Electrochemical Methods for 3D Cu Current Collectors Formation

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Copper current collectors (Cu CCs) impact the production technology and performance of many electrochemical devices by their unique properties and reliable operation. The efficiency of the related processes and the operation of the electrochemical devices could be significantly improved by optimization of the Cu CCs. Metallic Cu plays an important role in electrochemical energy storage and electrocatalysis, primarily as a conducting substrate on which the chemical processes take place. Li nucleation and growth can be influenced by the current collector by modulating the local current density and Li ion transport.

copper current collector Li ion battery

1. Template-Assisted Methods

Templating is one of the key methodologies for the controlled fabrication of micro- and nanostructured materials. This approach relies on formation/synthesis or assembly of a pre-existing pattern (template) with desired nanoscale features (first step). The role of this pattern is to direct the deposition of nanomaterials into forms that are otherwise difficult or impossible to obtain (second step). Finally, to obtain the desired structured Cu array, the applied template has to be removed (third step). In the following subsections, template-assisted approaches for Cu deposition relying on different types of template nature and formation are presented.

1.1. AAO Templates

An anodic aluminum oxide (AAO) template is a self-organizing porous anodic layer of Al_2O_3 that typically has a honeycomb-like structure of pores aligned in parallel to forming density arrays with a high number of pores. Among all templates, AAO layers are the most widely applied for nanowire growth. When using AAO templates, the nanowire diameter can be precisely determined by the AAO pore size [1]. The pore diameter can be adjusted in the range of five to several hundred nm, and the AAO thickness, which determines the nanowire length, can be controlled from a few tens of nanometers to a few hundred micrometers. Porous AAO templates are formed electrochemically by anodization of Al, typically in acid electrolytes, as reported by Masuda and Fukuda [2] and numerous later researchers [3]. Owing to their ordered uniform nanopores, AAO templates have been widely used in nanotechnology for the deposition of metallic and semiconductor nanowire arrays [3][4].

The first reports on the electrochemical deposition of Cu in AAOs date back to the early 2000s [5], when the application of AAO-grown Cu nanowires as field emitters was first demonstrated [5]. The early demonstration of this technique was followed by many studies of AAO-templated Cu CC electrodeposition, mainly for electronics [6], electrocatalytic purposes [7], and energy storage applications [4].

AAO nanoporous templates with different pore diameters and interpore distances can be easily grown by potentiostatic anodization of aluminum carried out in acidic electrolytes. In particular, electrolyte solutions based on sulfuric acid, [8], oxalic acid [2][9][10], or phosphoric acid [11] can be applied. The geometry of the obtained AAO membranes strongly depends on electrochemical conditions, i.e., on parameters such as anodization voltage, electrolyte composition, and anodization time [12]. Generally, the AAO pore diameter and the interpore distance depend linearly on the applied potential/voltage.

Barrier layer thickness is one of the most important AAO parameters; in order to realize nanowire electrodeposition, either its thickness has to be reduced or the entire barrier layer has to be removed. The AAO wall thickness and barrier layer thickness can be modified during the post-treatment process, which usually involves a chemical etching step [12].

The AAO-based strategies for the fabrication of Cu nanowire arrays can be generally categorized into three main groups [11]. In the first approach, a thin layer of conductive metal is deposited on through-hole AAO membranes, followed by galvanostatic Cu electrodeposition [13]. However, to obtain a nanoporous AAO layer with open-ended channels, the remaining Al metal has to be removed, and in certain cases an extrapore widening step is necessary. The second method consists of alternating-current (AC) Cu electrodeposition inside the AAO pores, and does not require any additional processing after Al anodization [14][15]. This technique allows for the growth of large quantities of Cu nanowires [15]. The third interesting approach applies a through-hole AAO template as a direct mask for the electrodeposition of Cu nanowire arrays [4][16]. The main advantage of this method is its simple manufacturing process. Nevertheless, this strategy relies on strong adhesion of the AAO template to the surface of the Cu substrate [16].

