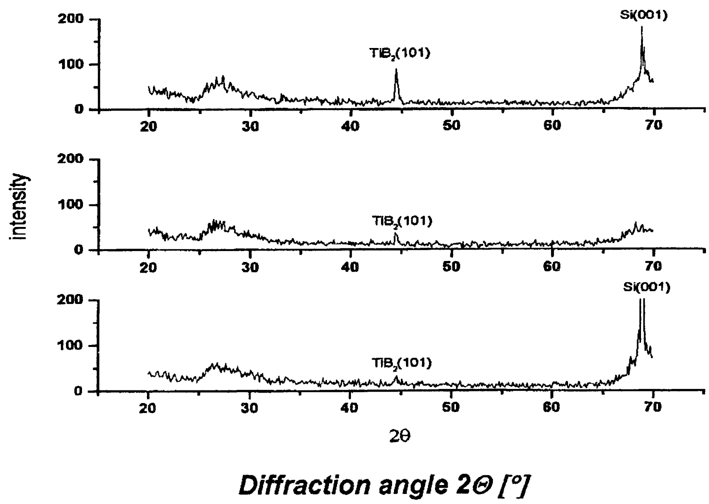

A.) Auger electron spectrum of TiB2 surface layer of a sample in Ar pressure 0.2 Pa

of 56,2% Boron, 33,1% Titanium, 4,9% Carbon and 5,7% Oxygen (Figure A). That is an assumption that coatings contamination by oxygen and carbon is resulted of too high initial pressure and in attendance of particles from oil rotation and diffusion pumps.



B.) AFM analysis of $TiB_2$ layers of samples by the deposition pressure 0.2 Pa, 0.6 Pa and 1 Pa

Dependence of the surface roughness on the deposition pressure was observed on Si – samples by AFM method. Centerline average values of roughness - $R_a$ are in range 0,2 nm, 0,3 nm, and 1,2 nm by the Ar pressure 0.2 Pa, 0.6 Pa, 1 Pa on silicon B.) AFM analysis of $TiB_2$ layers of samples by the deposition pressure 0.2 Pa, 0.6 Pa and 1 Pa samples. All deposited coatings shown a metallic grey and brilliant surface. It can be seen that surfaces roughness of $TiB_2$ layers of samples prepared by the various deposition pressures had increasing tendency (Figure B.).




C.) Typical XRD patterns of TiB2 coatings deposited on Si (001) substrates at 0.2 Pa, 0.6 Pa and 1 Pa

No significant texture of $TiB_2$ coatings was observed as shown by XRD patterns in the figure C. Clear diffraction peak was found the Si (004) substrate peak at $2\theta = 44.5°$ and can be attributed to (101) diffraction of $TiB_2$ phase. Coatings thickness was 2,5 μm. Coatings seem to be X - ray amorphous or had (101) diffraction peak of $TiB_2$ phase, which is the most intense diffraction peak of ideal $TiB_2$ polycrystalline. Coatings, in case of no diffraction reflexion, could be amorphous or nanocrystalline. In case of (101) peak the coatings could be polycrystalline or nanoncrystalline, with larger grain size. Coatings growth rate was 2,8 $nm.s^{-1}$. Despite of the absence of significant (001) structure $TiB_2$ coatings had very high microhardness. Such microhardness values can be explained, by the coatings behaviour as a nanocomposite. This is also in agreement with observed X – ray diffraction patterns. To determine the nanocomposite character of these coatings is necessary to analyse them with electron diffraction. Microhardness measurements exhibited values higher than 5000$HV_{0,05}$ and sufficient adhesion for all used deposition parameters. The presented study is still in progress.

**4. Conclusion**

This interesting new-coated material on the base $TiB_2$ is object of our research. Presented results indicate special properties of these coatings (which are still under basic research) and it could be predicted that they will form a new base of coated materials with expanded usage possibilities on cutting tools and tribological applications. Therefore, the our project is focused on deposition of coatings mentioned above and their systematic physic research of characteristic and the application obtained results to real cutting tools.

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