Available online at www.sciencedirect.com
ScienceDirect
Surface & Coatings Technology 202 (2008) 4153 – 4158
SURFACE & COATINGS TECHNOLOGY
www.elsevier.com/locate/surfcoat

Effect of geometry on void formation in commercial electroplating of thin strips to copper

M.K. Okelman, B.G. Thomas *, M. Powers [1]

Department of Mechanical Science and Engineering, University of Illinois: Urbana-Champaign, Urbana, IL 61801, United States

Received 15 October 2007; accepted in revised form 3 March 2008
Available online 14 March 2008

Abstract

Three plating trials comprised of six samples were performed to evaluate the ability to attach thin strips of varying cross section to a copper substrate via commercial nickel electroplating in a nickel sulfamate bath. Nickel plated to the top, bottom, and sides of the nickel strips, as well as to the substrate. A significant void formed beneath rectangular nickel strips for all geometries studied, including strip widths of 500 μm to 5000 μm and gap thicknesses from 100 μm to 1500 μm. This is due to the starvation of ions when two regions of growing grains impinge and entrap a volume of electrolyte, surrounding it completely by deposited nickel. Impingement often occurs just past the edge of the strip which causes the void width to be greater than the strip width and form seams at the edge of the void. The rate of plating is greatest in areas where sharp corners exist, due to higher current density. More plating reaches under the strip as the width decreases and/or gap thickness increases. Thus, the void size decreases with decreasing aspect ratio, defined as the strip width over gap thickness. The use of a cylindrical strip produces sound plating with no voids. This is because the lack of corners allows impingement to occur first beneath the center of the circular cross section, so liquid is never surrounded. The results of this study demonstrate how to minimize or avoid void formation in commercial electroplating of suspended strips which is of great importance in the installation of sensor strips in the coating layer of continuous casting molds.

Keywords: Electroplating; Defects; Voids; Metal embedding; Coating failure; Thin film microsensors

1. Introduction

Electroplating is one method to attach a thin strip to a metal substrate by embedding it into the coating layer. Other methods to attach dissimilar metals include diffusion bonding [1–4], ultrasonic welding [5–7], and metallic paste [8,9]. Diffusion bonding requires some solid solubility between the two metals to be joined, such as that between copper and nickel. The two metals must also survive the necessary temperature and pressure associated with the hot isostatic pressing (HIP) process. Ultrasonic welding is suitable for welding materials of different thicknesses, such as thin foils to thick substrates, and is less sensitive to surface contamination.

The electroplating method has the advantage of simplicity and low cost [10]. It also embeds the strip within the coating layer, which affords protection and a smooth exposed surface (after machining). This is very beneficial when the strip contains delicate sensors, or when the surface of the substrate and strip must be flat [11,12]. However, when electroplating over complex geometries, defects such as voids or seams often appear, due to different rates of deposition throughout the geometry [13,14]. Such voids can be catastrophic, leading to spalling of the coating layer, if it is subjected to sudden temperature changes [15,16] Previous electroplating studies include measurements of mechanical properties [17,18] and models of the process [19–21]. Films of different surface morphology, thickness uniformity, grain size, and properties can be achieved by adjusting the current density, deposition technique, and/or composition of the solution bath [17]. Mathematical models of electroplating focus on the macro and micro-scale, including analytical models [19] as well as numerical models [20,21] which are compared to

* Corresponding author.
E-mail address: bgthomas@uiuc.edu (B.G. Thomas).
[1] Siemens VAI Services, LLC – Benton Harbor, MI, Benton Harbor, MI 49022.

0257-8972/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.surfcoat.2008.03.004

experimental data. None of this previous work has quantified the effect of strip geometry on the formation of voids or seams during electroplating.

This work investigates the attachment of strips to a substrate by affixing the suspended strip above the substrate and electroplating in a commercial facility. Three plating trials comprised of six samples were performed to quantify the effect of strip width, gap thickness, strip geometry (rectangular versus circular), and strip conductivity (non-conductive versus conductive) on the ability to attach thin strips to a copper substrate.

