New type coatings on the base TiB_2 for technological applications

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ABSTRACT

In this contribution are presented results of basic research of the parameters TiB_2 coatings. TiB_2 coatings were deposited onto Si (001) substrates, stainless steel and on the cemented carbides by DC magnetron sputtering from the stechiometric target in the Ar inert atmosphere. It have been study the basic properties of TiB_2 like roughness profile by Augerov Electron Sectroscopy, details study of the texture by X-ray diffraction, microhardness was achieved more than $5000HV_{0,05}$ by Vicker's method and AFM analyze. The structure of the TiB_2 coatings and their required properties needed for successful usage were controlled by the different deposition parameters.

1. INTRODUCTION

At the present time with increasing requirements on the quality, efficiency and machining output, the requirement for more powerful and precise cutting tools cannot be ignored. Current progressive cutting tools made from high-speed steel, as well as from sintered carbides are characteristically used for multiple-layered hard, abrasion resistant coatings. These hard coatings are used for high-speed steel and hard metal progressive cutting and forming tools. One of the most perspective paths for the development of modern tools are new methods, where plasma technology definitely belongs, which creates secure sufficient core toughness of the tool and sufficient hardness of the coated surface. A concurrent feature when using these machining tools is their consistent dulling, surface degradation caused by adhesion and abrasion wears, corrosion, fatigue and as well as other influences. That is why the fundamental function of thin-filmed coatings is to decrease the intensity of tool wear, which in turn increases the lifetime, productivity and quality of cutting tool.

Deposition processes for prepared thin hard layers are known for several decades, but still are coming new inventions of layer and their combinations. Deposition of thin hard coatings has great importance in many engineering applications. There are known many types of coatings for example TiN, TiC, TiAlN, Al_2O_3, AlCrN, B_4C, CrC, SiO_2, TiAlSiN - multilayers, diamond coatings, nanocomposites *(Fig.1).*

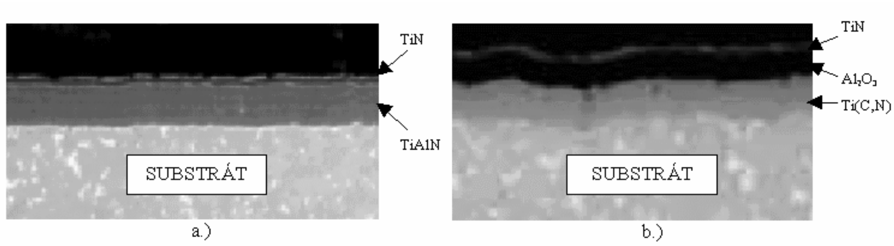


Fig. 1. a.) TiN+TiAlN – PVD coating with a great adhesive
b.) TiN+Al2O3+Ti(C,N)- multilayers coating [6]

Although it appears, that facilities of the improvement of the properties of coatings have been depleted is not true. The latest research shows on the perspective usage coated materials of the types TiB_2 which have the special properties. *Titanium diboride (TiB_2)* is ceramic compound with hexagonal structure in which boron atoms form a covalently bonded network within metallic. Specify resistance (6,4-9,1μΩcm) is low, temperature coefficient of dilatability ($4{,}6*10^{-6}$ K^{-1}). These chemical stability and high heat resistance ceramic materials with high hardness and high wear resistance fulfil high demands for modern progressive cutting tools and prepared diboride coatings show a high potential for tribological applications.

This paper addresses the results of basic research of coatings TiB_2. The coatings were prepared DC magnetron sputtering methods on the Si (001) substrates, stainless steel and on the hard carbides. The aim of the work was overview of the parameters, which influence generation of thin hard coatings and their required properties needed for the technological applications.

2. EXPERIMENTAL DETAILS

As substrate material Si (001) substrates and mirror polished stainless steel substrates were used. Before deposition, the substrates were cleaned in an ultrasonically pured acetone, izopropylalcohol and distilled water bath. The deposition was carried out using DC magnetron configuration. Visual characterization of the sputtering process indicated a concentration of the plasma at the target centre and that lead to higher growth rate for substrates located close to the centre of the substrate holder. TiB_2 target have diameter 40 mm and thickness 6 mm. The substrates have stationary positioned 5 cm from the TiB_2 target. The used process gas was argon with the purity of 99,994%. Starting vacuum was 2×10^{-3} Pa, the substrates were in-situ cleaned by argon-ion etched for 15 min using substrate bias voltage –500 V and an Ar pressure of 2 Pa. Deposition time was 15 min. The pressure was changed 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 was changed from 350 V to 390 V. Preferred orientation and phase composition was observed by X – Ray Diffraction (XRD) analysis in Bragg – Brentano geometry, using HZG4 equipment with CuK? ?monochromator (wave length – 0,015418 nm). Auger Electron Spectrometry (AES) was applied for control the coating's stoichiometry. Surface quality and roughness was analysed by Atomic Force Microscope (AFM) – Solver P47. Coatings morphology and thickness were examined by SEM (JEOL) microscopy. Parameters of sputtering process were optimized for Si (001) substrates. Stainless steel substrates were used for microharness measurement (INDENTEC indenter).

