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Anodic growth of copper oxide nanostructures in glow discharge

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ABSTRACT

Purpose: Application of plasma glow discharge to copper oxide nanostructure growth is studied. The simplicity of the proposed technique may be beneficial for the development of new plasma reactors for large-scale production of diverse metal oxide nanostructures.

Design/methodology/approach: Copper sample was placed on anode of a setup designed to ignite plasma glow discharge. The proposed approach allows eliminating the negative effects of ion bombardment, like sputtering and generation of defects on a surface of the growing nanostructures, but preserves the advantages of thermal growth. The growth process was explained in terms of thermal processes interaction occurring on a surface of the anode with the glow discharge plasma.

Findings: Plasma treatment resulted in generation of reach and diverse nanostructures that was confirmed by SEM images. Nanowire-like, flower-like, anemone-like nanostructures and nanodisks composed into the nanoassemblies are observed; the nanostructures are associated with microbabbles on CuO layer. These findings allow concluding about the possible implementation of the proposed method in industry.

Research limitations/implications: The main limitation is conditioned by the lack of heat supplied to the anode, and absence of independent control of the heat and ion fluxes; thus, the additional heater should be installed under the anode in order to expand the nomenclature of the nanospecies in the future studies.

Practical implications: High-productivity plasma process in copper oxide nanostructures synthesis was confirmed in this research. It may be applied for field emitter and supercapacitor manufacturing.

Originality/value: Oxide nanostructure synthesis is conducted by use of a simple and well-known glow discharge technique in order to expand the production yield and diversity of nanostructure obtained in the processes of thermal growth.

Keywords: Nanotechnology, Plasma synthesis, Copper oxide nanostructures, Glow discharge

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MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

For the past two decades, nanostructures [1] of various dimensionality and nanomaterials [2] have been gaining attention in the field of science and manufacturing. Nanoparticles are being used for the dye sensitized solar cell [3] and hyperthermia [4] applications, as well as for thin film deposition [5]; the reports on successful implementations of nanowires [6] and nanocellulose [7] are also known. The interest in the nanowires is explained by the outstanding properties of these nanostructures, and, usually, clear dependence of these properties on the growth modes maintained in the reactors. Nanowires of metal oxides such as Fe_2O_3, CuO, V_2O_5 and ZnO were successfully synthesized by non-catalytic method based on resistive heating of pure metal wires or foils at ambient conditions, as was reported by Rackauskas et al. [8]. Typically, the nanowires rate of growth exceeds 100 nm/s, with the nanowires length as long as 1-5 μm and diameters of 10 to 50 nm. At that, cold electron field effect measurements showed that CuO nanowires possess excellent characteristics with a very low threshold field of 4 V/μm at the current density of 0.01 mA/cm^2. High properties of CuO nanowires with respect to their field-emitting properties was also confirmed by Feng et al. [9]. A review of advancements for supercapacitor applications of nanomaterials based on copper oxide was performed by Majumdar and Ghosh [10], where a correlation between their morphology and electrochemical performances highlighted. A field effect transistor exhibiting p-type behaviour was reported by Liao et al. [11], and it was based on CuO nanowires; moreover, a gas sensor designed on base of a single CuO nanowire shown high and fast response to CO in air. A review paper reporting the results of the study on processing and microstructure of nanostructured oxides, their photoluminescence and photocatalysis properties was made by Zou and Gao [12]. It was concluded that large surface area and the interaction between different oxides are two factors that contribute to the high reactivity of the oxide nanostructures.

