MATEC Web of Conferences **41**, 01002 (2016)
DOI: 10.1051/matecconf/20164101002

A low cost - high efficiency electrodeposition device for the laboratory

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Abstract. A homemade electrodeposition device is presented, capable of depositing thin films of variable stoichiometry on cylindrical substrates offering control of electrolysis current, temperature and stirring velocity. The device design eliminates gravitational settling and uses a novel technique for fluid agitation in order to optimize deposition uniformity. The device surpasses traditional burato-style design in terms of gravitational settling of the solution and stirring homogeneity, as well as in terms of coating quality, i.e. cracking and blackened areas.

1 Introduction

The process of electrolysis involves an electrolyte, a solution of mobile ions which act as current carriers; a DC power supply which provides the energy to the electrolyte carriers; and two electrodes, anode and cathode, attracting the negative and positive, respectively, in the solution. The process may be used in the separation of materials or the deposition of material on one of the electrodes. The latter is called electrodeposition and is a versatile, simple and relatively low cost technique, popular in both research and industrial processes, for the coating of electrically conductive substrates of various geometries [1]. The properties of the deposited film depend on the Watts bath properties, the bath's temperature, the deposition time, and the applied voltage. The electrodeposition process is a complex kinetic phenomenon which cannot be entirely controlled by independent control of each of the above parameters. However, it is widely accepted that the relationship between deposition time and thickness is linear in the first 4-5 minutes gradually reaching a plateau for longer deposition times; higher voltage results in faster growth and therefore thicker films in a given time but moderate applied fields should be preferred over high fields for higher uniformity in the film surface; temperature increases the kinetic energy of the ions by approximately 2-3%/^{o}C and should be kept between 45^{o}C and 65^{o}C for NiFe film deposition. Therefore, it is necessary to control and manipulate deposition rate using the above parameters while also considering agitating the solution to avoid gravitational settling. Temperature is changed by applying external heat to the cell [2] or by controlled temperature water circulation inside the double wall of a cylindrical electrolytic cell [3]. Agitation is usually achieved by magnetic stirring [2] or by continuous peristaltic pumping [1].

The cell presented here was designed for the deposition of 50mm long NiFe thin films of variable stoichiometry and thickness on cylindrical austenitic steel substrates of 10mm diameter, using a novel approach of fluid agitation depending on the self circulation of the solution, which leads to more uniform depositions. Also the cell is designed in such a way as to eliminate gravitational settling.

2 Design Requirements

The requirements of the design were determined by our application: (i) stirring of the ion solution at a constant rate (ii) controlled heating for increased solubility (iii) controlled steady speed rotation of the cylindrical specimen, (iv) electrolysis current control.

The independent control of the above increase the adaptability of the system to the desired experimental process; furthermore, the proposed cell is a useful research and education tool which allows the study of the effect of each parameter to the deposition process and the obtained deposited films.

One of the novelties of the system is the cylindrical specimen's rotation resulting in controllable fluid agitation rather than stirring or pumping the fluid in the cell which ensures higher homogeneity in the deposited layer. The above functions are all controlled.

The requirements for the cell's cylindrical body are the following: it must be light, watertight, portable and modular so that is can be dismantled for servicing, cleaning and maintenance. It must allow for gradual and controlled filling and removal of the used solution. The

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material of the cell and its parts must be non-conductive in order not to interfere with the electrolysis process and tolerate high temperature and acidic solutions. Based on the above requirements, Teflon was used for the body and seals because it is hydrophobic, has one of the lowest coefficients of friction in solids, it is non-reactive, and therefore corrosion resistant, with excellent dielectric properties, hence an ideal insulator, and is subject to creep which is desired for the seals. The temperature of the solution will not exceed 100°C which is safely below the limiting temperature of the material.

3 Building blocks and functionalities

The electrodeposition system consists of the following distinct parts: i) the electrolytic cell ii) the base holding the cell and the AC motor iii) The electrical and control circuit iv) the DC power supply.

3.1 The electrolytic cell

Based on the requirements outlined above, 3D prototypes of the cell were generated using a commercial software package. Figure 1a shows the cross section of the 3D model along the longitudinal axis of the cylindrical cell.

The device is made of three parts (figure 1): 1) the main cylindrical body 2) the left end and 3) the right end; they are all made of Teflon. The parts are fastened together using stainless steel nuts and bolts. The volume of the cell is 250ml. The rotating cylindrical substrate is the cathode. The cylindrical 314 austenitic stainless steel anode lies in the interior of the cell on the right end. The left end holds a specially designed spiral propeller, which rotates with respect to the cathode, ensuring a uniform differential flow around the rotating cathode. A glass graduated test tube funnel is fastened with a screw at the specially designed hole at the top side of the cylinder. An outlet spigot is fastened the same way at the bottom to remove the used solution. O-rings with thermal and acid resistance are used for water tightness in all connections.