In nearly all scientific works in the field, $CuSO_4$ is the Cu source of the electrolyte composition and Cu electrodeposition in acidic and alkaline electrolytes is possible. Acid electrolytes are frequently used for this purpose due to their high efficiency and the higher current density that can be applied when using these solutions. Their main drawback is their low throwing power and the pronounced corrosion caused by the more chemically active metals in these media (e.g., Zn, Fe, Sn, etc.). Alkaline copper electrolytes display higher throwing power than acidic ones; they are less corrosive, and as such are especially important for the industrial plating of steel and other non-inert substrates. Nevertheless, the high toxicity and corresponding environmental issues of the most frequently used alkaline cyanide electrolytes motivate the development of less toxic alternative solutions based on other complexing agents such as sorbitol, glycine, glycerol, glutamate, and pyrophosphate [17]. Copper electrodeposition in AAOs is performed mainly in two electrode cells using different cell geometries. Furthermore, a variety of electrochemical procedures are used for deposition of Cu in the AAO nanopores, including DC galvanostatic and potentiostatic deposition and different AC (pulse) techniques. The specific cell and electrode geometries and the application of different substrates, procedures, and electrolytes make comparative analysis of the results reported in the literature difficult.

1.2. Track-Etched Polymer Membrane Templates

Track Etched Membranes (TEM) are precisely fabricated polymer layers, with multiple applications in life sciences, engineering, and nanotechnology. These functional materials can be produced with a pre-defined pore diameter, with each pore being able to form a single channel throughout the entire membrane thickness. The TEM can have a film thickness from 6 to 50 µm for different polymer materials, including polycarbonate (PC), polyethylene terephthalate (PET), polyvinylidene difluoride (PVDF), polypropylene (PP), and polyimide (PI). Among all polymer types, PC and PET are the most studied materials for application of TEM in nanotechnology. In particular, PC dominates research work in the field of metallic nanowire deposition. The process of TEM fabrication involves two steps, ion tracking and chemical etching [18]; the fabrication technique consists of the formation of continuous trails in the material by exposure to radiation (first step) and the formation of fine holes by preferentially dissolving the damaged trails in an appropriate etching bath (second step). These holes can be chemically enlarged by regulating the etching time until the necessary size is achieved [18][19]. There are two radiation-based methods for the formation of tracks on polymer films: (1) bombardment with heavy particles (e.g., alfa radiation), and (2) use of high-energy ion beams from accelerators (e.g., GSI, Darmstadt, Germany, and GANIL, Caen, France) [19]. Etching of the polymer membrane is easily achieved in alkaline solutions such as KOH or NaOH. This allows the pore size to be enlarged and chemical moieties to be added to the inner pore surface. PC and PET membranes have similar properties; however, PC is more soluble in organic solvents and more suitable for producing membranes with cylindrical nanopores, as the pore size is very homogeneous and smooth [19].

Similar to AAO, PC membranes with relatively narrow pore diameters (close to 10 nm) are commercially available; furthermore, they can be used as a template to grow metal nanowire arrays. Nevertheless, as discussed by Motoyama et al. [20], the geometrical periodicity of nanowire arrays is low compared to that of AAOs due to random hole formation during PC irradiation with heavy ion particles. Furthermore, the same authors observed the nonuniformity of nanowires along the length direction, which has been attributed to the original irregular shape of the nanopore wall in PC membranes after track-etching with heavy ions. In this context, a broader pore size distribution was investigated earlier by Chlebny et al. [21]. They concluded that electrochemical nanowire formation using such a template can be applied in fields such as energy conversion and storage where highly ordered arrays are not required [20].