Large number of techniques, which may be related to thermal, thermo-chemical, chemical, plasma-enhanced and other methods [13,14], are proposed. An extended and comprehensive review of synthesis, characterization, growth mechanisms, fundamental properties, and applications of CuO nanostructures was conducted by Zhang et al. [15], while chemical method of copper nanowire growth with alkylamines was reported by Kim et al. [16]. A review of the methods of the nanowire growth including solution-based, electrochemical, hydrothermal, thermal and plasma oxidation, as well as the corresponding growth theories, was proposed by Filipič and Cvelbar [17]. It should be mentioned that while the most reports describe the growth of CuO nanostructures [18], the room-temperature synthesis of small-diameter and phase-pure Cu_2O nanowires in inductively coupled plasma (ICP) discharge is also present [19]. Various effects of the operation parameters were observed during the studies, and the optimal parameters in order to obtain a specified characteristic were considered. Strong size effect and temperature dependence on the ferromagnetic behaviour of CuO nanowires was observed after a process of thermal oxidation at 400°C and 500°C for various growth times, as was reported by Zúñiga et al. [20]. Jafari et al. found that the crystallinity of nanowires is improved at the oxidation temperature rise, and the temperature of 700°C seems to be optimal in terms of number, density, and length of nanowires [21]. They also reported that for this temperature the nanowires showed the best photocatalytic performance and electrical conductivity. Mohamed and Al-Mokhtar carried out a characterization of Cu_2O/CuO nanowire arrays synthesized by thermal method at various temperatures, and estimated optical band-gap values withing a range of 2.56 to 2.27 eV, while the emission peaks are in the range of 335-480 nm for the nanowires grown between 550 and 650°C [22]. Sondors et al. investigated the effect of the external electric field for oxidation synthesis of nanowires in dry and wet air [23]. It was found that Young's moduli increase above 200 GPa for nanowires with diameters narrower than 50 nm, and the high aspect ratio nanowires with such diameters are grown by combining the electric field with additional water vapour at the first stage of synthesis. A possibility to affect the morphology of the nanowires with the use of the direct oxidation process was demonstrated by Xu et al. [24] who reported the growth of copper oxide nanowires from copper foils oxidized in wet air at temperatures between 300 and 800°C. At that, two different morphologies (curved and straight) were observed for the nanowires grown within the temperature range of 400-700°C; the nanowires have the lengths between 1 and 15 μm and diameters between 50 and 400 nm.

In spite of the advantages associated with plasma-enhanced methods, like the possibility to apply the magnetic control of the processing fluxes, and short processing time [25,26], thermal growth appears to be the most applied technique for the nanowire growth, that may be explained by its simplicity and cost-effectiveness. A lot of researchers have been conducting the studies with respect to establish the dependence of the growth parameters and crystallinity of the nanowires on the temperature, gas pressure and flows, which are considered as basic for this kind of treatment. Thus, for the thermal annealing process, Kumar et al.