2. Electroplating process

Commercial electroplating involves depositing a metal coating layer onto a metal substrate via electrolysis. The electroplating process requires an electric circuit be completed through the bath, which includes electrons flowing to the cathode (substrate) from the anode (source metal), as well as returning through a conducting electrolyte containing the metal ions, driven by a DC power source. For the nickel sulfamate plating bath used in this work, the reactions are:

Cathode: $Ni^{2+} + 2e^- \rightarrow Ni;$

Anode: $Ni \rightarrow 2e^- + Ni^{2+}$

Metal ions arrive at the cathode surface through three principle paths: electrical migration, diffusion, and convection, both natural and forced, which replaces low ion concentration electrolyte with high ion concentration electrolyte [22]. Electrolyte fluid flow can be well represented by solving the incompressible Navier–Stokes equations in the entire bath, including the effects of thermal buoyancy. An additional time-dependent advection–diffusion equation is needed for the mass transport of the ions, which are carried by the liquid while diffusing through the liquid. The electric field, assumed to be steady-state, can be determined from a Laplace equation knowing the electric potential and the electrical conductivity. The effects of ion migration can be neglected if there is excess supporting electrolyte [20], such as the case for a large commercial bath.

It is also sometimes important to include time-dependent film resistance variation which occurs during the plating growth. The changing thickness leads to variations in the ion concentration and electric field near the cathode surface [20]. The theoretical plating thickness (assuming 100% current efficiency) can be calculated using Faraday's law [23,24]:

$$T = \frac{ItM}{zF}\frac{1}{ad}$$

where T is the thickness of the plating (cm), I is the current (A), t is the time (s), M is the atomic weight (58.7 g/mol), z is the ion valence (2), F is Faraday's constant (96,500), a is the substrate area (cm^2), and d is the density of the plating material (8.9 g/cm^3). This equation holds both locally (if local values can be found), and on average (based on the total time and plating area).

3. Experimental procedure

Three plating trials comprised of six samples were performed. Samples 1 and 3 were placed on one copper substrate, samples 2 and 4 were placed on another copper substrate, and samples 5 and 6 were placed on separate substrates. This arrangement made it possible to simultaneously investigate the effect of gap thickness and strip width on the ability to attach thin strips to a copper substrate.

In the constant gap thickness trial (samples 1 and 2), a thin triangular strip of rectangular cross section was held a constant distance above a copper substrate to test how the width of the strip affects the ability of nickel atoms to plate around the strip (Fig. 1). For sample 1, the strip width ranged from 1623 to 4924 μm, while the gap thickness was kept constant at ~589 μm. For sample 2, the strip width ranged from 725 to 2759 μm, while the gap thickness was kept constant at ~1647 μm.

In the constant strip width trial (samples 3 and 4), a rectangular strip of constant width was attached to the copper substrate a varying distance above the copper substrate to test how the distance between the nickel strip and the copper substrate, (i.e. the gap thickness), affects the plating ability (Fig. 1). For sample 3, the gap thickness ranged from 96 to 302 μm, while the strip width was kept constant at 1825 μm. For sample 4, the

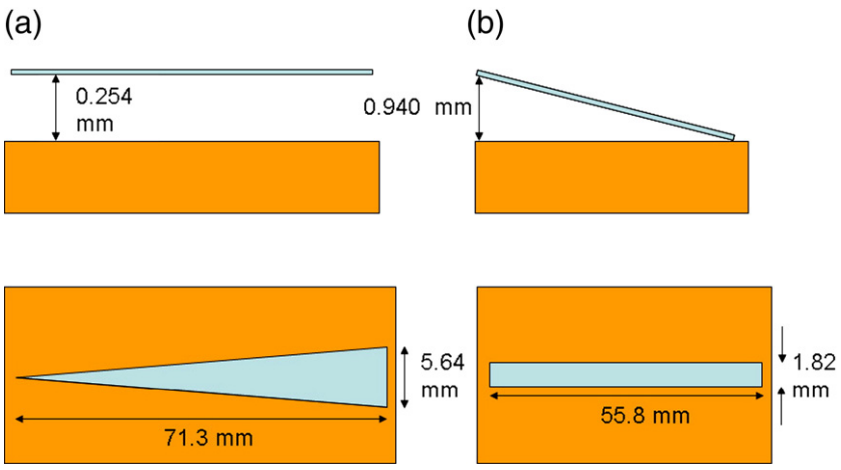

Fig. 1. Illustration of (a) constant gap thickness trial (sample 1) and (b) constant strip width trial (sample 3).