1.3. Particle Templates

Another template approach is based on the initial assembly of spherical particles on the substrate surface, followed by Cu electrodeposition and subsequent removal of the initially assembled template structure. This so-called colloid assembling technique has attracted many research groups owing to its easy and low-cost formation of 3D macroporous layers [22][23]. Liquid–air interfaces have been used for the self-assembly of colloidal particles with a diameter of several hundred nanometres, finally resulting in the formation of regularly ordered large particle arrays. When performing template self-assembly, PS or SiO_2 sub-micron-sized spheres are typically used. During self-assembly, capillary forces trigger the formation of a dry colloidal fcc crystal resulting in a structure called artificial opal, and the material deposited in the interstitial spaces is named inverse opal [22].

Following this approach, porous metallic structures can be formed through electrodeposition in self-assembled PS or other particles. This technique was initially used for gold [24], and later microporous copper material was deposited using the same method [25][26]. Following electrodeposition, the original opal material can be removed. For example, etching of silica opal is performed by immersion in a 5% solution of HF [27] and opal formed of PS can be removed by combustion at 450 °C or by dissolving the PS template in organic solvents, e.g., toluene [26].

The deposition of high-quality 3D porous copper and other porous metal films using this method depends critically on factors that control the permeability of the electrolyte solution in the nanochannels of the PS template. Ding et al. concluded that the permeability can be substantially increased by surface modification of the PS template, which has the aim of enhancing the hydrophilicity of the PS particles [25][26]. Such an effect was realized by modification of the PS surface with SDS in [25].

Another useful approach for mitigation of permeability issues when depositing PS opal is choosing electrolytes with better wetting properties. In this context, Tsai et al. [28] utilized ionic liquid (IL) electrolytes for the electrodeposition of macroporous silver using PS opal templates. The authors observed that their ILs ([BMP][TFSI] and [BMP][DCA]) displayed improved wetting on the hydrophobic PS templates compared to the aqueous electrolytes. This approach has been demonstrated for copper electrodeposition as well. Zein El Abedin et al. [29] reported on the electrodeposition of highly ordered macroporous copper films in 1-butyl-3-methylimidazolium dicyanamide ([BMIm][DCA]) using PS opal templates. PS colloidal spheres were assembled onto gold and indium tin oxide (ITO) substrates, displaying an ordered hexagonal structure. Afterwards, the interstitial spaces of the PS opal arrays were filled with copper by electrodeposition. Well-ordered macroporous copper films have been obtained after the chemical dissolution of the PS template. Furthermore, highly ordered 2Dand 3D macroporous Cu films with interconnected pore structures can be fabricated by varying the experimental conditions.

1.4. Dynamic Hydrogen Bubble Templates

As previously discussed, depending on the experimental conditions electrodeposition can lead to the formation of nanostructures of various geometric forms and dimensions. Dynamic Hydrogen Bubble Technique (DHBT) is a special type of electrodeposition technique that is particularly useful for the formation of 3D interconnected metallic structures (often called metallic foams). DHBT involves two parallel redox processes, namely, electrodeposition and hydrogen evolution reaction (HER), where the hydrogen bubbles act as a dynamic template for the growth of the porous material. The DHBT technique has a number of important practical advantages, including (1) simple growth of porous material without the assistance of a solid template, (2) formation of micron-size layers consisting of nanostructured pore walls with high surface area, (3) an increased number of active sites at the pore walls, which help to accelerate chemical processes and allow easy modification, and (4) high mechanical stability [30].

The DHBT approach has been used for the fabrication of micro- and nanostructured foams across a broad spectrum of single elements, including copper. Among the high number of examples for DHBT-grown metallic foams, copper structures are the most frequently applied as electrode substrates in energy technologies. Owing to their lower hydrogen reduction overpotential, Cu foams can easily be formed and precisely tuned by adjusting

DHBT parameters such as current density, deposition time, electrolyte composition, temperature, and substrate morphology [30].