observed that the diameter and density number of nanorods depend critically on the oxygen flow rate [27]; however, an orthogonal schematic of the oxygen supply was supposedly used thus showing the effect of the oxygen local concentration increase. At the same time the growth temperature was confirmed to be the most influential parameter. The experiments conducted by Li et al. [28] on in situ thermal oxidation of Cu_2O in the temperature range of 300-750°C in air resulted in a conclusion about the nucleation of CuO nanowires as a solid-state process. At that, the dependence of the nanowire length on the growth time obeys a parabolic law, and the growth exhibits a diffusion-controlled behaviour. A mechanism of growth based on a short-circuit, grain boundary diffusion, and experimental evidence of a bi-crystal structure growth with a boundary along the entire length of a nanowire was proposed by Hansen et al. [29]. In addition, a dependence of the morphology of the nanowire on the size of copper grains was also confirmed in the experiment on enhanced CuO nanowire growth through oxidizing nanocrystalline Cu. As a result, the authors suggested that the short-circuit diffusion of Cu ions across Cu_2O layer, followed by the short-circuit diffusion along the bi-crystal grain boundary and to the tip of the nanowire, where subsequent oxidation occurs, are the main processes of the nanowire growth. A set of experiments followed by a theoretical description of a formation of oxide nanowires from the oxidation of copper was reported by Yuan et al. [30]. On base of the research, a conclusion about the compressive stresses produced by the volume change associated with the solid-state transformation at the interface between CuO and Cu_2O layers, which stimulate the growth of CuO nanowire was made. In the experiments, the formation of twin boundaries in the nanowires was observed, and then it was attributed to the surface faceting of CuO grains which serve as the template for initiating the nucleation and growth of the nanowires. A review conducted by Rackauskas and Nasibulin is devoted to the noncatalytic synthesis of nanowires by the means of the in situ transmission electron microscopy (TEM) technique, where screw dislocation, twin boundary, and oscillatory transport are considered as the base for the formation mechanisms of the nanowires [31]. In recent research conducted by Shi et al. [32] different CuO nanostructures like single-crystal, bi-crystal and cone-shaped nanowires were synthesized in temperature range of 673 to 1073 K, and they coexisted. The authors proposed a complex stress-driven mechanism of the nanowire growth that includes Kirkendall effect, stress-driven grain-boundary diffusion, large surface free energy gap between Cu_2O and CuO crystals and an effective electric field along the nanowires caused by oxygen gradient. The model still needs a theoretical description and numerical validation to conclude about the contribution of each of the discussed effects. To study the effect of the stress that is generated in oxide layers while their growth, a theoretical model proposed by Jagtap and Chason [33] may be applied. According to it, the stress is generated due to the atomistic processes of reversible diffusion of atoms in and out of the grain boundary. The model provides a quantitative estimation of both the relaxation and recovery kinetics that may be compared directly to experiments and used to evaluate kinetic parameters of the oxide growth. The assumption about the compressive stresses caused by the formation of metal oxide layers as the driving force, and the formation of oxide molecules on surfaces of the nanowires was supported by the results obtained by Chen et al. [34]. As single as bi-crystalline structure with a twin-boundary along their growth direction was also reported by Altaweel et al. [35] who studied the compressive (respectively tensile) stress observed in Cu_2O (respectively CuO) layers. The authors concluded that the synthesis of CuO nanowires at the thermal oxidation is driven by stress-induced grain boundary diffusion processes due to the development of stress gradients. At the same time, the use of plasma afterglow increases the oxidation rate, thus enhancing the development of stress and enabling the synthesis of nanowalls (2D nanostructures) instead of nanowires (1D). A kinetic model describing the stress evolution that includes subsurface grain growth that directly induces stress in the layer and changes the grain size at the surface that affects the stress in new layers as they are deposited, is proposed by Chason et al. [36]. Further, the micro-afterglow of an $Ar\text{-}O_2$ microwave plasma operating at atmospheric pressure was successfully applied to grow even more elaborated nanostructures, like "cabbage-like" architectures [37]. At the same time, Nkhaili et al. highlighted the importance of heating rates when studying the thermal oxidation and copper oxide nanowire growth [38]. They found that dense arrays of nanowires are formed for low heating rates, while the nanowires end up being broken for quick heating.

As it follows from the literature analysis, a lot of problems still exist in the field of the thermal growth of 1D oxide nanostructures, with the controversial mechanisms of their formation. At the same time, thermal methods allow obtaining the nanostructures for hours, while plasma technologies are much more promising and the typical growth time does not exceed a few dozens of minutes. That is why the main purpose of the present research is to study the plasma enhanced growth of copper oxide nanostructures, with the arrangement of copper samples on anode of an electrical circuit that is used to sustain the simplest form of plasma discharges, namely glow discharge. The anode arrangement is intended to eliminate the negative effects caused by the severe ion bombardment associated with passing the cathode sheath by the ions, and to create the conditions that are close to those involved for the thermal growth of copper oxide nanowires.

2. Research methodology

To perform the experiments on the growth of copper oxide nanostructures, a plasma reactor utilizing the glow discharge plasma was used. Copper samples were put on the anode and exposed to the action of plasma. The electrodes were installed in the cylindrical vacuum chamber with the outer diameter of 310 mm, inner diameter of 300 mm, and height of 350 mm. A diameter of an anode was constant through the whole sets of the experiments, while a diameter of a cathode was varied in order to obtain a stable discharge at a specified oxygen pressure. The chamber was filled with oxygen, and the pressure was maintained in the range of 160 to 2000 Pa. The first set of the experiments was caried out for the samples installed on the anode, so the samples were exposed to the direct action of plasma passed a relatively low anode voltage drop of a few volts. A schematic of the experimental setup is shown in Figure 1a.

