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Microstructure, near infrared reflectance, and surface temperature of Ti-O coated polyethylene terephthalate fabrics prepared by roll-to-roll high power impulse magnetron sputtering system

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ABSTRACT

Among numerous coating techniques used to deposit Ti-O thin films, high power impulse magnetron sputtering (HIPIMS) has attracted a great deal of attention recently due to its high plasma density, that forms a strongly adhered film at a relatively low temperature. In this study, a roll-to-roll high power impulse magnetron sputtering (R2R-HIPIMS) system to produce multifunctional Ti-O coatings on polyethylene terephthalate (PET) fabric substrate has been used, which takes advantage of large-scale production and high-quality coatings. Microstructure, NIR reflectance, and surface temperature of Ti-O coated PET fabrics have been investigated. Results indicate that the R2R-HIPIMS is a promising sputtering process in fabricating Ti-O coatings for applications in functional textiles.

1. Introduction

Titanium related materials with outstanding properties, such as titanium alloy, titanium oxide, titanium nitride, and titanium carboxylate, have had a wide range of applications in the fields of optics, microelectronics, mechanics, and biomedical and environmental engineering [1]. Ti-O system was proposed during decades of investigations with various stoichiometries [2] in which titanium monoxide (TiO) and titanium dioxide (TiO₂) are widely used materials in many applications, such as decorative and protective coatings [3], micro-electronic layered structures [4], and photocatalysis, photovoltaic, and self-cleaning materials [5–7].

Various processing techniques have been used to deposit Ti-O thin films, including sol-gel process [8], thermal evaporation [9], micro-arc oxidation [10], RF reactive sputtering [3, 11], and DC reactive magnetron sputtering [12], and high power impulse magnetron sputtering (HIPIMS) [13–15]. HIPIMS is a sputtering process that has recently attracted a great deal of attention due to its high plasma density [16, 17], that forms a strongly adhered film at a relatively low temperature [18]. In producing Ti-based oxide films on PET fabric, HIPIMS shows advantages in comparison with the wet process due to the convenience of adjusting the oxygen flow rate to obtain different stoichiometries with high-film quality and avoided thermal damage. Moreover, according to findings by Vergöhl et al., HIPIMS was used to improve

refractive index and density of titania coatings [15]. These coatings can have a high reflectance in the near-infrared (NIR) region, and hence they are able to reflect most of the solar heat reaching the coated surface, providing a cooling effect for the wearer by reducing surface temperature of the clothes when exposing under the sun [19]. Therefore, the present study has used a roll to roll high power impulse magnetron sputtering (R2R-HIPIMS) system to produce Ti-O coatings (the term Ti-O coatings includes pure Ti coating in most cases in this study) on polyethylene terephthalate (PET) fabric substrate. This takes advantage of large-scale production and high-quality coatings. Process parameters consisting of oxygen/argon flow rates were changed to obtain a series of Ti-O coatings. Microstructure, NIR reflectance, as well as their effect on the fabric surface temperature or the cooling effect of the R2R-HIPIMS Ti-O coatings were investigated. The results indicate that the R2R-HIPIMS is a promising sputtering process in preparing Ti-O coatings for applications in functional textiles.

2. Experimental details

2.1. R2R-HIPIMS deposition of Ti-O on PET fabric

R2R-HIPIMS system was designed as three parts: power supplies (HIPIMS and RF), vacuum chambers, and a control system, as shown in Fig. 1. The three vacuum chambers, including the loading chamber, the

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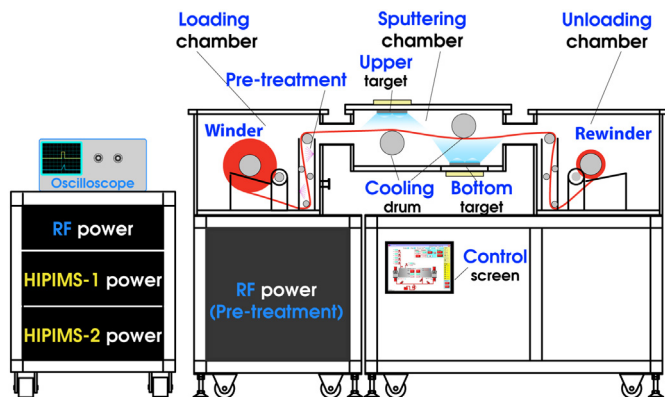


Fig. 1. Schematic drawing of R2R-HIPIMS system.

sputtering chamber, and the unloading chamber, were combined. There were 4 pumps in this system, in which a rotary pump (pumping speed: 600 l/min) worked as rough vacuum pump combined with a mechanical roots pump (pumping speed: 3000 l/min), and two diffusion pumps worked as high vacuum pump. The winder (fabric roll) was put in the loading chamber that was installed with an RF power for pre-treatment (glow-discharge sputter cleaning), then deposited in the sputtering chamber, and finally rewound in the unloading chamber. A single rectangular magnetron source was placed at the sputtering chamber with a target area of 362 cm² (32.3 cm × 11.2 cm). The HIPIMS power supply was manufactured by Taiwan Power Tech. The main body and the matching box of the RF power supply were provided by ENI (ACG-6B-01) and Rich Field, respectively [20].

