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Short Communication

Titanium Carbide Obtained by Magnetron Sputtering of Graphite on Heated Titanium Substrate

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Titanium carbide was synthetized by sputtering graphite target on heated titanium substrate by magnetron sputtering process. The obtained samples were characterized by X-ray diffraction (XRD) analysis and Raman spectroscopy, the elemental analysis was made by Energy-dispersive X-ray spectroscopy (EDX). Titanium carbide (TiC) structure was obtained by deposition of sputtered carbon atoms and clusters to the resistively heated titanium substrate surface with temperatures 700 °C, 800 °C, 900 °C and 1000 °C. The XRD analysis showed that the formation of TiC structure is take place when the substrate is heated to 1000 °C. The Raman spectroscopy showed that when the incident power of laser is 100 % (35 mW) the structure is unstable in samples with the substrate temperatures 700 °C, 800 °C and 900 °C and the most stable titanium carbide structure is created when the substrate temperature is 1000 °C.

Keywords: Titanium carbide, Magnetron sputtering, Raman spectroscopy, Transition-metal carbide.

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1. INTRODUCTION

Transition-metal carbides have unique physical, chemical and mechanical properties. They usually have very high melting points ranged from 2000 °C to 4000 °C. Also these materials can be used in electronics and there are some predictions that transition-metal nitrides could have superconducting properties [1]. Moreover they are very chemically stable at room temperature and can be affected only by few acid solutions. However, their main commercial use results from their mechanical properties like high hardness. Thus, these compounds can be used in cutting tool production and in production of wear-resistant materials [2]. Wear resistance of cutting tools and drills can be significantly increased by deposition of thin films of TiC and other transition-metal carbides and nitrides to the surface of the material [3, 4]. Also there is a good application of these materials as erosion and corrosion resistant coatings [5, 6]. Physical vapor deposition (PVD) and chemical-vapor deposition (CVD) processes are generally used to make thin film coatings of titanium carbides and nitrides [7].

2. EXPERIMENTAL

Titanium carbide was obtained by magnetron sputtering of a graphite target to the heated titanium surface in vacuum chamber. Vacuum system VUP-5 with diffusion and roughing pump can produce the vacuum with pressure 10^{-4} Pa. Magnetron cooled by water with graphite target was placed in chamber (see Fig. 1). Direct current (DC) power supply with the negative electrode was connected to the cathode of the magnetron. The voltage and current between magnetrons anode and cathode was 600 V and 100 mA, respectively. The deposition time was 15 minutes. Pure argon gas (99.999 %) was used as a working gas.

The substrate was prepared from titanium foil with the 60×10^{-6} m thickness by cutting it with the 0.5 mm × 1.5 mm area. In order to resistively heat the substrate, the electric potential difference was applied to the titanium substrate. The temperature was measured by thermocouple (chromel-alumel). Four different samples were obtained at four different substrate temperatures (700 °C, 800 °C, 900 °C and 1000 °C).

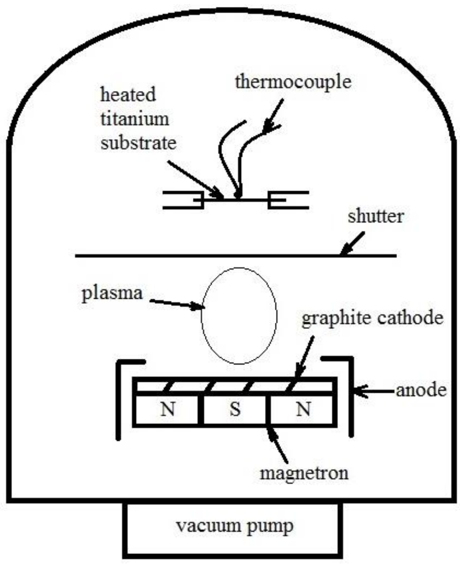

Fig. 1 – Schematic diagram of vacuum chamber

3. RESULTS AND DISCUSSION

Samples were characterized by Raman spectroscopy (AFM-Raman instrument Solver Spectrum, NT-MDT). To take a spectrum we used blue laser with wavelength

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473 nm and maximum laser power 35 mW. In order to get good spectrum, we used smoothing operation (Savitzky-Golay method, Points of Window 5, Polynomial Order 2). Four different samples at four different substrate temperatures (700 °C, 800 °C, 900 °C and 1000 °C) have almost same the Raman peak intensities at 30 % power of incident laser (approximately 11 mW, 100x objective and 2×10^{-6}m spot size). With 30 % of laser intensity we observed the characteristic spectrum of TiC, with vibrational modes at 257 and 437 cm $^{-1}$ (see Fig. 2). Peaks at 260 and 420 cm $^{-1}$ corresponds to TiC according to paper [8]. There is also report that the characteristic peaks of $Ti_3C_2O_2$ and Ti_3C_2 have vibrational modes at 347, 730 and 621 cm $^{-1}$ respectively [9], while samples which was synthetized in this work have peaks at 344, 735 and 634 cm $^{-1}$. We presume that the small variations in vibrational modes comparing to literatures, can be explained by different laser wavelengths.