Both the rate of Cu dendrite growth and the HER are greatly influenced by the applied current density and the corresponding overpotential. Here, the hydrogen bubble nucleation rate and its lifetime on the electrode play an important role. For example, at a lower current density a low number of irregularly distributed H_2 bubbles are generated on the substrate. This results in porous Cu with an irregular morphology. On the other hand, increased current density leads to HER acceleration, and consequently to improved pore regularity and decreased pore size [31].

Deposition time is another important factor that affects Cu pore morphology. It has been shown that the prolongation of deposition time at a constant current density results in enlarged pore diameter. This effect can be explained by the fact that the generated H_2 bubbles participate in physical processes such as collisions, deformation, coalescence, etc., each of which depends on the deposition time and influence the pore geometry [30].

The porous Cu morphology can be greatly influenced by temperature. It has been observed that increasing the electrolyte temperature leads to more intensive hydrogen evolution, resulting in a larger bubble template and an increase in the interstitial distance between the bubbles. Consequently, the pore size of the electrodeposited porous Cu foam increases, although electrolyte temperatures beyond 40 °C have a negative effect on grain size refinement and dendrite stability [31].

Adjusting the electrolyte composition is a useful tool for tuning the morphological properties of DHBT-synthesized Cu foams. Control over branching during Cu electrodeposition has been observed [32][33]; in particular, a reduction in branching has been found after addition of sulphates, chlorides, and bromides. Shin et al. [34] demonstrated that the pore size and wall structure of Cu foams can be changed by varying the electrolyte composition. The same authors observed that the addition of bubble stabilizer (e.g., acetic acid) suppresses the coalescence of H_2 bubbles, resulting in a lowering of their hydrophobic properties and a consequent reduction in pore size. Additionally, they found that the addition of chloride ions to the electrolyte leads to a decrease in branch size in the Cu foam walls as a result of the catalytic effect of chloride. In particular, it was observed that the pore size at the surface was reduced from 50 µm to about 25 µm when 0.1 M acetic acid was added to the electrolyte. A similar effect was achieved after the addition of cetyltrimethylammonium bromide (CTAB) [35]. Additionally, the branches formed on the pore walls were significantly reduced in size after the addition of HCl [34].

The influence of electrolyte components such as NH_4^+, Cl^-, polyethylene glycol (PEG), and 3-mercapto-1-propane sulfonic acid (MPSA) was demonstrated by Kim et al. [36]. Their analysis showed that, among all the additives, NH_4^+ was the most effective suppressant of both Cu deposition and hydrogen evolution, which was due to its adsorption on the electrode surface and the increase in the overpotential of the reactions. This led to a larger pore diameter and lower branching, resulting in mechanically stabilized porous Cu.

An interesting practical approach for the formation of mechanically stable free-standing porous Cu by means of DHBT has been demonstrated by Kurniawan et al. [37]. This methodology consists of three main steps. During the first step, porous Cu structures are electrodeposited galvanostatically on a flat Cu substrate at current densities in the typical range for this purpose (i.e., $j = -0.075$–-2.25 A cm^{-2}) for up to 400 s. In the second step, reinforcement of the obtained Cu dendritic porous structure is performed by applying constant current deposition in the same electrolyte at lower current density (−20 mA cm^{-2}) and longer deposition time (between 5 and 120 min). Because the dendritic reinforcement at the interface between the Cu substrate and porous Cu is less effective, in the third step it is possible to easily detach the porous Cu layer from the substrate by short ultrasonication and finally obtain a stable free-standing porous Cu framework [37].