A PET fabric roll with a width of 0.17 m was used as a substrate. The fabric has a density of 75 D × 75 D, a weight/volume of 60.5 g/m², and a fabric thickness of 0.16 mm. Titanium (Ti, 99.8%) was used as the upper target material with a working distance of 7 cm. During the reactive sputtering, the PET fabric roll was run at a constant web speed of 0.16 m/min, and the oxygen/argon flow ratio was changed from 0/40 to 40/40 to produce different Ti-O coatings. In addition to the PET fabric substrate, the glass substrate was also put into the sputtering chamber to confirm the crystal structure of the Ti-O coatings, as shown in Fig. 2. The depositions were operated at a constant average current of 3 A. The base pressure was 6.67 × 10⁻⁴ Pa (5 × 10⁻⁶ Torr). The experimental parameters, including the pre-treatment and deposition, are shown in Table 1. Two modes, namely winding mode and stationary mode, were carried out in turn during the coating work. The coatings at higher O₂/Ar flow ratios (from 10/40 to 40/40) were also produced on PET fabric in stationary mode separately, owing to very low deposition rates. The argon flow rate was kept at a constant of 40 sccm, while the

Table 1
Pre-treatment and deposition parameters for Ti-O coatings on PET fabric.

Pre-treatment condition (Argon pre-treatment parameters for winding & rewinding)							
RF power (W)	100						
Working pressure (Pa)	0.667						
Web speed (m/min)	0.45						
Ti-O coating condition (Winding and stationary mode operated separately)							
Sputtering target	Ti (99.8%)						
Target area (cm ²)	362						
Target-to-substrate distance (cm)	7						
Discharge frequency (Hz)	854.7						
T _{on} /T _{off} (μs/μs)	70/1100						
	Winding mode						
Working pressure (Pa)	0.159	0.169	0.175	0.191	0.223	0.253	0.283
O ₂ /Ar ratio (Ar fixed at 40 sccm)	0/40	3/40	5/40	10/40	20/40	30/40	40/40
Discharge voltage (V)	752	748	740	653	601	580	560
Peak discharge current (A)	135	140	150	193	194	195	195
Web speed (m/min)	0.16						
Deposition time (min)	5						
	Stationary mode						
Deposition time (min)				30	30	30	30

oxygen flow rate was increased from 0 to 40 sccm. The working pressure increased from 0.159 Pa at an O₂/Ar ratio of 0/40 to 0.283 Pa at an O₂/Ar ratio of 40/40.

2.2. Microstructural characterization of the Ti-O coatings

The crystal structure of the deposited films was identified using a Bruker D8SSS grazing incident angle X-ray diffractometer (GIXRD) with CuKα radiation at a grazing angle of 0.7°. A field-emission scanning electron microscope (FE-SEM, HITACHI S4800) was used to observe the surface morphology and cross-section of the deposited Ti-O films.

2.3. Near-infrared reflective measurement

The diffuse Vis-NIR reflective property of the Ti-O coatings on PET fabric was analyzed by a Vis-NIR spectrometer (Avantes, AvaSpec-NIR256-1.7) (400–1650 nm) with a 30-mm integrating sphere for reflection (250–2500 nm) and a 6-mm diameter sample port. The white standard is made of a white diffuse polytetrafluoroethylene (PTFE)-based material.

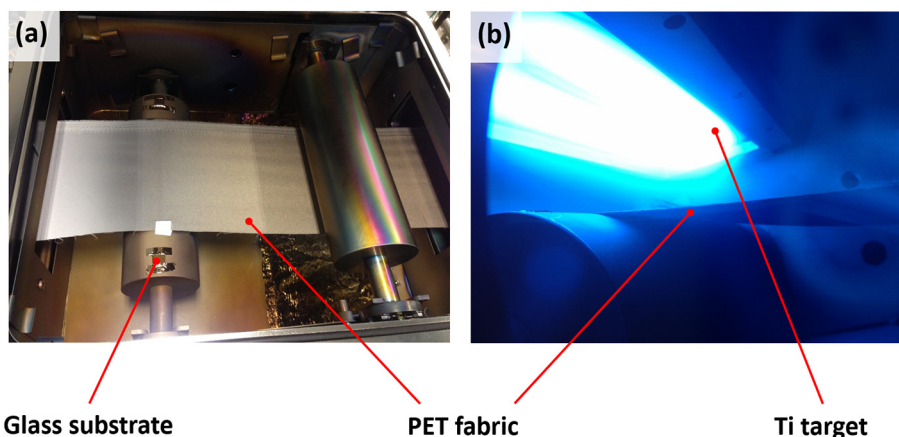


Fig. 2. The sputtering chamber in the R2R-HIPIMS system with (a) top view and (b) side view.

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