Applied Surface Science 321 (2014) 173–178

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Laser-induced nanostructures on a polymer irradiated through a contact mask

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ARTICLE INFO

Article history:
Received 6 May 2014
Received in revised form 27 September 2014
Accepted 29 September 2014
Available online 7 October 2014

Keywords:
Excimer laser
Polymer
Atomic force microscopy
Contact mask
Periodic nanostructures
Gold deposition

ABSTRACT

The nanopatterning method applied through micrometer slit for polyethylene naphthalate (PEN) substrate was proposed in this paper. Surface roughness, formation of nanoscale ripple-like structures and the dependence of their dimensions on the value of laser fluence was determined by atomic force and laser confocal microscopy, and compared with values obtained from samples irradiated directly (without a contact mask) under similar conditions. The morphology of the unirradiated surface of the substrate in between the slits is also studied, as well as the morphology of the transitional area between the irradiated and unirradiated surface. Thin layer of gold was deposited on selected samples. Chemical composition of the surface was determined from XPS spectra. The potential application of this research can be found predominantly in the field of selective sensor applications, where the designated area for the consecutive grafting procedures is of great importance.

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1. Introduction

Properties of a surface are highly dependent on its morphology. Roughness of a surface increases its area, which means more space for chemical reactions to take place in. Surface roughness also influences tribological and optical properties [1]. Chemical properties of surface and its morphology also play an important role when it comes to the interaction of living cells with the polymer substrate [2]. There are several methods of surface modification for an appropriate application, which include plasmatic discharge modification, ion beam modification and modification by laser radiation [2–4]. In the process of excimer pulse laser treatment of the surface of a polymer, the bulk of the substrate remains unaltered while the morphology and the chemical properties of the surface undergo changes [3,4].

Irradiation by a laser beam of appropriate properties causes various periodic structures to form on the surface of a polymer. There are several kinds of these periodic surface structures, ranging from dots to ripples. The most common pattern is a ripple-like structure, where the direction of the ripples is parallel with the main polarization of the laser beam. The distance between individual ripples (their period) depends on several factors, namely the chemical structure of the polymer, wavelength of the laser radiation and the angle of incidence [1]. The classification of the surface structures depends on the ratio between their spatial periodicity and the wavelength of the laser radiation. Structures with a spatial periodicity comparable with the wavelength of the laser radiation are classified as structures with low spatial frequency (LSFL), while those with spatial periodicity much lower than the wavelength of the laser radiation are classified as structures with high spatial frequency (HSFL) [5]. Periodicity of the structures with low spatial frequency is determined by the wavelength and the angle of incidence of the laser radiation [15] and their orientation is determined by the vector of polarization [16]. Laser-induced formation of such periodic structures has been studied on several different polymer substrates, including polyethylene terephthalate [10,14], polystyrene [11], polyimide [17] and polyethylene naphthalate [12]. Current research aimed on the application of these periodic surface structures focuses on surface functionalization [6].

The value of the laser fluence at which the formation of the surface structures occurs can be close to the intensity above which the whole surface layer of the polymer melts. Irradiating the polymer with fluence higher than is necessary for the formation of period surface structures but lower than the ablation threshold lowers the crystallinity of the surface. This is most likely caused by the reorganization of the polymer chains as the melted material quickly

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http://dx.doi.org/10.1016/j.apsusc.2014.09.187
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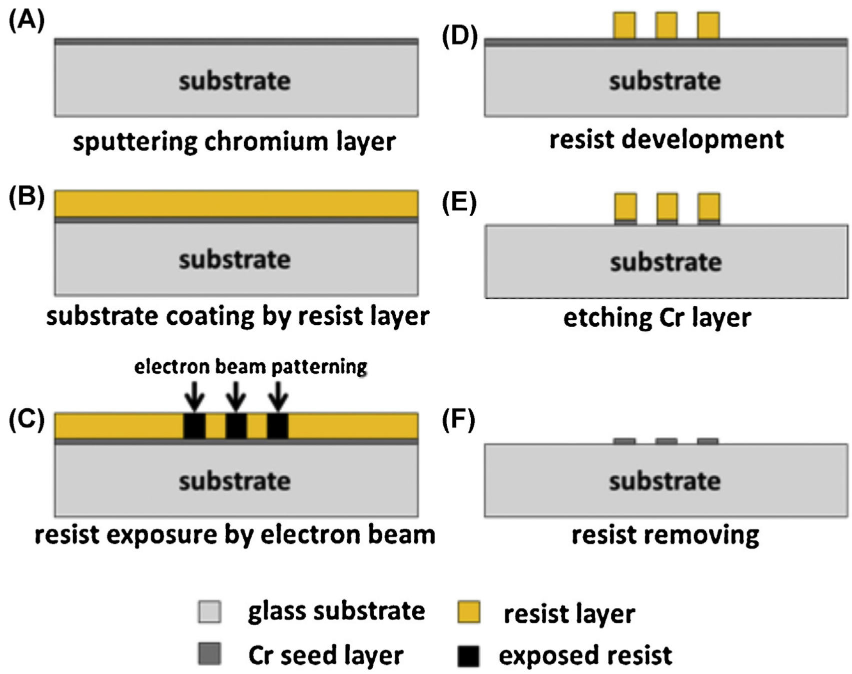