3. RESULTS AND DISCUSSION

A focused electron beam irradiates a sample surface producing Auger electrons. The energies are characteristic for the element from which they are generated. Compositional depth profiling was accomplished. Auger Electron Spectrometry 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. 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. Dependence of the surface roughness on the deposition pressure was observed on Si – samples by AFM method. Surfaces roughness increased with working pressure. Mean roughness values R_a were in range 0,2 nm, 0.3 nm, 1.2 nm by the Ar pressure 0.2 Pa, 0.6 Pa, 1 Pa.

All deposited coatings showed a metallic grey and brilliant mirror surface. It can be seen that surfaces roughness of TiB_2 layers of samples prepared by the various deposition pressure had increased with working pressure on the *Fig.2.* No significant texture of TiB_2 coatings was observed, as XRD patterns shows on *Fig. 3.*

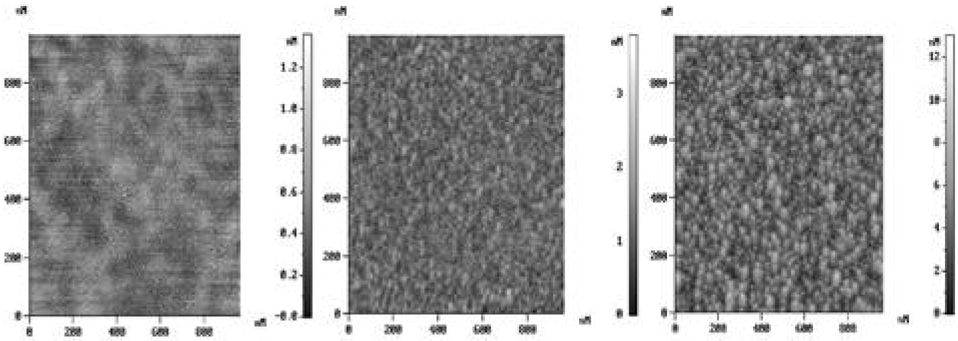
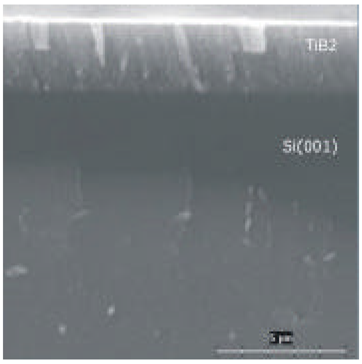


Fig.2. AFM images of TiB_2 surface deposited at Ar pressure a) 0, 2 Pa b) 0, 6 Pa and c) 1 Pa

The only sharp diffraction peak besides the Si (004) substrate peak is 2? = 44.5° and it can be attributed to (101) diffraction of TiB_2 phase. Typical SEM image of fractured coating format deposited on silicon substrates is shown in Fig.4. The coatings thickness was 2, 5 µm. The micro hardness of TiB_2 was higher than 5000 $HV_{0, 05}$ for all applied deposition parameters.

It follows from Fig. 2 that the roughness increased with increasing Ar pressure. X - ray analysis shows (101) the diffraction peak of TiB_2 phase, which is the most intense diffraction peak of ideal TiB_2 polycrystalline. Coatings growth rate was 2, 8 $nm.s^{-1}$. Despite the absence of significant (001) TiB_2 peak (often observed for values of micro hardness over 5000 $HV_{0, 05}$, the coatings had very high micro hardness. Such micro hardness values can be explained by nanocomposite character of the coatings. This is also in agreement with observed X – ray diffraction patterns. In order to determine the nanocomposite character of these coatings, it would be necessary to analyze them with electron diffraction. The presented study is still in progress.



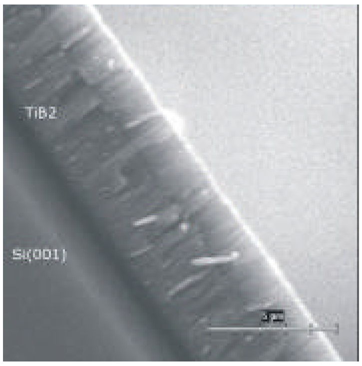


Fig.3. Typical SEM micrograph of fractured TiB_2 coating

4. CONCLUSION

It can be concluded that TiB_2 coatings prepared by DC magnetron sputtering exhibit the high micro hardness and small roughness thus they are very promising for fabrication of supercond ucting MgB_2 oriented thin films with hexagonal structure, as well as for practical applications in tool engineering.

Present results indicate special properties of these coatings, still examined in basic research and it can be predicted that they will form new base coated materials with expanded recourses for usage for cutting tools and tribological applications. This paper addresses the results of basic research of coatings TiB_2 and the research is getting on.

ACKNOWLEDGMENT

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