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Hybrid laser-magnetron deposition of TiC and TiCN thin films

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INTRODUCTION

Hybrid laser-magnetron deposition system was developed and tested for study carbon based thin films. Films were fabricated in argon (TiC) and in argon/nitrogen (TiCN). Crystalline films were grown at room substrate temperature. Film properties were studied by SEM, XRD, XPS and GDOES.

Hybrid deposition systems are used to overcome some of the limitations of particular deposition techniques, to find new deposition possibilities, and to grow films with a new properties or under technologically acceptable conditions[1,2]. One group of hybrid systems is based on the combination of pulsed laser deposition and magnetron sputtering (PLDMS). The combination of high- energy laser plasma with low-energy magnetron plasma was used for producing hard, tough and low friction carbon based wear protective coatings, for the deposition of nanocomposite films as well as for fabrication of composite "chameleon" coatings with adaptation to dry/humid environments. The pioneering works in MSPLD were done by A. Voevodin[3,4]. The PLDMS technique allows growing of complicated multicompound and graded layers from two separate targets, at specific deposition conditions and to control film morphology, stoichiometry and crystal structure. In graded layers the fine, gradual change in composition and microstructure results in a corresponding change in the material properties.

EXPERIMENTAL

The experimental hybrid set-up consists of pulsed laser deposition and magnetron sputtering system. Laser and magnetron targets are placed in stainless steel deposition chamber. Both sputtering systems are running together and fluxes of material intersect on Si substrate. The focus was on creation of TiC and TiCN films. KrF excimer laser was used for deposition of carbon compounds and magnetron (2 inch K.J. Lesker) simultaneously sputtered Ti species. Basic arrangement of PLDMS deposition is in figure 1. Target-substrate distances X and Y, declination angle Z and eccentricity of substrate axis E were changed. Films were fabricated in argon (TiC) and argon-nitrogen (TiCN). The substrates were cleaned in acetone and toluene. Each substrate was subsequently cleaned in the deposition chamber, before deposition, by RF discharge (100 W) in a mixture of argon and nitrogen. Substrate was rotated to ensure film thickness homogeneity over the surface of 3 cm×3 cm.

The film properties were structurally characterized by XRD. A parallel beam optic geometry with a Huber two-circle diffractometer, powered by a rotating anode generator X-ray source (300 mA, 55 kV, Cu Kalpha radiation), RIGAKU Rotaflex RU 300 was used. The carbon, titanium, nitrogen and silicon concentrations along a film thickness profile were measured by the GDOES/QDP method (Glow Discharge Opti-

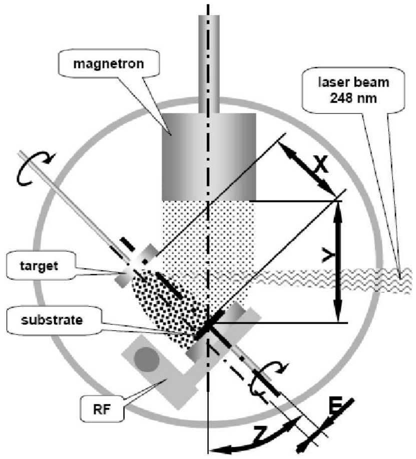

Figure 1 : MSPLD arrangement

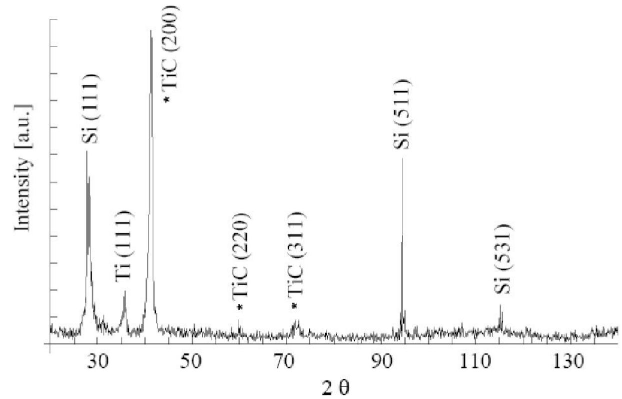

Figure 2 : XRD spectrum of TiC sample prepared by PLDMS technology (magnetron power 50 W, laser energy density 11 J.cm^{-2}, rep. rate 20 Hz, growth rate 14 nm/min)