Fig. 1. Scheme of the process of the chrome-on-glass contact mask construction. Cr opaque layer is sputtered onto a glass substrate (A). The substrate is covered by electron beam lithography (EBL) resist (B) and the resist is patterned by electron beam (C). Then the resist is developed (D) and Cr layer is etched (E). Finally the resist layer is completely removed from the substrate (F).

cools off, not giving the molecules enough time to re-assemble into a highly ordered structure [18].

Surface excimer laser treatment of various substrates is currently being utilized in biotechnology, as the laser radiation breaks chemical bonds on the surface, allowing the formation of new bonds and free radicals, which allows the grafting of new functional groups, such as amino acids and carboxyl groups, which promote protein adsorption and the adhesion of eukaryotic cells [7]. Furthermore, laser treatment of the polymer surface can lead to changes in heat conductivity [8], as well as facilitating metallization, which is necessary for many microelectronic and biotechnological applications, as adhesion on the metal–polymer interface is highly dependant on the structure and chemical properties of the surface [9]. It is also frequently utilized in the process of surface modification [19], chemical vapor deposition [20], and lithography [21,22]. For surface analysis, power spectral density is also a powerful tool. It can be obtained by computing autocorrelation function in Fourier space. From physical point of view, high frequency wave number of the Power Spectral Density of a 2D image, can be related to sharp variations in intensity values of the pixels in the image domain [23–25].

In this paper we present the study of ripple formation of PEN surface induced by irradiation through a contact mask. The surface morphology and dimensions of prepared structures were evaluated and confronted with those prepared earlier on pristine samples without mask [12]. The surface chemistry was determined and compared with values obtained from samples irradiated without mask. The detail study of oxygen bonding on the irradiated surface was performed. Laser confocal microscopy was also used for the morphology determination. The study of gold nanolayers growth on such prepared nanopattern was also studied.

2. Materials and methods

Biaxially oriented polyethylene naphthalate foils (1.36 g cm^{-3}, thickness of 0.05 mm, $T_m \sim 250$–$290\,^{\circ}$C, $T_g \sim 120\,^{\circ}$C, supplied by Goodfellow Ltd., UK) were used.

The contact mask was created on a glass substrate (thickness 1 mm, material Spectrosil®, supplied by UQG Ltd., UK). Opaque chromium layer (thickness 100 nm) was deposited on cleaned substrate by sputtering technique (Q150T Turbo-Pumped Sputter Coater/Carbon Coater, Quorum Technologies Ltd., UK) and a series of 20 × 1000 μm slits with a period of 40 μm were patterned by electron beam lithography (VEGAII, TESCAN Brno, s.r.o., Czech Republic) (Fig. 1).

Samples were mounted on a translation stage with the mask attached to them, the chrome-coated side being in contact with the surface of the polymer.

Samples were irradiated by a KrF excimer pulse UV laser (Lambda Physik Compex Pro 50, wavelength of 248 nm, frequency of 10 Hz) for the duration of 6000 pulses under the angle of incidence of 0°, with laser fluence in the range from 6 to 12 mJ cm^{-2} (steps of 2 mJ cm^{-2}).

The gold layer was deposited from a gold target (99.999%) by means of diode sputtering technique (BAL-TEC SCD 050 equipment). Typical sputtering conditions were: room temperature, time 300 s, total argon pressure of about 5 Pa, electrode distance of 50 mm and current of 40 mA. For the measurement of the layer thickness gold layer was deposited under the same conditions on a Si (1 0 0) substrate. The gold layer thickness was determined from scratches measured with AFM. Typically five measurements on three scratches each were accomplished on each sample. The thickness of the gold layer sputtered under above described conditions was about 100 nm.

Surface roughness and the dimensions of the ripple-like structures were measured by atomic force microscopy in a tapping mode. Surface roughness measurements and dimensions of the surface structures were taken from 10 μm^2 × 10 μm^2 scans. The error of measurement in case of surface roughness determination did not exceeded 5%. All scans were acquired at a line scanning rate of 1 Hz. The concentration of the elements on the surface was obtained from ARXPS spectra measured by Omicron Nanotechnology ESCAProbeP spectrometer and evaluated by CasaXPS software. Both the mask and the polymer samples were

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