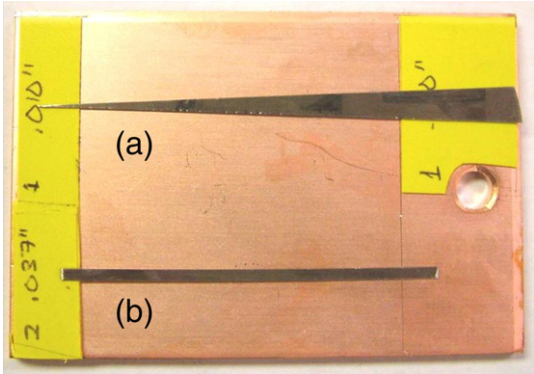

Fig. 2. Nickel strips mounted to the copper substrate (a) sample 1 (b) sample 3.

gap thickness ranged from 156 to 1317 μm, while the strip width was kept constant at 500 μm. Observed aspect ratios (strip width to gap thickness) ranged from 0.35 to 19.0.

The circular strip geometry trial (samples 5 and 6) investigated the effects of cross section shape and strip conductivity by plating over fibers with circular cross section at various distances above the substrate. Sample 5 consisted of a cylindrical "strip" of non-conducting optical fiber suspended 0 to 1524 μm above the substrate. The optical fiber was composed of a plastic protective outer coating surrounding a cladding layer and glass inner core. With the plastic coating the fiber has a diameter of 200 μm, while the cladding layer surrounding the glass core has a diameter of 125 μm. The glass core itself is only 9 μm in diameter. Sample 6 consisted of a cylindrical tube of 316 stainless steel with an outer diameter of 254 μm and inner diameter of 127 μm suspended a constant distance of about 10 μm above the substrate.

The copper substrate was etched with nitric acid prior to the installation of the strips to clean the copper surface before electroplating. The nickel strips were cleaned by scrubbing with calcium carbonate and a 3-M burgundy Scotch-Brite abrasive pad. As shown in Fig. 2, the strips were then suspended just above the copper substrate to perform a constant gap thickness trial and a constant strip width trial simultaneously. Yellow vinyl tape was attached to the copper substrate to achieve the desired gap thickness profile between the strips and the substrate. An investigation of the effect of electrically insulating the suspended strip from the grounded substrate was initiated by preventing some strips, via tape, from contacting the copper substrate. Each assembly was then masked prior to electroplating. The optical fiber (sample 5) and the stainless steel tube (sample 6) were not treated before plating.

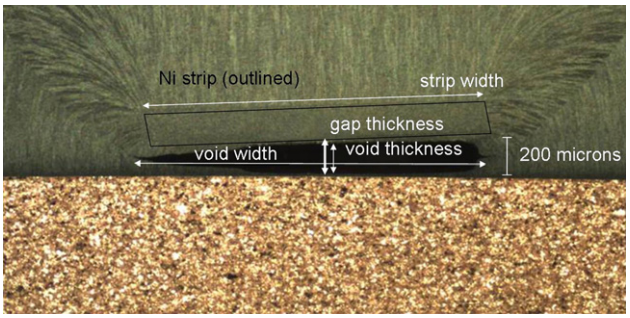

Fig. 3. Close-up of void region where quantitative measurements were made.

Each assembly was pretreated by rinsing with DI water and immersing in a 10% sulfamic solution to clean the surfaces. The strips did not seem to suffer any negative effects due to this pretreatment. Each assembly was then placed in a nickel sulfamate bath with a concentration of 1.5 mole/l at a commercial plating facility in Benton Harbor, Michigan. Due to the high solubility of the salts of sulfamic acid, high concentrations of nickel ions are possible, permitting high rates of plating. This same commercial facility and procedure is used to plate copper continuous casting molds for the steel industry. The substrate area was 2500 mm^2; so 0.75 amps of current were required for 0.0254 mm of plating per hour. A potential of 1.8 V was applied between the copper substrate and suspended nickel strips (cathode) and a grated metal basket of nickel pieces that comprised the anode located ~150 mm away in the bath. After 65 h a total of 2.08 mm of nickel (ultrasonic measurement) was deposited on the copper substrate.

