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PLASMA-ENHANCED THERMAL GROWTH OF COPPER OXIDE NANOSTRUCTURES ON ANODE OF GLOW DISCHARGE SETUP

Plasma-enhanced growth of copper oxide nanostructures is widely explored in science and manufacturing, since it provides the flexibility, productivity, and cost-effectiveness necessary to meet the growing demands of customers. However, in the field of growth of metal oxide nanostructures, thermal methods still prevail in plasma methods in spite of long production time up to ten hours. Radiofrequency and microwave plasma sources were applied to grow CuO nanostructures, which are of high interest in various branches of industry, and allowed obtaining a large variety of the nanostructures, and nanowires in particular. At that, high price of the equipment limits the implementation of the results and urges to find cheaper plasma-enhanced method of growth. For this purpose, a common glow discharge plasma setup was engaged to grow the nanostructures in an oxygen atmosphere on surfaces of samples installed on the anode of the electric circuit designed to sustain the glow discharge. An additional heater was mounted under the anode. The proposed combination allowed conducting the growth process under conditions of the delivery of the necessary heat flux and removal the excessive ion flux that can destroy the growing nanostructures because of sputtering. In the first set of experiments, the additional heater was not used, and the observed nanostructures were presented by grains (2D) of about 370 nm in diameter and 80 nm in thickness. This structure is supposedly formed because of action of the internal stresses in the oxide layer. After turning on the heater, the nanowires (1D) were the only nanostructures observed in the experiment, and since no nanowires were found in a case of heating the anode without plasma ignition, one can consider the plasma as a factor determining the nanowire growth.

Keywords: *plasma; glow discharge; copper oxide; nanotechnology; oxide nanostructures.*

Introduction

Metal oxide nanomaterials are considered as promising material in the scientific community due to their outstanding size and morphology dependent properties. Among the nanomaterials, copper (II) oxide nanostructures are the most studied; at that, 1D nanostructures called nanowires, are of considerable interest. The reasons are explained by their high surface-to-volume ratio and perfect crystallinity, which were useful in catalysis [1] and photocatalysis [2-4], field emission [5], optoelectronics [6], gas sensing [7-9], and energy conversion [10] application, to mention but a few. Among various methods of synthesis [11], thermal growth appears to be the most employed as it follows from the literature analysis. Large number of experimental and theoretical results is reported on this topic yet the growth mechanisms are not clear up to now. Copper oxide nanowires usually exhibit bi-crystalline structure with twin boundary, however single crystalline nanowires have also been reported [12]. At it follows from the reports, there are different temperatures at which the nanowires grow, but most commonly nanowires are grown between 400 ºC and 700 ºC [13] at atmospheric pressure.

A mechanism of the growth is another controversial issue, and the only indisputable statement is about the growth of the nanowires on their tops [14–16]. However, two main assumptions on copper diffusion to the top prevail in the reports: (i) diffusion, where Cu ions diffuse along the side surface of a nanowire [14] and (ii) diffusion through twin boundary [15, 16]. The later fact was observed by *in-situ* TEM study [16]. Concentration of oxygen in the surrounding gas phase was also found to play a critical role in the growth, the optimal concentration depends on the temperature in the processing chamber. Initial roughness of the copper substrate is another factor influencing the density of the nanowire arrays [17] with higher densities of nanowires grown on rougher surfaces.

Despite of the facts that thermal methods were the tool to collect the abundant database on the nanowire formation, high cost-efficiency and simplicity, their practical implementation is limited by the treatment time that usually reaches the values between 5 to 10 hours. The necessity to decrease the time lap in order to increase the total productivity of the equipment urges engineers and scientists to search other possibilities. Chemical [18], thermo-chemical [19], and plasma-enhanced [20] methods were developed as a result of this search. Filipič *et al.* reported [21] about the growth of CuO nanowires in a reactor provided with inductively coupled (ICP) plasma source, while Altaweel *et al.* suc-

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cessfully applied micro-afterglow of microwave (MW) plasma for the purpose [22]. However, these technologies imply the necessity of using the expensive equipment like ICP and MW plasma sources; the cost can be as high as 20,000 euro per source. That is why the search for the new methods of the nanowire growth is still pressing.