1.5. Summary

Four different approaches for 3D Cu formation relying on template assistance have been discussed in Section 2.1. These techniques offer final 3D Cu materials with different geometrical forms and dimensions that are strictly controlled by the template geometry. The application of AAO and spherical particle templates can result in the fabrication of ordered Cu structures with near-ideal orientation, i.e., ordered 3D Cu NW arrays and 3D Cu inverse opal, respectively. The utilization of a polymer membrane template enables the formation of Cu NW with random surface distribution and higher diameter variation compared to the AAO approach. One serious challenge in the application of all three solid template approaches is the extra effort necessary for the setting and perfection of the template assembly. In contrast to the solid templates, DHBT does not require additional technological steps to create the template; therefore, this strategy is technologically simpler and more economical. This method allows the formation of 3D-supported or free-standing Cuf layers with micrometer-sized pores that have a disordered spatial distribution and fine surface morphology at the nanoscale, which can be tuned by changing the electrolyte composition. In recent years, simple and low-cost DHBT has gained much attention from the scientific community as a practical approach for manufacturing 3D Cu CCs with high active surface areas, which enable a wide variety of applications.

2. Template-Free Methods

Template-free methods for Cu CC structuring do not rely on applying any technique for shape control of the copper deposits. This group of methods comprises more traditional approaches based on anodic electrochemical procedures, i.e., anodization and electrochemical de-alloying, electropolishing, etc.

2.1. Electrochemical De-Alloying

De-alloying can be generally classified as a corrosion phenomenon, and has attracted high attention due to the possibility of effectively producing nanoporous metals with a three-dimensional ligament-channel structure. Created in this way, the interconnected material displays an enhanced specific surface area, which makes it suitable for a number of applications, similar to the other types of porous metallic structures. Usually, the necessary materials for the processed bimetallic alloys are prepared via the thermal procedure of powder mixture annealing, which may have a mechanical step as well (e.g., milling, hot-rolling, cold rolling, etc.) [38]. An alternative practical way of alloy formation for certain metal couples is electrochemical deposition [39]. The process of alloy formation is followed by a chemical or electrochemical oxidation step, which has the aim of selectively dissolving the domains of the less noble metal phase (i.e., the metallic component with lower standard electrochemical potential).

The idea for electrochemical de-alloying of Cu alloys dates back to the early 1980s [38][40]. Keir et al. investigated the corrosion process of different binary copper alloys in NaCl solution at various fixed anodic potentials. Cu-Mn, Cu-Zn, and Cu-Ni alloys have shown de-alloying behavior, with the selective loss of Mn able to be optimized by regulating the potential at which the alloy is held [38]. Comprehensive corrosion analysis of Cu-Al, Cu-Mn, Cu-Zn, and Cu-Ni alloys was later performed by Pryor et al. [40]. It was shown that the copper dealloying rate of the binary alloys in NaCl solutions at E = −0.25 vs. SHE decreases in the order Cu-Al > Cu-Mn > Cu-Zn > Cu-Ni. The properties of the resulting metallic porous structures have been studied as well. Following these early works on electrochemical de-alloying, many possible Cu binary alloys have been investigated, including Cu-Zn [41][42][43], Cu-Al [44][45], Cu-Zr [46], Cu-Ag [47], and Mg-Cu-Y [48], for effective fabrication of Cu porous materials. The most promising de-alloying results have been obtained for the frequently used copper alloys Cu-Zn and Cu-Al.

The electrochemical de-alloying of Cu-based alloys is usually performed potentiostatically in highly corrosive concentrated chloride [41][42][45][49] or acidic media [46][47]. The chosen de-alloying potential has to be positive enough to selectively dissolve the Cu alloying element (i.e., Al, Mn, Zn, etc.); however, its value and that of the alloy corrosion potential determine the de-alloying rate.

The evolution of the morphology of porous copper surfaces during Cu-Zn de-alloying in 1-ethyl-3-methylimidazolium chloride (EMIC) electrolyte was studied by Lin et al. [41] through SEM imaging. As the de-alloying process began, Zn atoms were selectively removed from the alloy surface, followed by pitting. It was found that the dissolution of Zn from the alloy begins preferentially at the grain boundaries. As de-alloying proceeds, this selective dissolution of Zn leads to Cu enrichment, finally resulting in larger ligaments and increased pore size. A similar morphological pattern has been observed for products obtained in other electrolytes.

