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Preparation and characterization of fully separated gold nanowire arrays

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ABSTRACT

We reported on the development of an unconventional approach for the physical synthesis of laterally ordered self-organized arrays of well-separated metallic nanowires supported on nanostructured dielectric templates. The method, combining nanoscale patterning of the polyethylene terephthalate substrate by polarized light of excimer laser with glancing angle deposition of the gold, provides an interesting alternative to lithography-based nanopatterning approaches. Separation of individual nanowires is manifested by anisotropy of their electrical properties. Focused ion beam equipped scanning electron microscopy analysis revealed that the resulting nanowires were organized in parallel arrays of approximately 2500 wires with an average length of about 1000 μm and uniform width of about 150 nm.

1. Introduction

One-dimensional (1D) nanostructures in the form of wires, rods, belts and tubes have long been the focus of intensive research owing to their unique applications in mesoscopic physics and fabrication of nanoscale devices [1–3]. Moreover, nanostructured materials with high aspect ratios such as nanorods, nanowires, and nanoline patterns often exhibit anisotropic electronic and optical properties that differ from those observed in the bulk materials. These unique materials can be used to create many interesting devices, including fast responding chemical and biochemical sensors [4–10]. The high aspect ratio of nanowires should also make them interesting for the use in two-dimensional photonic crystals [11]. Field emission from nanowires has also been reported [12], suggesting the possibility of devices such as field emission displays (FEDs) with nanowires acting as cathodes.

Of the many elements and compounds from which nanowires may be made, gold is technologically important for its low electrical resistivity (2.21 μΩ cm) [13], its inertness to attack by air and its resistance to sulfur-based tarnishing [14]. Additionally, gold is more biocompatible than most metals, rendering it suitable for implantation in [15,16] or electrical interfacing with cells [17,18] and tissues in nano-biological applications [19–21].

Within this context, we report on development of an unconventional approach for synthesis of well-separated self-organized arrays of metallic nanowires on dielectric templates. The method combines the self-organized formation of a ripple pattern on a dielectric substrate by laser irradiation, followed by metal deposition at glancing angle to form isolated metallic nanowires.

2. Experimental

2.1. Materials, apparatus and procedures

The rippled templates were produced on biaxially oriented polyethylene terephthalate (PET) foils with a thickness of 50 μm (melting point $T_m \sim 260$ °C, glass transition temperature $T_g \sim 80$ °C, supplied by Goodfellow Ltd., England) exploiting the self-organization mechanism induced by laser irradiation. For details see our former papers [22,23].

After ripple formation on the PET substrate, deposition of Au (Goodfellow, purity 99.99%) was accomplished using vacuum evaporation device LEYBOLD-Heraeus, Univex 450. The evaporation was performed at room temperature at a rate of 0.33 nm s^{-1} by means of resistively heated tungsten crucible, at a glancing angle of incidence $\varphi = 70°$ (with respect to the surface normal, as shown in the sketch of Fig. 1a). The evaluation of the deposition rate and thickness of Au was based on readout of a quartz crystal microbalance and also controlled by direct gold thickness measurement by scratch test carried out by atomic force microscopy (AFM) on silicon wafer which was deposited simultaneously with PET sample.

2.2. Analytical methods

The surface morphology was examined using an atomic force microscopy (AFM). The AFM images were taken under ambient

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conditions on a Digital Instruments CP II set-up. The samples with an area of about 1 cm × 1 cm were mounted on sample holders using double-sided adhesive tape. We used a small area scanner allowing to image areas up to 5 mm × 5 mm. The 'tapping mode' was chosen for the measurements to minimize damage to the samples surfaces. A Veeco oxide-sharpened P-doped silicon probe RTESPA-CP attached to a flexible micro-cantilever was used near its resonant frequency of 300 kHz. The scans were acquired at ambient atmosphere at a line scanning rate of 1 Hz. Four areas of each sample were scanned in order to obtain representative data. AFM working in contact mode was also used to control the effective thickness of evaporated gold by scratch method. The scratch on silicon substrate, which was simultaneously deposited together with PET templates in glancing angle geometry, was made in five different positions and scanned in contact mode. In this case a Veeco phosphorous doped silicon probe CONT20A-CP with spring constant 0.9 N m^{-1} was chosen. Thickness variations did not exceed 5%.

The electrical properties of the gold structures were examined by measuring its electrical resistance (R). The sample arrangement during resistance measurements is displayed in Fig. 1B. Electrical resistance was determined with respect to the nanowire orientation in both longitudinal (a–a' electrodes) and transversal (b–b' electrodes) directions. Longitudinal resistance (R^{L}) was determined first and transversal (R^{T}) one afterwards. For this measurement, additional Au contacts (a–a' and b–b'), about 50 nm thick, were created by vacuum deposition. The resistances up to $10^6\ \Omega$ were measured using multimeter UNI-T. Resistances exceeding $10^6\ \Omega$ were determined using picoampermeter KEITHLEY 487. The electrical measurements were performed at a pressure of about 10 Pa to minimize the influence of atmospheric humidity. Typical error of the resistance measurement did not exceed ±5%.