Formulation of the problem

Plasma-based, or plasma-enhanced methods are widely applied in modern industry to sputter, etch, modify a surface or deposit a layer of new material on it [23]. High flexibility, productivity, and controllability of these methods are explained by the high chemical activity of a matter in plasma state, since plasma is composed on ions, electrons, excited molecules, radicals and other species. At that, unlike the matter in neutral charge state, plasma can be a subject to electromagnetic control [24] to create wide or focused fluxes, or control the ion-to-neutral ratio in the fluxes [25, 26]. The chemical activity is also enhanced by the functionalization of the treated surface caused by ion bombardment, when large number of defects like vacancies and dislocations is generated on the surface and changes the energies of adsorption of the species from the plasma, as well as the energies of their diffusion and association [27]. However, the ion bombardment causes also the negative effects when dealing with the growth of surface structures, since the defects are generated in the lattice of the growing structure, and, generally, the nanostructure can be wiped out by the ion flux.

That is why in this research the copper samples were put on the anodic side of the electric circuit responsible for the glow discharge ignition. At that, the anode was heated by use of a heater to provide the additional heat flux to the growing nanostructures. The main idea was to combine the heat flux with the fluxes of charged particles extracted from the remoted plasma whose highest density region was located near the cathode.

Experimental part

To perform the experiments on the growth of copper oxide nanostructures, a plasma reactor utilizing the glow discharge plasma was used. The diameters of the anode and cathode were 20 mm, the discharge gap was set to 16 mm. Copper samples with a diameter of 8 mm and a height of 5 mm were put on the anode and exposed to the action of plasma with ions passed a relatively low anode voltage drop of a few volts. The electrodes were installed in the cylindrical vacuum chamber with the outer diameter 310 mm, inner diameter 300 mm, and height of 350 mm. The chamber was filled with oxygen, and the pressure was maintained in the range of 160 to 460 Pa. A schematic of the experimental setup is shown in Fig. 1.

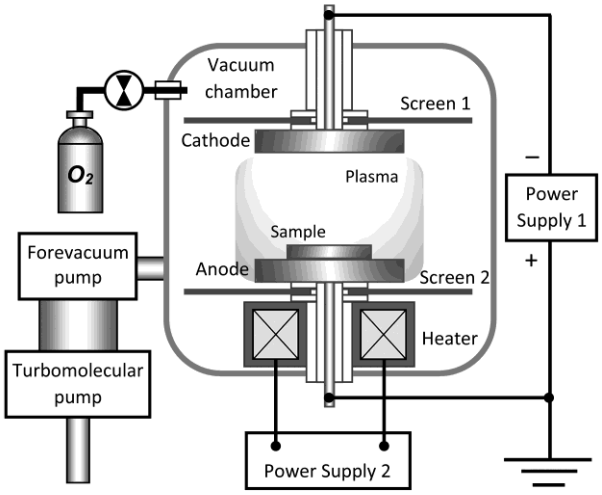

Fig. 1. A schematic of the experimental setup

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 Fig. 2.

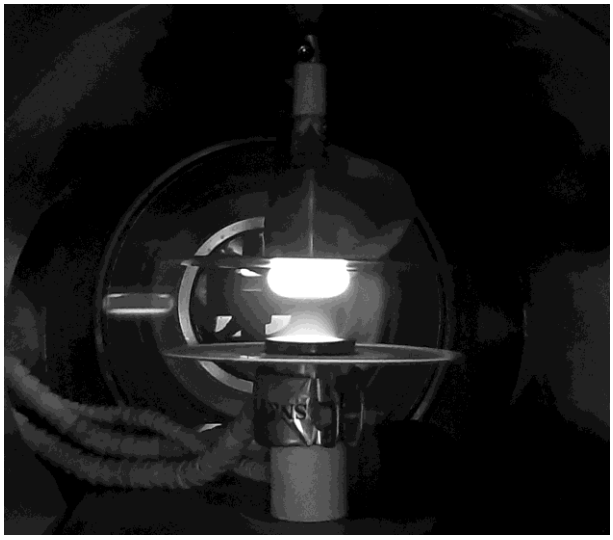

Fig. 2. A photograph of the plasma discharge

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 stayed in the chamber for 30 min, and then were passed to the scanning elec-

tron microscopy (SEM) to study the effect of the plasma treatment to the surface of the sample.

Results and discussion

In the first set of experiments, the anode was not heated additionally. Fig. 3 shows SEM images of a sample exposed for 1 h to the action of the glow discharge maintained at the pressure of 240 Pa. The discharge voltage and current were 590 V of 0.06 A, respectively, and the discharge power was 36 W. In this experiment, the nanostructure was presented by the grains (2D) of about 370 nm in diameter and 80 nm in thickness. This structure is supposedly formed as a result of the internal stresses in the oxide layer.