To ignite plasma, a negative potential of a few hundred volts was supplied to the cathode, with respect to the grounded anode and chamber walls. At that a common glow discharge was set with a bright glow region near the cathode and even faint plasma column near the anode, as it is shown in Figure 1b. However, at the high-pressure modes, anode spots were observed. The elements of the copper electrodes exposed to the plasma, undergo quick oxidation exhibited by changing their colour from red to black. After the plasma oxidation, the samples were left in the chamber for 30 min, and then were passed to the scanning electron microscope (SEM) to study the effect of the plasma treatment to the surface of the sample.

3. Research results and their analysis

Figure 2 shows SEM images of a sample treated in plasma for 1 hour in oxygen maintained at the pressure of 260 Pa. The sample with a diameter of 8 mm and a height of 5 mm was installed on the anode separated from the cathode (diameter of the cathode was 20 mm) by a gap of 11 mm. The glow discharge was sustained at the voltage drop of 580 V between the electrodes, and the discharge current of 0.05 A. The sample was cooled at the same pressure for 30 min after the plasma treatment.

The investigation with the use of SEM technique revealed the following results. The whole surface of the copper oxide formed on the sample after its oxidation exhibited 3D nanostructures found on the microstructures, that are associated with a peeled off area of the copper oxide layer (Fig. 2a), which was confirmed by the magnified view of the microstructures in Figure 2b and Figure 2c. It was found that 3D nanostructures are composed of 2D nanostructures with the size of 200-400 nm stacked to form the 3D nanostructures, as it can be seen in Figure 2d.

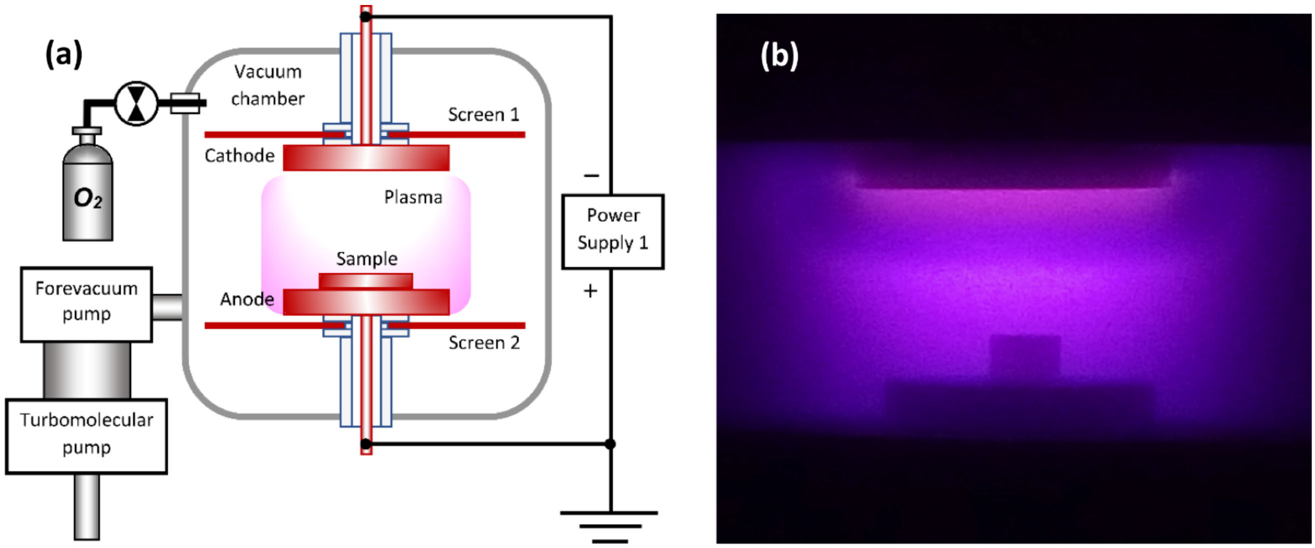

Fig. 1. Growth of copper oxide nanostructures in glow discharge: a) a schematic of the experimental setup, when a copper sample is installed on the anode; b) a photograph of the glow discharge