For the constant gap thickness trial and constant strip width trial the electroplated nickel strip was cut into cross sections using wire electrical discharge machining (EDM). Each sample was mounted in epoxy, rough ground with 180 grit SiC paper, hand ground with 240, 320, 400, and 600 grit, and machine

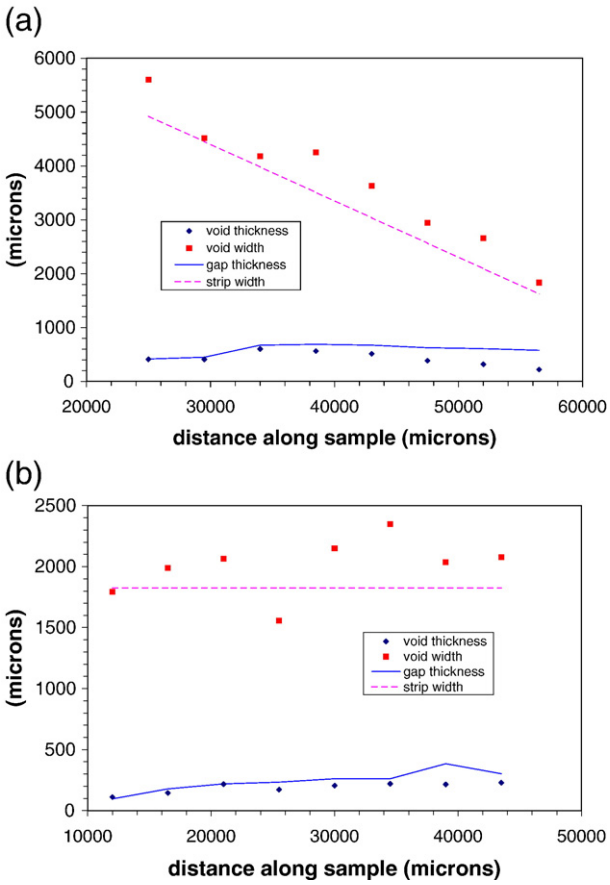

Fig. 4. Impingement often occurs just past the edge of the strip which causes the void width to be greater than the strip width and form seams at the edge of the void (a) sample 1 (b) sample 3.

ground using 800 and 1200 grit. Machine polishing was also performed with a short knap cloth and 1 μm solution and 0.3 μm solution when necessary to obtain a mirror-like finish. Etching was performed by immersing each sample in 1 part nitric acid, 1 part acetic acid (glacial), and 1 part DI water for 15 s.

4. Results and discussion

Optical microscopy was used to identify the strip location and the surrounding plating structure. The void thickness and width were measured from traces of the photographs. In addition, the gap thickness and the thickness of the nickel plating under the strip were measured at 6 different locations: above and below the void at left, right, and center (Fig. 3). For both trials, a plot of the void thickness, void width, gap thickness, and strip width versus the distance along the copper substrate was created (Fig. 4).

Plating growth impingement often occurs just past the edge of the strip which causes the void width to be greater than the strip width and form seams at the edge of the void. Fig. 4 also shows that as the strip width decreases, the void thickness decreases and more plating occurs under the nickel strip. Based on this observation, it is proposed that the extent of plating under the nickel strip is related to the aspect ratio, defined as the ratio of the strip width to the gap thickness (Fig. 5). Specifically, more plating reaches under the nickel strip as the aspect ratio decreases. In the constant gap thickness trial, this is due to the decrease in strip width, while in the constant strip width trial, the same observation is due to the increase in gap thickness.