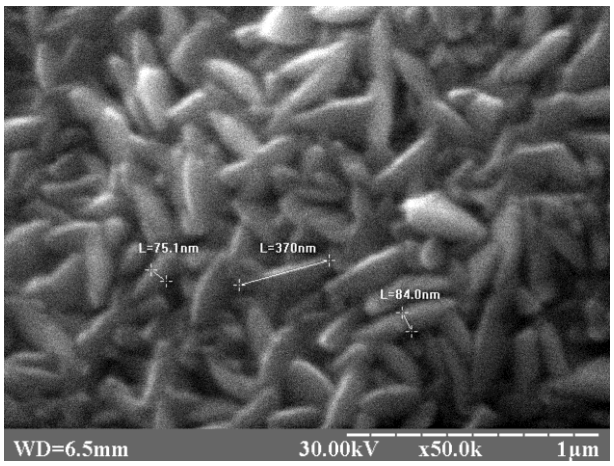

Fig. 3. SEM image of a sample treated on anode for 1 h in oxygen plasma (240 Pa, 590 V and 0.06 A). Here the nanostructures are presented by the 2D grains with the sizes of about 370×80 nm

During the next stage of the research, the second set of experiments was conducted in the conditions similar to those used before yet the anode was additionally heated. For the purpose, a heater shown in Fig. 1 was used for the whole time of the plasma treatment. The temperature of the sample measured at the ambient air conditions without plasma action was 160 °C. Fig. 4, a illustrates the results obtained for a sample treated in plasma for 2 hours in oxygen maintained at the pressure of 160 Pa. The glow discharge was sustained at a voltage drop of 600 V between the electrodes, and the discharge current of 0.06 A. The sample was cooled at the same pressure for 30 min after the plasma treatment.

As it can be seen, even at such low discharge power (36 W) the additional heating drastically changed the growth process, since the nanowires (1D) are the only nanostructures observed in the experiment (Fig. 4, b). It should be mentioned that no nanowires were found in a case of just heating the anode without plasma ignition, so one can consider the plasma as a factor determining the nanowire growth.

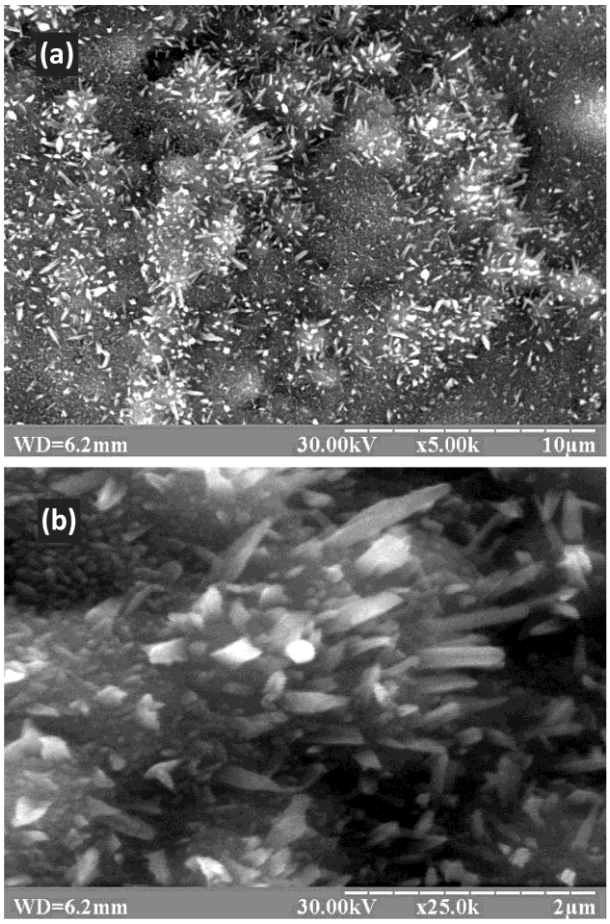

Fig. 4. SEM images of a sample treated on a heated anode for 2 h in oxygen plasma (160 Pa, 600 V and 0.06 A): a – copper oxide bubbles covered by 1D nanowires; b – magnified view of the nanowires showing their sizes of about 1 μm in length and 100 nm in diameter

Next sample studied by use of SEM (Fig. 5) was additionally heated during the plasma treatment for 30 min (the temperature of the sample measured preliminary in ambient air was 160 °). The voltage drop and the discharge current were 600 V and 0.1 A, respectively, at the oxygen pressure of 460 Pa.

