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CHARACTERIZATION OF TUNGSTEN BORIDE LAYERS DEPOSITED IN PULSED LASER ABLATION PROCESS

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1. Introduction

Tungsten boride compounds are very promising new hard and super-hard materials. This kind of materials could be used in production of high-speed tools, durable bearings and nonabrasive surfaces. The hardness of tungsten borides strongly depends on boron content as well as material microstructure. For example hardness of W_2B is about 12.4 GPa, WB hardness is 18 – 36 GPa, WB_2 hardness is 28.5 – 39.7 GPa and WB_3 hardness is 28.6 – 36.9 GPa [1]. Moreover production of tungsten borides in the form of layers or nano-grained structured increase its hardness even above 40 GPa. Although the properties of tungsten boride structures are known from theoretical calculations, the data concerning structures produced experimentally are scare. Therefore, the properties of tungsten boride layers deposited by pulsed laser deposition (PLD) process are investigated in this paper. Layers were deposited using two types of targets: with boron to tungsten ratio of 2.5:1 and 4.5:1 and with the use of two laser wavelengths: 355 and 1064 nm.

2. Materials and methods

In the ablation process Nd:YAG laser (Quantel YG 981 E10) was used. Laser parameters were as follow: wavelength 355 and 1064 nm, repetition rate 10 Hz, pulse duration 10 ns and spot area 3.5 mm^2. Laser fluence was 4.9 and 9.3 J/cm^2 for 355 and 1064 nm laser wavelength, respectively. During the deposition the laser beam was directed at an angle of 45° to the target surface and target to substrate distance was 42 mm. The silicon substrate (1 0 0) (Spi Supplies) was placed in front of the target and heated to temperature of 520 °C.

The target used in PLD process was sintered in spark plasma sintering process from boron and tungsten powders mixed in the molar ratio 2.5:1 and 4.5:1. The details of the manufacturing process and the properties of targets are given elsewhere [2, 3].

Crystal structure and phase composition of deposited layers were investigated in X-Ray Diffraction (Bruker D8 Discover, Cu radiation source λ=1.5418 Å) and „2 θ" scans were performed. The angle between incident radiation and layer surface was 8°. Scanning resolution was 0,02°.

Hardness of layers was measured in nano-indentation test (CSM Instruments) with the use of Berkovich indenter. Each indent was repeated at least 5 times.

3. Results

The layers produced by PLD have good adhesion with the exception of layer deposited from $WB_{2.5}$ target with laser wavelength of 1064 nm. This particular layer has weak adhesion and it almost delaminates from the substrate. The surfaces of all layers are very rough and layers are composed with droplets that size are from several nanometers to micrometre. The size and number of the droplets increased with shorter wavelength and higher born content in target.

XRD and EDS analysis shows that in comparison with target composition the layers have over - stoichiometry of tungsten. Detailed XRD analysis is presented in Fig. 1 and Tab. 1. The layer deposited from $WB_{2.5}$ target with 1064 nm laser wavelength is not presented in the table due to difficulty in identification of diffraction peaks.

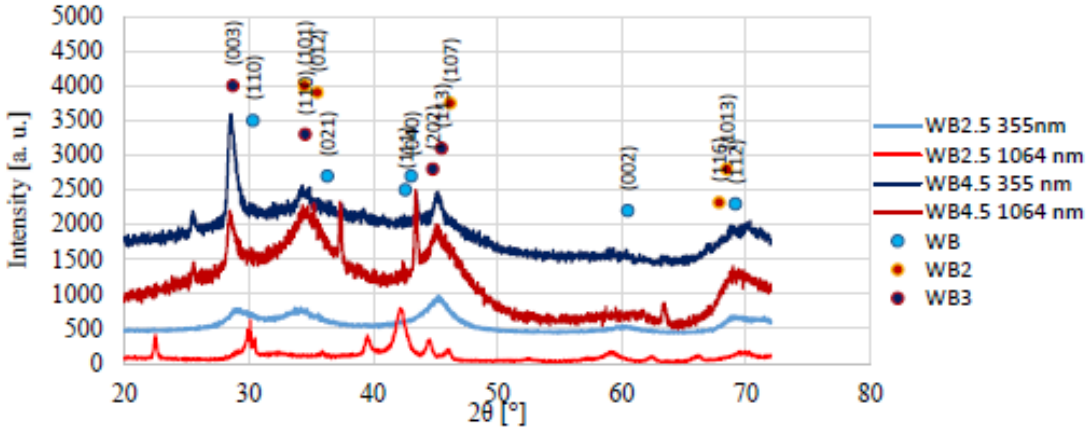

Figure 1. XRD pattern of PLD layers

Target	Wavelength [nm]	Composition
$WB_{2.5}$	355	WB:54%, WB_2: 46%
$WB_{4.5}$	355	WB_2: 59%, WB_3: 41%
$WB_{4.5}$	1064	WB_2: 32%, WB_3: 68%

Table 1. Phase composition of deposited layers. Cell parameters are as follow: WB: a=3.177, c=8.485; WB_2: a=3.016, c=21.052, WB_3: a=5.213, c= 9.408

The hardness of deposited layers was measured with loads from 5 to 30 mN. Hardness of layers deposited using various wavelengths was similar. Layers deposited from $WB_{2.5}$ target have hardness of about 54 GPa and layers deposited from $WB_{4.5}$ target have hardness of about 48 GPa. Presented values are only indicative because of layers roughness.

4. Conclusions

Presented layers change their elemental and phase composition with the change in laser wavelength. Deposited layers have high hardness but very rough surface. It is worth to investigate the reason of droplets formation because it may allow quality improvement of layers deposited by PLD.

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