The sequence of events leading to the observed microstructure and void shape is sketched in Fig. 6. Nickel plated onto the copper substrate and to the top, bottom, and sides of the nickel strip, even when there was no physical connection to the copper substrate. The plating layers on each of the nickel strips grew outwards at the same rate regardless of whether or not it was insulated on its ends. This indicates that the nickel strip is effectively grounded through the conductive bath across the short distance to the copper substrate. The theoretical average deposit thickness was calculated as 2.40 mm, which is very close to the 2.08 mm actually measured. Minor plating thickness abnormalities at the edges of the small copper substrate were responsible for the difference. However, the plating

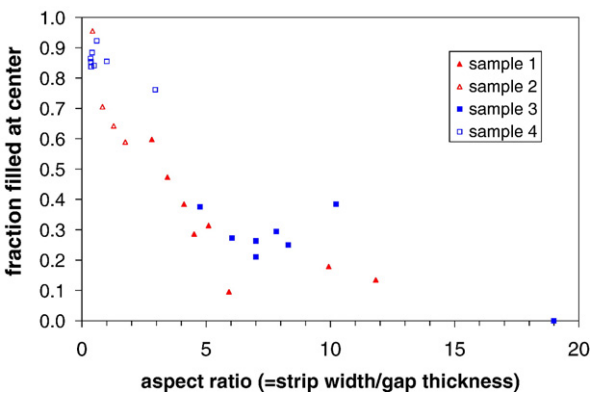

Fig. 5. More plating is obtained underneath the strip as the aspect ratio decreases.

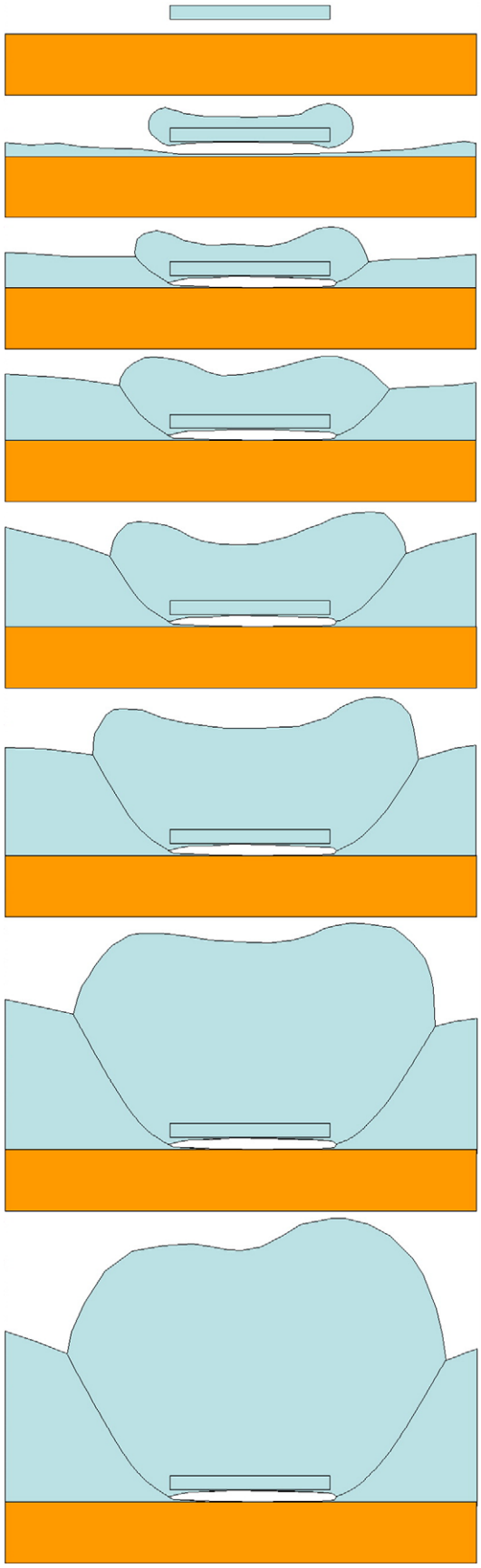

Fig. 6. Early on the plating growing from the corners of the strip contacts the plating originating from the substrate and "starves" the region beneath the strip from nickel ions, creating a void.