In this experiment, the additional resistive heating also resulted in formation of short nanowires while they were not observed at the same heating mode without plasma (Fig. 5, a). Thus, the plasma treatment with the discharge power of 60 W is beneficial for the nanowire growth. A cylindrical shape and small length of the nanowires speaks in favour of this conclusion, since the thermally-grown nanowires usually exhibit the shape of the uniformly long and thin needles, or relatively short nanocones. The magnified view demonstrates rather

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short nanowires of 1 μm in length and 150 nm in diameter, as shown in Fig. 5, b. SEM observation confirm that the area of the buckling of the copper oxide promotes the growth of the nanowires (Fig. 5, c). Although the nanowires are not long, their density is large – of about 20 μm^{-2}.

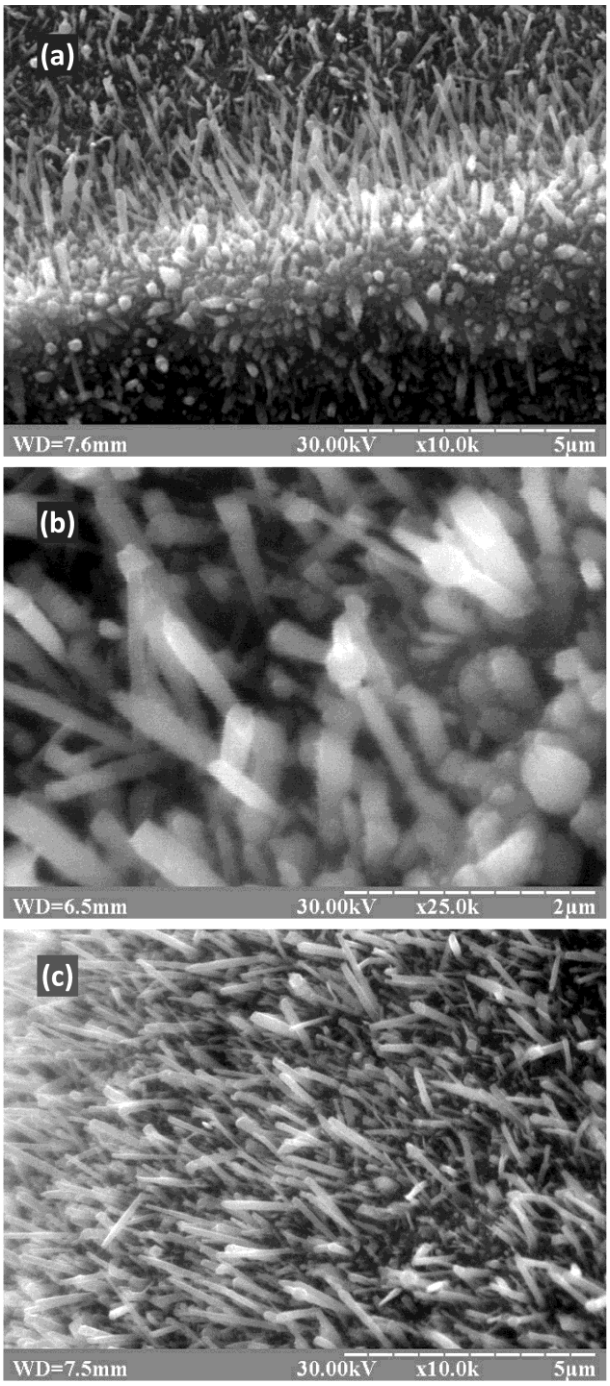

Fig. 5. SEM images of a sample treated on a heated anode for 30 min in oxygen plasma (460 Pa, 600 V and 0.1 A): a – nanowires of CuO grown on the surface; b – magnified view of the nanowires with a length of 1 μm and diameter of 150 nm; c – abundant yield of the nanowires with the density of about 20 μm^{-2}

The same conditions were applied for the next sample (30 min, additional heating of the anode, 600 V) yet at the decreased oxygen pressure of 300 Pa, which resulted in the current decrease to 0.065 A. The morphology of the surface structures is shown in Fig. 6.