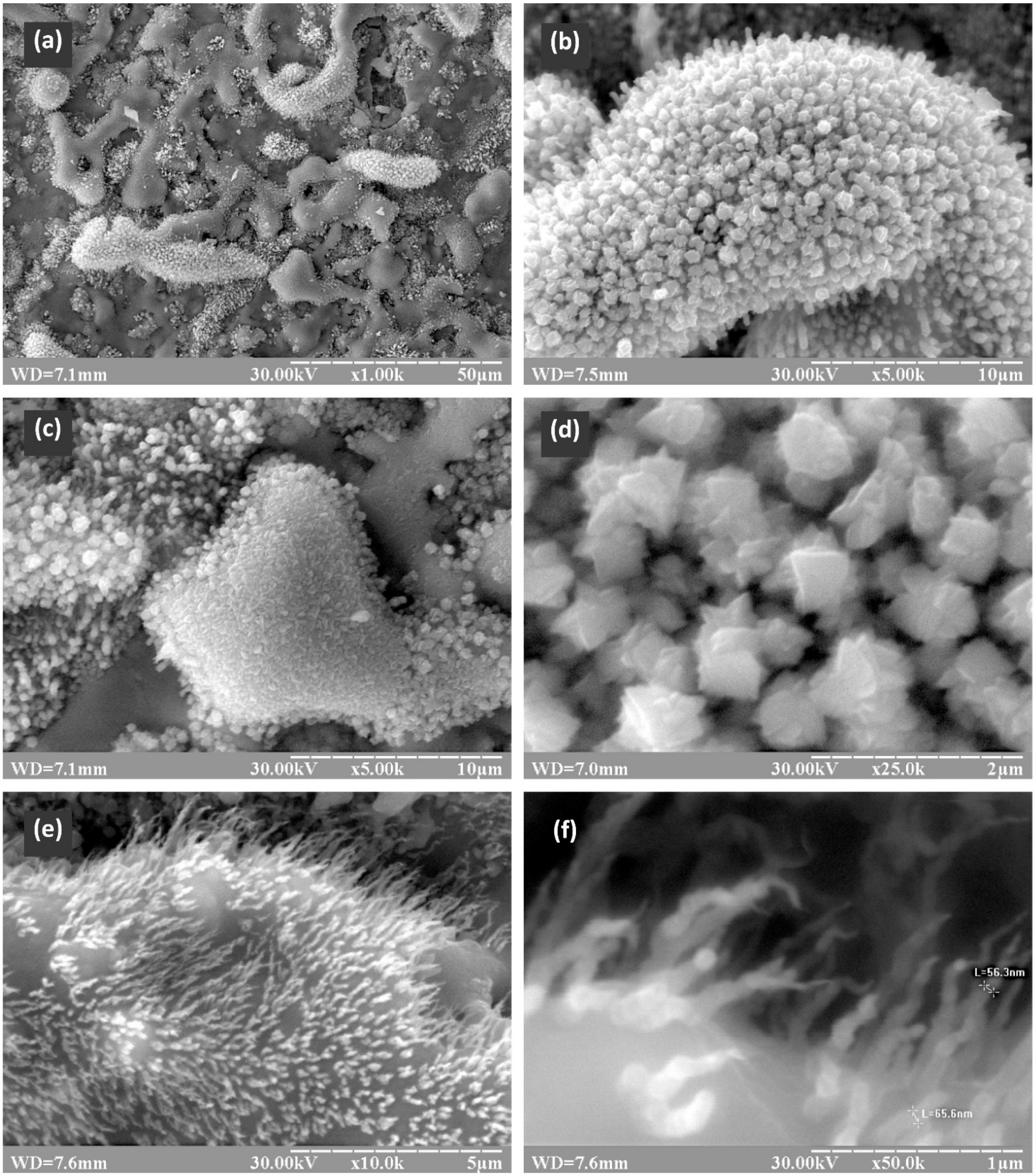

Fig. 2. SEM images of a sample treated on the anode for 1 h in oxygen plasma (260 Pa, 580 V and 0.05 A): a) CuO microstructures covered by the nanostructures; b) magnified view of 3D nanostructures; c) it can be seen that 3D nanostructures are associated with the microbabbles on CuO layer; d) the nanostructures are stacks of 2D nanostructures; e) 1D nanostructures (nanowires) found on some CuO bubbles; f) magnified view of the nanowires with the sizes of about 1 µm in length and 60 nm in diameter