Focused ion beam (FIB) cuts were prepared with an adapted scanning electron microscope (FIB–SEM, 1540 XB CrossBeam, Zeiss). The FIB cuts were made with a Ga ion beam at a current between 2 nA and 200 pA. After cutting at the current of 2 nA, the surfaces were polished at the lower ion current of 200 pA. The polishing procedure was performed to clean and flatten the investigated surfaces.

An Omicron Nanotechnology ESCAProbeP spectrometer was used to measure angular resolved X-ray induced photoelectron spectra (ARXPS) [24]. The analyzed areas had dimensions of 2 mm × 3 mm. The X-ray source provided monochromatic radiation of 1486.7 eV. The spectra were measured stepwise with the step in the binding energy of 0.05 eV at each of the nine different sample positions with respect to the detector axis, which translated into different angles ranging from −80° to 80° (with respect to the surface normal). The spectra evaluation was carried out by using CasaXPS software. The composition of gold and carbon elements is given in at.%.

3. Results and discussions

The work is a continuation of our previous experiments on polymer nanostructuring by excimer laser beam [22,23,25], with the special emphasis to produce well-separated and thickness controlled gold nanowires. Fig. 2a shows the PET surface after laser irradiation. Well defined ripple modulation with an average periodicity of Λ = 210 nm and peak to valley amplitude of about 70 nm are clearly resolved. Fig. 2b shows the same structure after the gold deposition (20 nm). One can see that even after the Au coating the original ripple structure is preserved. The laser modulated and Au coated PET exhibits similar morphology. The main difference is in a fine microstructure which is in the case of Au coated samples superimposed on the top of original ripple structures. This fine microstructure is related directly to the deposition at glancing angle of incidence which leads to significant shadow effect. The rippled polymer surface, due to the shadow effects, modulates the spatial distribution of the Au flux, inducing the nucleation of metal preferentially near the top of the illuminated ridges, where the flux is much higher [26]. With increasing Au coverage, agglomeration and coarsening of the clusters proceed, until a polycrystalline array of Au nanowires is formed on the top of the illuminated ridges. The occurrence of forks and PET lines terminations which are clearly visible in Fig. 2a are due to the positive feedback mechanism breakdown owing to the presence of surface inhomogeneities. Since the distribution of inhomogeneities over the polymer surface is completely random the distribution of such defect is random too. Additionally to the AFM images, showing the presence of arrays of polycrystalline gold nanowires grown on the polymer pattern, we quantitatively estimated the lateral width of the nanowires using the derivative signal of the AFM morphology pattern.

The derivative AFM signals of six consecutive line profiles measured on laser patterned and Au coated samples are shown in Fig. 3 (three before (Fig. 3a) and three after (Fig. 3b) Au deposition, thickness 20 nm). One can see that there is significant difference between line profiles from uncoated (Fig. 3a and a′) and coated (Fig. 3b and b′) samples. While the line profiles of three consecutive cross-sections

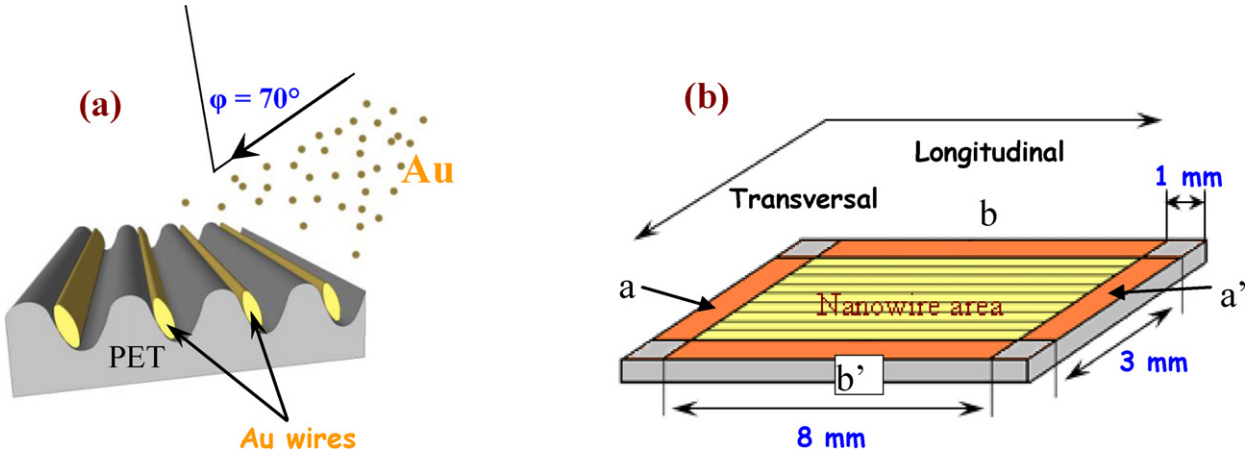

Fig. 1. Sketches of experimental set-up during gold evaporation (a) under the glancing angle of $\varphi = 70^\circ$ and during electrical resistance measurements (b). Strips a,a',b,b' represent additional Au contacts evaporated over the nanowires area. Longitudinal and transversal electrical resistance was measured between a–a' and b–b' contacts, respectively.