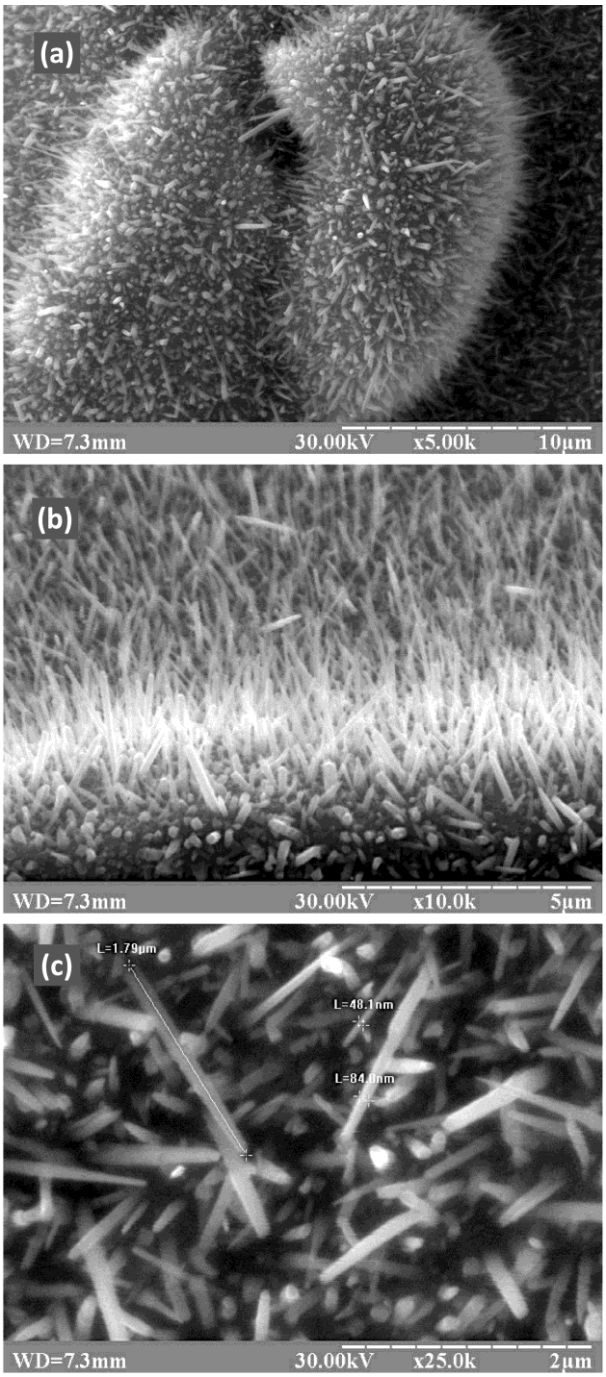

Fig. 6. SEM images of a sample treated on a heated anode for 30 min in oxygen plasma (300 Pa, 600 V and 0.065 A): a – CuO nanowires grown on the treated surface; b – grass-like dense array of the nanowires; c –magnified view of the nanowires with a length of about 2 μm and diameter of about 60 nm; the density of the nanowires is about 10 μm^{-2}

At that the nanowires found on the surface (Fig. 6, a) are also produced in a large number yet their appearance more corresponds to the thermally-grown nanostructures (Fig. 6, b), since their average length reaches the value of 2 μm at the diameter of 100-200 nm. Among them, even thinner nanowires can be observed as it is shown in magnified view of the nanowires with a length of about 2 μm and diameter of about 60 nm; the density of the nanowires is about 10 μm^{-2} (Fig. 6, c).

Application perspectives

Up to now, the density of arrays of copper oxide nanowires grown in plasma, was reported to be about 1 μm^{-1}. At that, rather expensive equipment was employed like inductively coupled or microwave plasma sources. In this study the density of the arrays reached the value of about 20 μm^{-2}, so it increased by 20 times, and the applied technique, namely glow discharge plasma setup, can be considered as very cost-efficient solution. By taking into account the fact that the glow discharge implies an even distribution of plasma along a treated surface, its possible application is beyond any doubts. However, an introduction of a magnetic field in the discharge gap can be beneficial in obtaining a separate control of the discharge current and voltage, which, in turn, can expand the application perspectives of the proposed method.

Conclusions

In this paper a method to grow an abundant yield of copper oxide nanowires is reported. Plasma-enhanced thermal growth of the nanowires on samples mounted on an anode of a glow discharge plasma setup proved to be a reliable, flexible and highly productive tool to increase the productivity of the growth process by about 10 times, as compared with common thermal method where the duration of the process is about 5 to 10 hours. In addition, the density of the nanowires on the sample surfaces was twenty-fold increased with respect to the results shown by use of radiofrequency and microwave discharges.