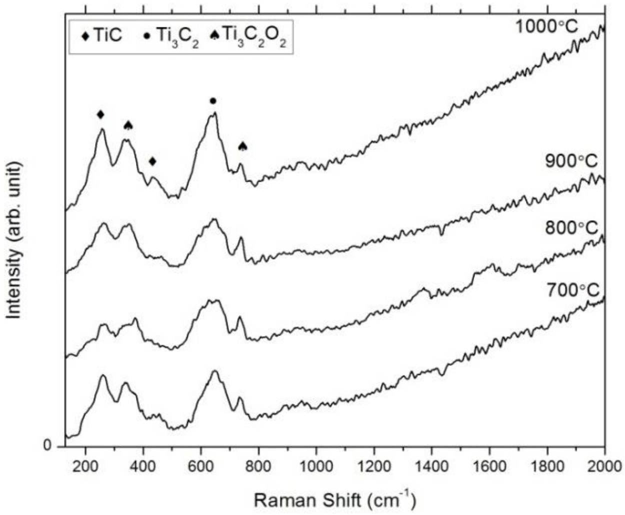

Fig. 2 – Raman shift analysis with 30 % of incident laser power

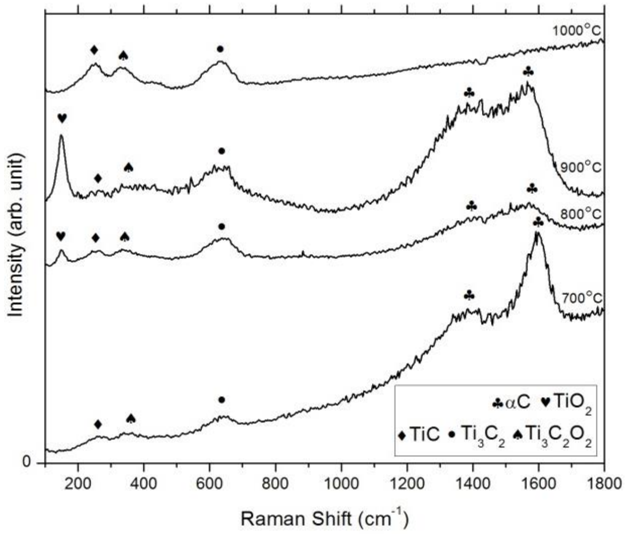

Fig. 3 – Raman shift analysis with 100 % of incident laser power

It was determined that with full power (100 %, 35 mW, 100x objective and 2×10^{-6}m spot size) of incident laser the sample which was synthesized at 700 °C has traces of TiC, Ti_3C_2 and $Ti_3C_2O_2$, but predominantly the amorphous carbons vibrational modes occurred at 1385 and 1597 cm $^{-1}$ (see Fig. 3). Which can be explained that at this temperature only small amount of carbon create the Ti-C bondings and the rest carbon creates a thin film at the surface of titanium substrate. At 800 °C and 900 °C there is also additional TiO_2 (anatase) peak at 147 cm $^{-1}$[10]. Anatase phase occurs due to the laser induced oxidation process of titanium, which did not create bondings with carbon atoms. Stable TiC, Ti_3C_2 and $Ti_3C_2O_2$ phases are formed when the substrate temperature is 1000 °C, there are no amorphous carbon and anatase (TiO_2) peaks, which indicates that all carbon atoms has finished Ti-C bondings with titanium.

Energy-dispersive X-ray spectroscopy (EDX) analysis (FEI, Quanta 3D 200i) results show the contents of titanium, carbon and oxygen by atomic percent (at. %). According to the histogram, the carbon is increasing gradually from 23 % to 37 %, while the oxygen is exhibiting relatively stable value at the substrate temperature range between 700 °C to 1000 °C (see Fig. 4). The scanning electron microscopy (SEM) microphotographs show the surfaces of the samples regions from where the EDX analysis was taken (see Fig. 5).