However, the diversity of the nanostructures was not limited by the 2D and 3D nanostructures, since 1D nanostructures (nanowires) were also found. The sizes of the nanowires are about 1 μm in length and 60 nm in diameter (Fig. 2e,f). The results allow suggesting two different mechanisms of nanostructure growth. Stacked 3D nano-structures were formed as a set of growth steps, when a defect of a certain size was generated on a side surface of 2D nanocrystal exposed to the ion flux from plasma. The defect served as a seed for the formation of the next 2D nanocrystal. Opposite to that, the nanowires (1D) were formed from a seed formed at the beginning of the growth process. The nature of the seed is a discussion issue yet the most probable assumption associates them with the grains of copper oxide that started anisotropic growth under the action of the surface stress developed in the oxide layer at the quick oxidation.

The results of the exposure to the glow plasma of the same size samples, and treated for 1 h in the same setup yet at a pressure of 490 Pa are shown in Figure 3. The discharge current and voltage were 0.08 A and 600 V, respectively. Although in this experiment the sample was not heated additionally, the appearance of the nanostructures changed drastically.

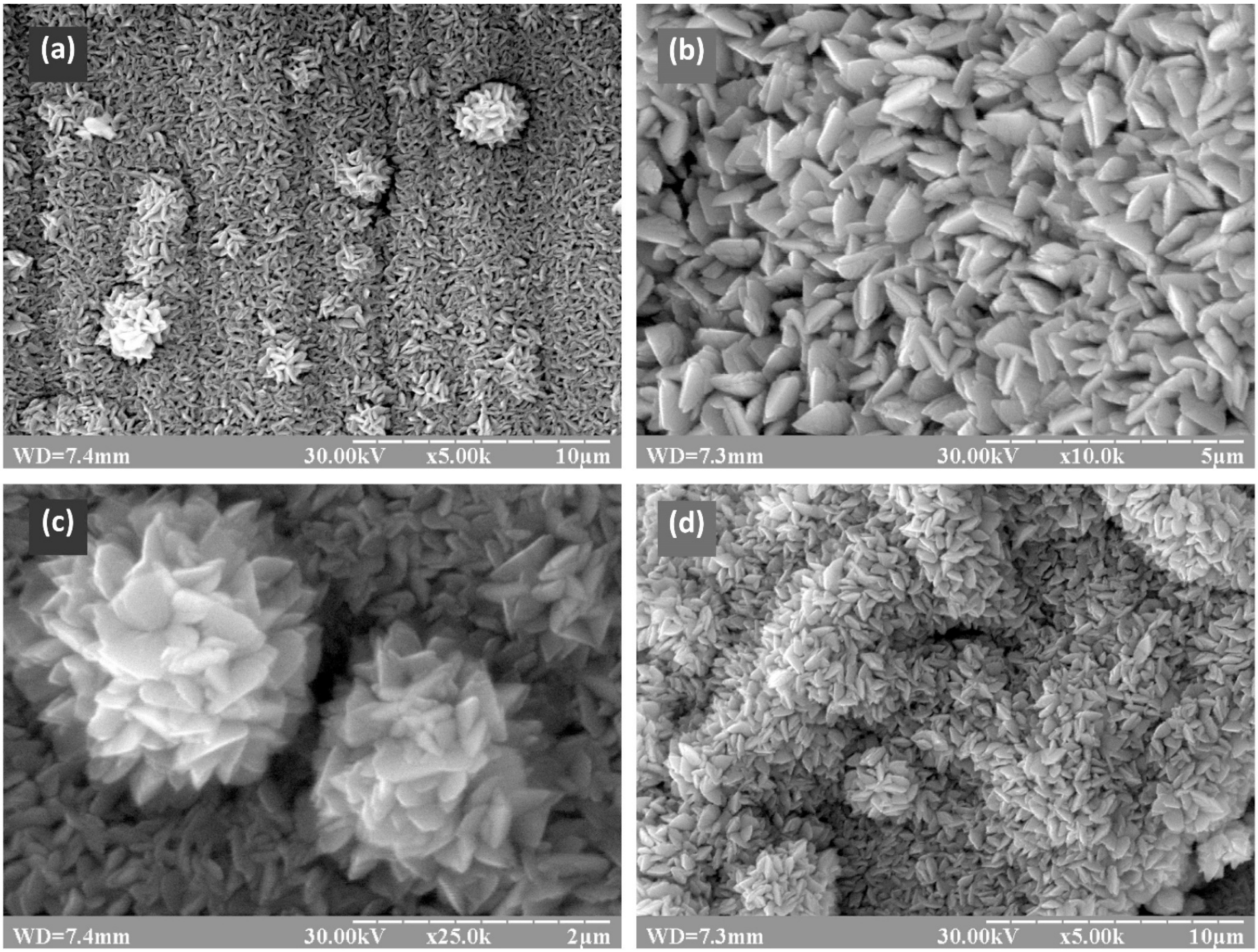

Fig. 3. SEM images of a sample treated on anode for 1 h in oxygen plasma (490 Pa, 600 V and 0.08 A): a) general view of a surface covered with the nanostructures; b) magnified view of the 2D nanostructures with the sizes of about 500×100 nm; c) magnified view of the flower-like microstructures composed of the disk-shaped nanostructures; d) fragment of the surface showing the different morphologies composed of the same nanostructures