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СТИМУЛЬОВАНЕ ПЛАЗМОЮ ТЕРМІЧНЕ ЗРОСТАННЯ НАНОСТРУКТУР ОКСИДУ МІДІ НА АНОДІ ПРИСТРОЮ ДЛЯ ТЛІЮЧОГО РОЗРЯДУ

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Зростання наноструктур оксиду міді за допомогою плазми широко досліджується в науці та виробництві, оскільки воно забезпечує гнучкість, продуктивність та економічну ефективність, необхідні для задоволення зростаючих запитів клієнтів. Проте в області вирощування наноструктур оксидів металів термічні методи все ще переважають над плазмовими, незважаючи на дуже тривалий час виробництва – до десяти годин. Радіочастотні та мікрохвильові джерела плазми були застосовані для вирощування наноструктур CuO, які представляють великий інтерес у різних галузях промисловості, та дозволили отримати велику різноманітність наноструктур, зокрема нанодротів. При цьому висока ціна обладнання обмежує впровадження результатів і спонукає до пошуку дешевшого плазмового методу вирощування. В роботі була задіяна звичайна плазмова установка тліючого розряду для вирощування наноструктур в атмосфері кисню на поверхнях зразків, встановлених на аноді електричного кола, призначеного для підтримки тліючого розряду. Під анодом був встановлений додатковий нагрівач. Запропонована комбінація дозволила проводити процес росту за умови постачання необхідного теплового потоку та видалення надлишкового потоку іонів, який може руйнувати зростаючі наноструктури в результаті розпилення. У першій серії експериментів додатковий нагрівач не використовувався, а спостережувані наноструктури були представлені зернами (2D) діаметром близько 370 нм і товщиною 80 нм. Імовірно, ця структура утворюється в результаті дії внутрішніх напружень в оксидному шарі. Після включення нагрівача нанодроти (1D) були єдиними наноструктурами, які спостерігалися в експерименті, і оскільки нанодроти не були знайдені у випадку простого нагрівання анода без плазмового запалювання, можна розглядати плазму як фактор, що визначає зростання нанодроту.

Ключові слова: плазма; тліючий розряд; оксид міді; нанотехнологія; оксидні наноструктури.

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СТИМУЛИРОВАННЫЙ ПЛАЗМОЙ РОСТ НАНОСТРУКТУР ОКСИДА МЕДИ НА АНОДЕ УСТРОЙСТВА ДЛЯ ТЛЕЮЩЕГО РАЗРЯДА

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Рост наноструктур оксида меди с помощью плазмы широко исследуется в науке и производстве, поскольку он обеспечивает гибкость, производительность и экономическую эффективность, необходимые для удовлетворения растущих потребностей клиентов. Однако в области выращивания наноструктур оксидов металлов термические методы все еще преобладают над плазменными, несмотря на очень долгое время производства – до десяти часов. Радиочастотные и микроволновые источники плазмы были применены для выращивания наноструктур CuO, которые представляют большой интерес в различных отраслях промышленности и позволили получить широкий спектр наноструктур, в частности нанопроволоку. При этом высокая цена оборудования ограничивает внедрение результатов и побуждает искать более дешевый плазменный метод выращивания. В работе использовалась обычная плазменная установка тлеющего разряда для выращивания наноструктур в атмосфере кислорода на поверхностях образцов, установленных на аноде электрической цепи, предназначенной для поддержания тлеющего разряда. Под анодом был установлен дополнительный обогреватель. Предложенная комбинация позволила провести процесс роста при условии подачи необходимого теплового потока и удаления избыточного потока ионов, способного разрушить растущие наноструктуры в результате распыления. В первой серии экспериментов дополнительный нагреватель не использовался, и наблюдаемые наноструктуры были представлены зернами (2D) диаметром около 370 нм и толщиной 80 нм. Предполагается, что эта структура образуется в результате действия внутренних напряжений в оксидном слое. После включения нагревателя нанопроволоки (1D) были единственными наноструктурами, наблюдаемыми в эксперименте, и, поскольку нанопроволоки не были обнаружены в случае простого нагрева анода без поддержания плазмы, можно рассматривать плазму как фактор, определяющий рост нанопроволоки.

Ключевые слова: плазма; тлеющий разряд; оксид меди; нанотехнология; оксидные наноструктуры.

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