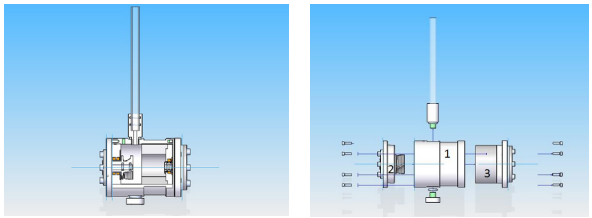

Figure 1: (a) Cross section of the 3D model of the electrodeposition cell (b) exploded view

The left end has a stationary and a rotating part. The stationary part is a custom designed Teflon flange which accommodates the connections to the rotating part and the water tight seals and holds the ball bearing. The rotating part (figure 2) is a Teflon cylinder whose one end is cut into the propeller stirring the solution and the other end holds the ball bearing. Special processing ensures that the rotating cathode will drift the rotating part with its move, as to stir the fluid the way it was described earlier. The cylindrical cathode is fastened inside the Teflon cylinder with three O-rings. As the cylindrical substrate is rotated, the Teflon cylinder rotates with it and so does the propeller.

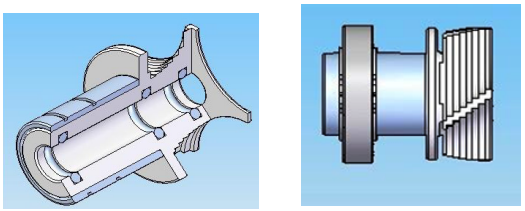

Figure 2 –The left end: the rotating cylinder with the propeller on the one end and the ball bearing at the other.

The right end of the cell has also a stationary and a rotating part. The stationary part is again a Teflon custom made flange which fits in to the central piece of the body and holds the ball bearing and the anode. The anode is fitted snugly to the inner side of the flange entering the inside of the main body. The rotating part is a Teflon cylinder which, similarly to the left end, has a sleeve of AISI 316 stainless steel which fits in the right ball bearing. The cylindrical substrate is inserted through the left end and goes through the right end stabilized with O-rings as described. It is secured in such a way as to allow for its uniform rotation with no translation and seals the cell.

Finally, the main body of the cell has two holes on top, one for the insertion test tube funnel used to fill the cell with the electrolytic bath and one to let air come out; and one hole on the bottom for the removal of the used bath. On the right end where the anode is placed in the inside of the cell, an indenture has been made on the external. This is used to adjust an electrical resistor used to heat the Teflon body, which, through its good thermal conductivity, in turn heats the anode and the bath within. A thermocouple inserted through the top hole measures the temperature which is controlled by the electrical circuit presented below.

This construction approach against the traditional burato-style electrodepositions mainly used in laboratories showed that typical problems like cracks or blackened areas on the deposited films, gravitational settling of the solution and no homogeneous stirring of the fluid during the process, have been eliminated. Also it allows for the control of several parameters independently and/or in combination, which leads to more reliable results.

3.2 The base and the AC motor

The cell is mounted and kept in position inside a fixture on an aluminum base on which the AC motor used for the cathode rotation is also adjusted (Fig. 3). The relative distances can be adjusted to accommodate cathodes of various lengths and the coupling between cathode and motor is electrically sealed. The base also holds the brush carbon contact connected in series with the rotating cathode.

3.3 The electrical and control circuit

The rotation speed, the temperature and the electrolysis process are all controlled through the electrical circuitry of the device (figure 4). The asynchronous three-phase AC motor controlled by an inverter is responsible for the cathode's rotation. The inverter controls the rotation speed, while the current through the thermal resistor used to control the bath's temperature is regulated as shown in figure 4.

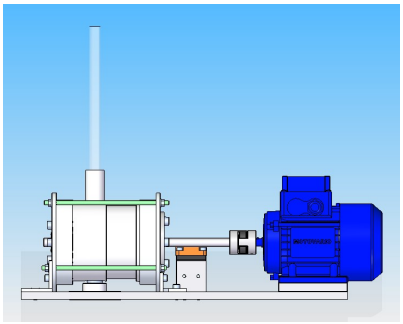

Figure 3 –The aluminum base holding the cell fixture, the carbon contact, the AC motor and the rotating cathode. A hole underneath the cell allows for the removal of used bath.

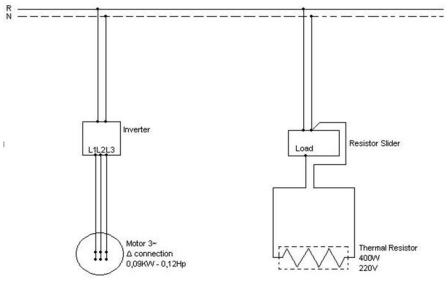

Figure 4 – Motor and temperature control circui

Figture 5 shows the DC power supply and its connection to the device, while figure 6 shows a picture of the actual device with the Teflon cell, the heating resistor around it, the electrical contacts and the AC motor on the aluminum base, all being manipulated by the control circuit shown behind the motor.