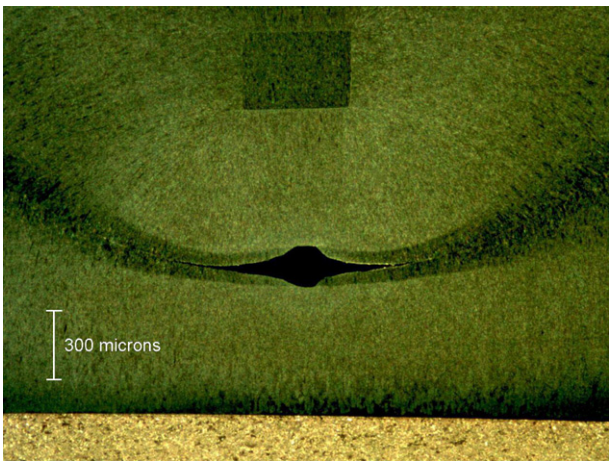

Fig. 7. Optical microscope photograph of cross section of nickel plated copper substrate (sample 4). Even for small aspect ratios the geometry of the strip causes the plating growths to contact in a way which produces seams and a void.

depositing onto the nickel strip corners is faster than elsewhere, owing to the higher local current density. Thus, it quickly grows to contact the plating layer originating from the copper substrate. This traps a region of electrolyte beneath the strip. This region becomes "starved" of nickel ions because the transport of ions is prevented (i.e. migration and diffusion), owing to its isolation from the rest of the bath, as convection is unable to replace the low ion concentration electrolyte with high

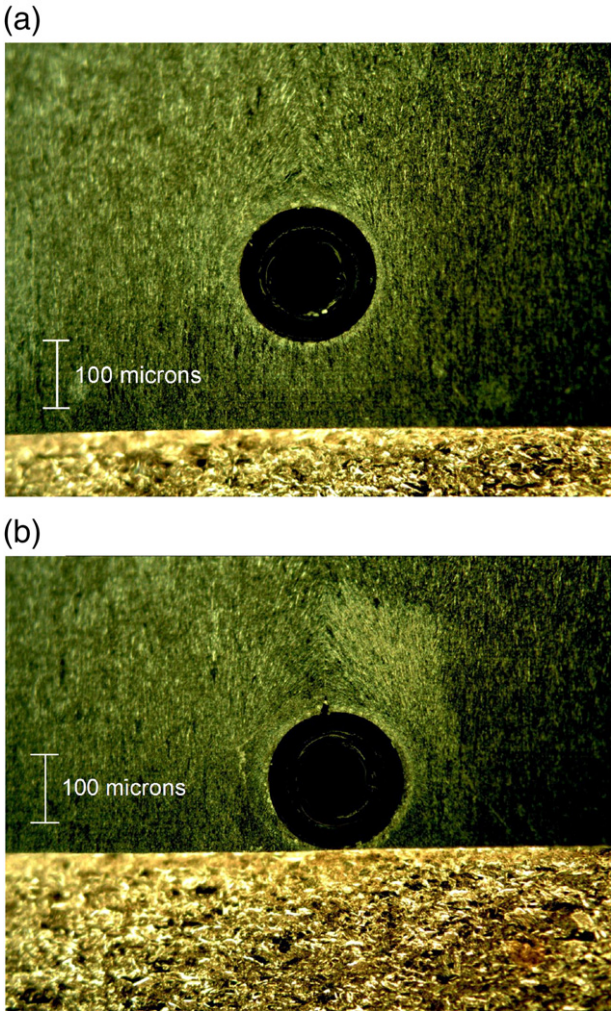

Fig. 8. Optical microscope photograph of cross section of nickel plated copper substrate with non-conductive cylinder (sample 5). No voids or seams formed (a) cylinder near substrate (b) cylinder contacting substrate.