A regular microscale relief of the surface was observed, and it was associated with a radial distribution of the internal stress supposedly generated in the oxide layer, which was followed by the formation of the waves (Fig. 3a). In general, the relative increase in the discharge power of 48 W accompanied by the pressure increase resulted in formation of a uniform structure composed of 2D nanograins (Fig. 3b). Among them, nanoflowers (Fig. 3c) can be distinguished, formation mechanism of which is not clear. In contrast, the bulky nanostructures observed in Figure 3d can be explained by the oxide layer distortion caused by the surface stress.

Further decrease of the pressure to 150 Pa followed by the current decrease to 0.04 A (the voltage remained unchanged, i.e., 600 V) leads to the changes shown in Figure 4 (time of treatment was 30 min). In this mode, the discharge power is low (24 W), and only sparse nanostructures are grown. A typical one is an anemone-like nanostructure (Fig. 4a), whose magnified image reveals the sizes of the nanowires composing the nanostructure: of 1 μm in length and 100 nm in diameter (Fig. 4b). Among them, the smaller nanowires are distinguished, like shown in Fig. 4c, where the diameter is about 50 nm. Unfortunately, the magnified view of the nanowires only suggests more complicated structure of them (Fig. 4d). It may be speculated that they also can be composed of the stacked nanodisks, that is mentioned above.