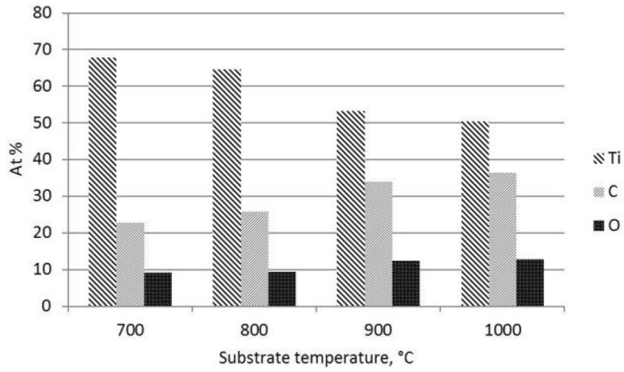

Fig. 4 – Histogram of EDX analysis results of the samples

X-ray diffraction (XRD) analysis was performed by X-ray diffractometer DRON-7, with 0.1 degree scanning step, Cu K_{α} irradiation with wavelength 1.5404×10^{-10} m(Å). XRD pattern shows the alpha titanium structures [11] at 700 °C and 800 °C with no trace of the TiC structure and at 900°C there are few peaks referring to the TiC structure [12] at 42 and 61 degrees (see Fig. 6). When the substrate temperature is 1000 °C, there is an additional peak at 35 degrees, which belongs to TiC structure.

4. CONCLUSION

Titanium carbide was synthetized by sputtering of a graphite target during the deposition of carbon atoms on heated titanium substrate. The substrates were resistively heated to 700 °C, 800 °C, 900 °C and 1000 °C. The samples were analyzed by Raman spectroscopy, XRD analysis, EDX spectroscopy and SEM. The XRD analysis showed that TiC structure is created when the

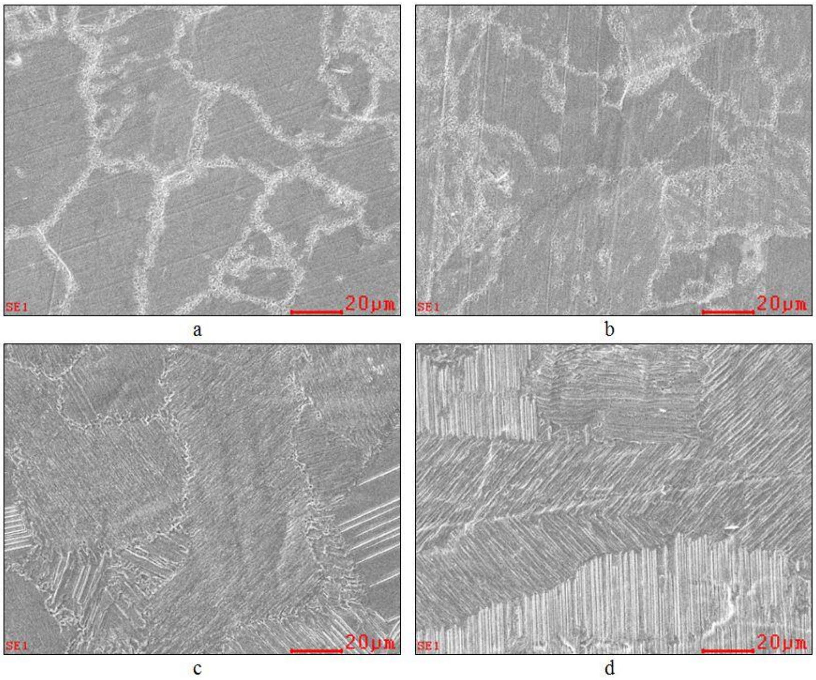

Fig. 5 – SEM images of the samples surfaces synthesized at different temperatures: a) 700 °C; b) 800 °C; c) 900 °C; d) 1000 °C

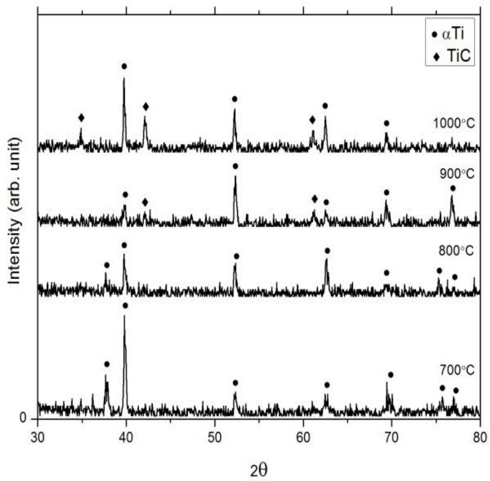

Fig. 6 – XRD analysis results

substrate is heated to 1000 °C. The Raman spectroscopy showed that with 30 % of power of incident laser there are no significant differences in Raman peaks positions and in all four samples and there are TiC, Ti_3C_2 and $Ti_3C_2O_2$ vibrational modes occurred. With 100 % of incident power of laser (35 mW) the structure is unstable in samples with the substrate temperatures: 700 °C, 800 °C and 900 °C and the most stable titanium carbide structures occurs when the substrate temperature is 1000 °C.

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