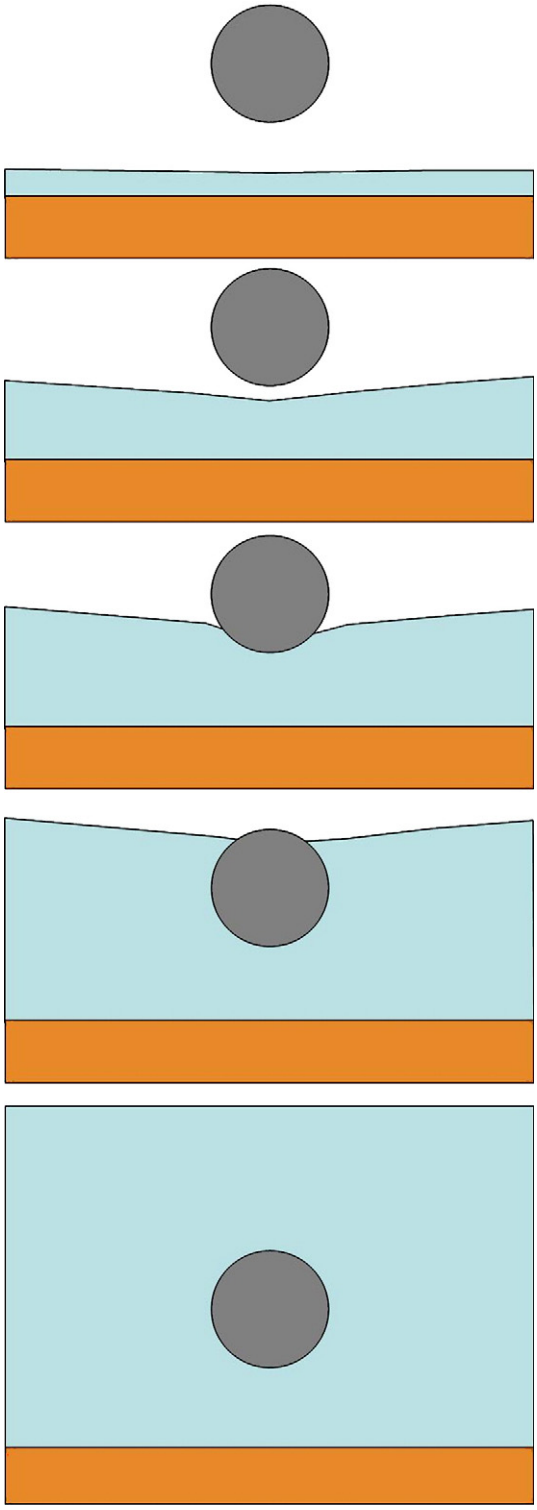

Fig. 9. Complete filling is observed when plating over a non-conductive cylinder. The plating growing from the substrate does not contact the cylinder in a way which produces trapped electrolyte.

ion concentration electrolyte. As in shrinkage-cavity macroporosity observed in metal foundry castings [25–27], a liquid region completely surrounded by solid will result in a void, as seen in Fig. 7. As a consequence, every rectangular specimen contained a void between the rectangular strip and substrate. Even moving the strip almost three widths away from the substrate is not enough to prevent void formation.

When plating over a non-conductive cylinder (sample 5), complete filling is observed, as seen in Figs. 8 and 9. The lack of a void in this geometry is expected because 1) without corners, the growth rates from the cylinder surface are almost uniform, and 2) the plating growing from the copper substrate has further distance to reach the cylinder extremities. Fig. 8 shows that the nickel grain structure originated from the copper substrate and grew up and around the non-conductive cylinder. The cylinder remained non-conductive and uncharged so no detectable deposit formed on its surface. In the case of a conductive cylinder as in sample 6 (not pictured), the plating layer growing from the cylinder is almost uniform, owing to the lack of corners. Thus, the plating from the copper substrate first contacts the cylinder near its center. This avoids entrapping electrolyte and therefore prevents void formation. Thus, cylindrical fibers experience sound plating, regardless of their insulating properties.

5. Conclusions

This work investigates the attachment of suspended strips to a copper substrate by electroplating. Three plating trials comprised of six samples were performed to evaluate the plating ability to attach thin strips to a copper substrate by measuring the occurrence and size of voids found under the strips. As the strip width decreases, or gap thickness increases, more plating penetrates under the strip. In other words, the aspect ratio roughly governs the fraction of the gap filled by the plating. Voids form beneath rectangular strips even for aspect ratios less than one. The plating growing from the strip corners contacts the plating originating from the substrate and “starves” the region beneath the strip from ions. The use of a cylindrical strip produces sound plating with no voids because the lack of corners allows impingement to occur first beneath the center of the circular cross section. The results of this study can be used to minimize or avoid void formation in commercial electroplating of suspended strips which is of great importance in the installation of sensor strips in the coating layer of continuous casting molds.