2.2. Anodic Treatment of Cu Substrate

Anodic electrochemical treatment comprises a group of methods that can be used to modify the morphology of the metal surface, or, depending on conditions, to promote formation of an oxide layer, which can be useful for different functional applications. In this section, the most frequently used anodic treatments of copper substrates, namely, anodization, and electropolishing, are briefly discussed.

Anodization

Anodization is an electrochemical passivation process applied to increase the thickness of the natural oxide layer on certain metal surfaces. The process received its name because it is performed under constant anodic potential, constant positive current, or anodic linear polarization, leading to oxidation of the metal surface and corresponding oxide formation. The anodization technique is often used to passivate Al or Ti and obtain Al_2O_3 and TiO_2 layers for different purposes. In addition to this frequent use, other metals, including Cu, have been anodized.

Reyter et al. [50][51] demonstrated that a nanostructured and highly electrocatalytic surface can be obtained by anodization of a polycrystalline copper electrode in 1 M NaOH. They showed that this process results in the formation of $Cu(OH)_2$ nanorods on the Cu surface. These nanostructures can then be thermally reduced under H_2 atmosphere and converted to metallic copper nanowires with a rough nanostructure surface. The same approach was applied by Wang et al. [52] for the formation of Cu nanowires on Cu foam.

Chen et al. [53] prepared core–shell $Cu@Cu_6Sn_5$ nanowires with a three-dimensional structure through an electrodeposition process using $Cu(OH)_2$ nanostructures obtained by anodization of the Cu substrate. Anodization was performed in 1 M NaOH aqueous solution under a constant current of 8 mA and at room temperature. The anodized Cu foil prepared in this way was used for electrodeposition of the Sn–Cu composite [53].

Anodic Treatment in Acid Electrolytes

Pauric et al. [54] demonstrated that a Cu surface with a regular morphology and a porous structure at the sub-micrometer scale can be obtained by anodic treatment of highly concentrated phosphoric or sulfuric acids. This approach requires a very viscous electrolyte with sufficient conductivity. Essential for this method is the presence of a small residual amount of water and a sufficiently high voltage to provide an intensive oxygen bubble evolution. Furthermore, it has been demonstrated that the thickness of the porous layer can be flexibly scaled up to over one micrometer and that the formation process can be performed independently of substrate purity or crystallographic features [54].

Another original electrochemical approach for tuning the morphology of structured Cu was proposed by Pham et al. [55]. They reported a complex strategy for controllable modulation of multiple parameters, including porosity, wetting properties, and transport, by applying different electrochemical steps. In particular, hierarchically designed copper inverse opal was created for enhanced liquid transport through the pores. This step was followed by electropolishing to systematically modulate the structural porosity. Finally, electrodeposition of the oxide layer and further electrochemical oxidation enabled functionalization of the porous structure [55].

2.3. Summary

Template-free methods for 3D Cu CC formation do not involve the use of any techniques for shape control of the copper deposits, which makes these approaches generally uncomplicated. By means of electrochemical de-alloying, a three-dimensional ligament-channel 3D Cu structure can be obtained. Materials created in this way offer a large specific surface area, which is typical for the other types of 3D Cu CCs as well, enabling several potential applications. Critical aspects of this method involve the alloy’s composition and structure and the de-alloying potential, fine-tuning of which are essential for the structural morphology, physical properties, and composition of the obtained 3D material. These challenges limit the easy and straightforward practical application of these methods to a certain extent, as strict control over those parameters is necessary.

Depending on the electrolyte media, anodic treatment of the pure Cu phase provides additional possibilities for structuring and modification. In particular, anodization of already-formed 3D Cu CCs in alkaline media results in the formation of $Cu(OH)_2$ with a nanomorphology that can be further chemically modified. Alternatively, anodic treatment of Cu in a highly concentrated acidic solution can be used to obtain sub-micrometer porosity for modulation of the fine surface morphology of 3D Cu materials synthesized by other methods.

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