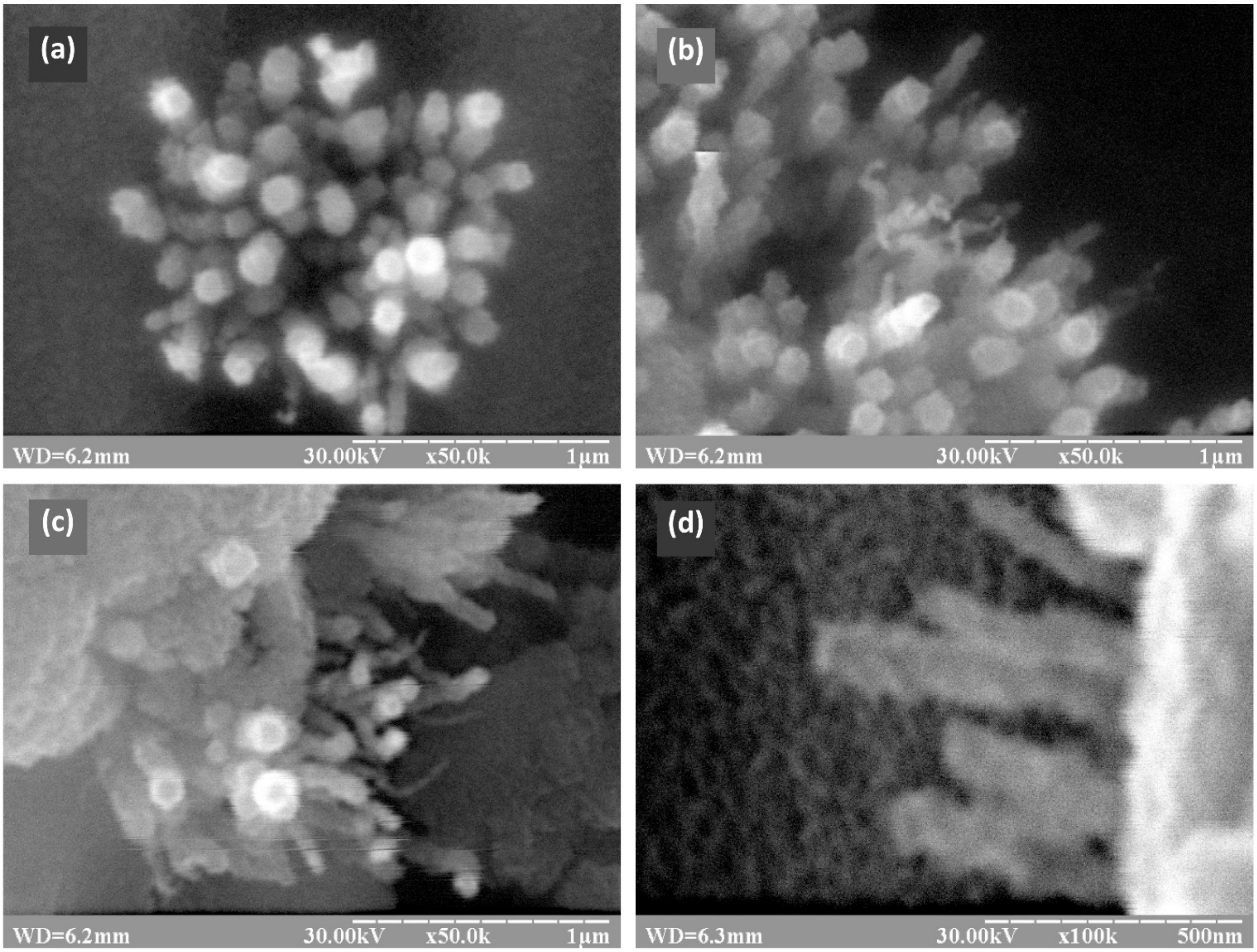

Fig. 4. SEM images of a sample treated on anode for 30 min in oxygen plasma (150 Pa, 600 V and 0.04 A): a) anemone-like nanostructure composed of nanowires; b) magnified view revealing the sizes of 1 μm in length and 100 nm in diameter; c) same nanostructures but with a diameter of 50 nm; d) magnified view of the nanowires

4. Conclusions

Glow discharge was successfully applied to grow reach and diverse yield of copper oxide nanostructures in the dependence on the operation mode of the plasma reactor. Arrangement of the samples on the anode allowed excluding the negative effects of the ion bombardment intrinsic for the cathode layout, thus confirming the perspectivity of the proposed plasma setup for the nanotechnology purposes. CuO nanowires found in the experiment, differ radically from 1D nanostructures observed usually in thermal processes, since they possess much more curved morphology and thinner appearance. Moreover, complex structures with the developed coverage of the nanowires with smaller nanostructures are also obtainable. Nanodisks, nano-flowers, and anemone-like nanostructure grown by use of the setup are another kind of nanostructures that can be grown into a nanoassembly. Thus, the anodic growth of the copper oxide nanostructures is proved to be a useful tool completing the thermal growth in order to expand the nomenclature of the nanostructures [39].

In future, one more important problem should be solved with respect to the growth of copper oxide nanostructures, namely, weak adhesion with copper substrate of the oxide layer that incorporates the nanostructures. It was found in a large number of experiments, that the appearance of the nanostructures can be controlled by changing not only the growth parameters like surface temperature, oxygen pressure, plasma density and so on, but changing the copper sample sizes as well. Thus, the growth modes cannot be considered as independent on the sample shape, which means that the additional control parameter to split the dependence on the operational modes and the sample sizes should be found. This task is considered by the authors as a challenge for their future research.

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