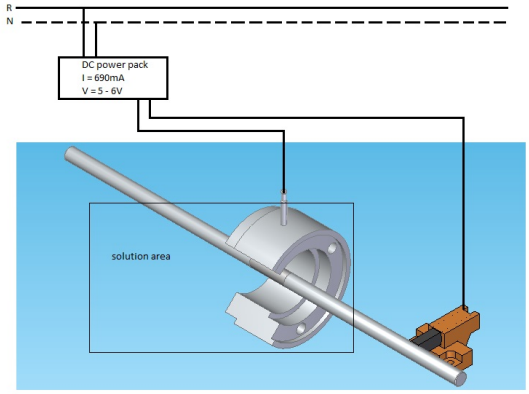

Figure 5 – Connection of the DC power supply to the device.

4 Electrodeposition procedure and sample characterization

In this section the sample preparation procedure is presented as a demonstration of the capabilities of the electrolytic device. The device has been used for the deposition of 50mm long NiFe thin film samples on

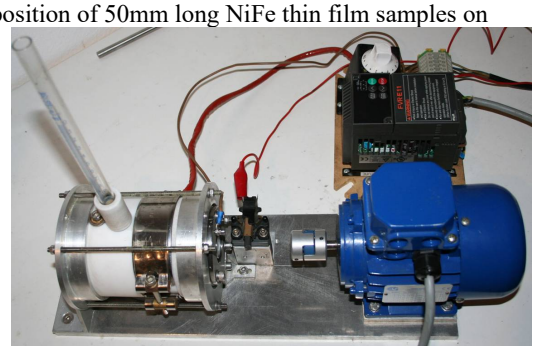

Figure 6 – The Teflon cell, with the heating resistor, the input tube, the electrical contacts, the AC motor and the control circuitry.

cylindrical 316L austenitic stainless steel of 10mm diameter. The substrate material was chosen to be nonmagnetic and resistant to chlorine solutions such as the electrolytic bath. The cylindrical substrates are polished before the electrodeposition.

The aqueous bath solution consisted of 25g/L H_3BO_3, 5g/L $NiCl_2$ and $NiSO_4.6H_2O$:$FeSO_4.7H_2O$ from 0:200 to 200:0 in order to deposit NiFe films of varying stoicheiometry Ni:Fe from 0:100 to 100:0. The bath pH was adjusted[2] to 3.0 adding H_2SO_4 with the current density set at 7-8 mA/cm^2. The temperature was regulated from 55 °C to 67 °C. The rotation speed was set to 620 rpm (32.5 cm/s). Two sets of samples were prepared for all stoichiometries at two different deposition times: 3 and 4 min. Optically, the deposited films are very good, with no cracks or blackened areas. Figure 7 shows the XRD results of samples prepared [4] with various stoichiometries Ni:Fe.

The electrolysis device was used continuously for over 300 minutes with no fault or problem.

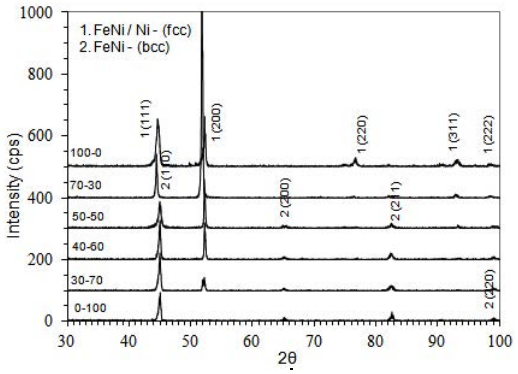

Figure. 7 XRD results of samples prepared with our electrolysis cell [4]

5 Conclusion

We have presented a novel design for electrodepositing devices. The proposed device is portable, easily maintained and offers controlled ion solution heating and stirring, electrolysis current control and controlled steady speed rotation of the cylindrical substrate. Its main advantages are increased deposition uniformity, less cracks and blackened areas. Furthermore, its increased controllability allows the adaptation to the specific experimental procedure. As a result, the device is equally suited to both experimental and educational purposes, since it can be used to demonstrate the effect of the various parameters to the electrodeposition process and quality.

ACKNOWLEDGMENT

This research has been co-financed by the European Union (European Social Fund – ESF) and Greek national funds through the Operational Program "Education and Lifelong Learning" of the National Strategic Reference Framework (NSRF) - Research Funding Program: ARCHIMEDES III, Investing in knowledge society through the European Social Fund.

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