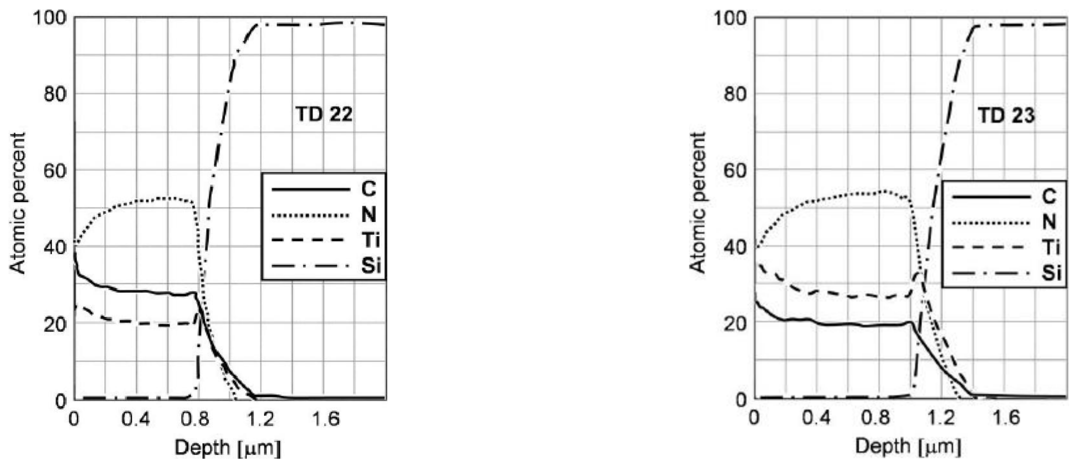

Figure 3 : Example of atomic concentration of elements in TiCN films (GDOES)

cal Emission Spectroscopy/ Quantitative Depth Profiling) using a LECO system type SA 2000. Information was taken from 4 mm diameter surface. XPS data were measured in a 2×10^{-8} Pa base pressure UHV chamber equipped with Mg Kalpha radiation at 1253.6 eV X-ray source.

RESULTS AND DISCUSSION

The focus was on TiC and TiCN layers. Titanium carbide based materials exhibit an unique combination of physical properties such as high hardness, low friction coefficient, high thermal conductivity, and high thermal stability. TiCN thin films are used for improvement of adhesion of carbon based thin films as an interlayer prior to carbon based deposition[5,6].

TiC

TiC magnetron was used for sputtering of titanium target and laser for ablation of carbon target. Films were grown at room substrate temperature (T_s) on Si substrates in nitrogen ambient of 0.3 Pa. Magnetron power was changed between 50-200 W. The following deposition configurations were tested (Figure 1).

X = 45 mm, Y = 63 mm, angle Z = 23°, E = 5-10 mm,
X = 40 mm, Y = 85 mm, angle Z = 45°, E = 10 mm,
X = 45 mm, Y = 75 mm, angle Z = 45°, E = 0 mm.

The best film homogeneity was reached for configuration (b). In all three configu-rations a crystalline TiC was measured (Figure 2).

TiCN

TiCN for deposition the configuration (b) of magnetron and laser target was used. DC magnetron sputtered Ti and operated at 150 W. Laser fluence on the carbon target was 15 J.cm^{-2}. The stream of material was changed by laser repetition rate (4-14 Hz). Ambient argon-nitrogen pressure was adjusted in region from

TABLE 1 : TiCN films - deposition conditions and XPS analysis (laser density 15 J.cm^{-2}, magnetron power 150 W)

Sample	Laser rep. rate [Hz]	RF discharge [W]	Amb. Ar + N_2 [Pa]	Av. thick. [nm]	Concentration of elements in TiCN Layers(at %) XPS			
					C	Ti	N	O
TD 22	7	-	1.5	710	63	17	17	3
TD 23	4	-	1.5	743	-	-	-	-
TD 26	4	-	5	635	40	30	18	12
TD 27	4	30	1.5	467	35	37	26	2
TD 28	4	10	1.5	643	39	34	24	3
TD 29	4	70	1.5	303	43	30	23	4

1 Pa to 5 Pa. Substrate (111) Si was held at room temperature. Film growth rate was between 6 - 24 nm/min. Differently from crystalline TiC layers fabricated in vacuum at similar conditions all TiCN films were amorphous. Very smooth films on area as 3 cm x 3 cm were fabricated by adding RF discharge (13.56 MHz, held between substrate and chamber) into PLDMS process. The concentration of nitrogen in the films was in the region 17-26 at %, as confirmed by XPS measurement (TABLE 1).

CONCLUSIONS

In this contribution there were summarized our experiences and reached results with newly constructed hybrid laser-magnetron deposition system. Several configurations was tested to cover homogeneously area of 3 cm×3 cm. Layers of TiC and TiCN were synthesized. Smooth, nanocrystalline TiC were created at room substrate temperature. The films of TiCN deposited also at room temperature T_s were amorphous. The XPS measurement confirmed TiC or TiCN bonds. The PLDMS is much complicated compared to PLD, because of additional deposition variables as geometrical configuration of deposition arrangement. Two different energetic fluxes (up 1 keV for laser ablation and several eV for magnetron species) of material deposited on the same substrate and geometrical versatility offer many possibilities to create films of new properties. The method allows also easily and smoothly changes composition profile along layer thickness (Figure 3) by changing of laser repetition rate and magnetron power. The PLDMS system is a suitable tool for fabrication of specially tailored layer at new deposition conditions.

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