Acknowledgments

Funding for this work was provided by the National Science Foundation under contract CMMI 05-28668 and the UIUC Continuous Casting Consortium. Optical microscopy was carried out in the Frederick Seitz Materials Research Laboratory Central Facilities, University of Illinois, which is partially supported by the U.S. Department of Energy under grants DE-FG02-07ER46453 and DE-FG02-07ER46471.

References

[1] I.S. Batra, G.B. Kale, T.K. Saha, A.K. Ray, J. Derose, J. Krishnan, Mater. Sci. Eng. A369 (2002) 119.
[2] L. Esposito, A. Bellosi, S. Guicciardi, G. De Portu, J. Mater. Sci. 33 (1998) 1827.
[3] M. Eroglu, T.I. Khan, N. Orhan, Mater. Sci. Technol. 18 (2002).
[4] F.M. Coughlan, H.J. Lewis, J. Mater. Process. Technol. 153–154 (2004) 240.
[5] J.M. Peterson, H.L. McKaig, C.F. DePrisco, IRE Natl. Conv. (1962) 3.
[6] J. Antonevich, IRE Transactions on Ultrasonic Engineering, vol. 71, 1960, p. 26.
[7] S.A. Francis, IEEE Transactions on Product Engineering and Production, vol. 71, 1963, p. 13.
[8] K. Hayashi, A. Ishikawa, T. Endho, H. Yamagishi, IEEE First World Conference on Photovoltaic Energy Conversion, vol. 1, 1994, p. 535.
[9] P.W. Krempl, C. Reiter, W. Wallnofer, J. Neubig, Proceedings of the IEEE 2002 Ultrasonics Symposium, vol. 1, 2002, p. 949.
[10] M. Schlesinger, M. Paunovic, Modern Electroplating, 4th ed.Wiley, New York, NY, 2000.
[11] X. Li, F. Prinz, J. Manuf. Sci. Eng. 125 (2003) 1.
[12] H. Choi, A. Datta, X. Cheng, X. Li, J. Microelectromechnical Syst. 15 (2) (2006) 322.
[13] J. Jorne, A.M. Tran, Proceedings of the IEEE 2001 International Interconnect Technology Conference, 2001, p. 27.
[14] S. Chiu, J. Shieh, S. Chang, K. Lin, B. Dai, C. Chen, M. Feng, J. Vac. Sci. Technol. B: Microelectron. Nanometer Struct. 18 (6) (2000) 2835.
[15] V. Teixeira, Surf. Coat. Technol. 146 (2001) 79.
[16] S. Amirhaghi, H.S. Reehal, R.J.K. Wood, D.W. Wheeler, Surf. Coat. Technol. 135 (2001) 126.
[17] A. Ibanez, E. Fatas, Surf. Coat. Technol. 191 (2005) 7.
[18] J.R. Tuck, A.M. Korsunsky, R.I. Davidson, S.J. Bull, D.M. Elliott, Surf. Coat. Technol. 127 (2000) 1.
[19] T. Kessler, R. Alkire, J. Electrochem. Soc. Electrochem. Sci. Technol. 123 (7) (1976) 990.
[20] G. Ritter, P. McHugh, G. Wilson, T. Ritzdorf, Solid State Electronics 44 (5) (2000) 797.
[21] P.C. Andricacos, C. Uzoh, J.O. Dukovic, J. Horkans, H. Deligianni, IBM J. Res. Develop. 5 (1998) 567.
[22] E. Raub, K. Muller, Fundamentals of Metal Deposition, Elsevier Publishing Company, 1967, p. 41.
[23] H.J. Lyons, in: F.A. Lowenheim (Ed.), Modern Electroplating, 3rd ed., John Wiley, New York, 1974, p. 1.
[24] S. Glasstone, The Fundamentals of Electrochemistry and Electrodeposition, Franklin Publishing Company, 1960, p. 17.
[25] J. Campbell, *Castings*, Butterworth-Heinemann Ltd, Linacre House, Jordan Hill, Oxford, 1991, p. 175.
[26] T.E. Morthland, P.E. Byrne, D.A. Tortorelli, J.A. Dantzig, Metall. Mater. Trans. B 26B (1995) 871.
[27] P. Hausild, C. Berdin, P. Bompard, N. Verdiere, Intern. J. Pressure Vessels and Piping 78 (2001) 607.