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Electroplating of Nickel in Grooves Under the Influence of Low and Medium Frequency Ultrasound

By N. G. Sarius^{1,2}, P. Leisner^{1,3}, J. Hald⁴, and L. Hultman²

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1 Introduction

Micro electroforming is used for producing tools for replicating CD and DVD, micro fluid systems, and 3D MOEMS (micro-opto-electromechanical systems) [1]. When electroforming 3D replica masters, cavities can be left in holes and grooves if aspect ratios are too large. These cavities are the result of mass transport limitation and lower current density in high aspect ratio grooves. Cavities can then be sealed by the plated metal at the top before the groove is filled completely by plating. Since electroformed replication tools are thick (several hundred micrometers or more), plating speed, i.e., current density, is important for reducing process time, which is a problem concerning the current distribution that increasingly deviates between the bottom of a hole and its top surface when current density rises. To improve filling of high aspect ratio holes and grooves, additives [2] and pulse reversal [2] plating can be combined or used separately. It is difficult to agitate the electrolyte in the high aspect ratio holes with conventional agitation methods such as pumping, sample rotation, and air bubbling. Mass transport in the deep grooves is critical in this case, and makes it close to impossible to fill a groove from bottom to top.

Ultrasonic agitation during deposition of metals has been studied over the years, and it is thought to have various benefits compared to conventionally agitated electrodeposition and electroless deposition. In the literature, the use of ultrasonic agitation has been reported to reduce internal stress in electroplated nickel [3–5], to increase internal stress in electroless nickel-phosphorous [6], to increase deposition rates of nickel-phosphorous [7], to increase hardness of chromium [8] and nickel [4, 5, 9], as well as to reduce grain size [4] and smoothen surface finishing [10]. The benefits from using high power ultrasound [11] are due to the emerging and collapsing cavitation bubbles, causing acoustic streaming that results in continuous cleaning/activation of the electrode, and increased mass transport [12]. This increased mass transport caused by ultrasound can change a diffusion-controlled system into a charge transfer controlled system [13], which means that current density can be increased in an electrolyte with low metal concentration. The influence of ultrasound during electroplating has been studied earlier on nickel [4, 5, 9, 14, 15], copper [10], chromium [8], zinc [13], and iron [9].

Earlier studies with 1 MHz ultrasonic standing waves parallel to the cathode surface while depositing nickel in a modified Watts electrolyte showed improved filling capacity of trenches [16]. The effect of using ultrasound while depositing copper has also been studied with improvement of filling in substrate trenches [17, 18]. Interest arose to investigate the

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influence of ultrasonic agitation on filling properties during electroplating using a 25 kHz transducer, low frequency mode (*LF*-mode), fronting the cathode surface, with no obstacles between transducer and cathode in the present study. This setup gives a more defined and uniform ultrasonic effect, and also a controlled direction of the ultrasonic agitation on the cathode surface. Emerging and collapsing of bubbles on and near the surface and in the holes and grooves on the cathode, caused by directed ultrasound, should enhance mass transportation into high aspect ratio grooves and holes. A risk, however, is that the violently collapsing bubbles cause pits on the plated surface. Another setup, medium frequency mode (*MF*-mode), with 100 kHz and 400 kHz ultrasonic standing waves parallel to the master surface was also investigated in order to study the influence of different standing-wave frequencies on filling properties at different aspect ratios of holes and grooves. Furthermore, ultrasound has been suggested to increase liquid flow through holes during processing of printed circuit boards [19].

2 Ultrasonics Theory

When using high intensity low frequency ultrasound (here 25 kHz), two effects should be considered: finite amplitude pressure variations and radiation pressure. These primary effects lead to a number of second order phenomena, among these are cavitation and various types of acoustical streaming which, as shown in the present investigation, lead to enhancements in relation to electroplating, given appropriate experimental parameters and boundary conditions.

In the present study, vaporous cavitation induced by high intensity 25 kHz ultrasound is considered to be the main driver for enhanced plating properties. Vaporous cavitation (very short lived collapsing bubbles) is characterized by high impact of the surface area by vigorously collapsing cavitation bubbles that increase mass transport due to locally very high temperatures and variations in pressure. In parts of the plating process, gaseous cavitation can increase agitational flow speeds in the bulk fluid when the bubbles are moved towards the plating surface opposite to the transducer. Dragging the surrounding fluid due to viscous forces, the bubbles are moved by the radiation pressure acting on them. This is referred to as quasi-acoustic streaming. Moreover, oscillating bub-

bles produce so-called micro streaming which generally occurs in the extremely inhomogeneous acoustic field generated by the transducer. Micro streaming causes significant mass exchange close to the surface of the substrate [20, 21].

The challenge when using low frequency ultrasound generating cavitation and agitation is to find the optimum distance between transducer and substrate and the acoustic intensity where the substrate is not heated too much and cavitation activity does not disturb plating, but still has an enhancing effect on plating from enhanced agitation and mechanical treatment of the deposit reducing internal stresses. Pin-holes on the deposit are a well known difficulty when using high-intensity ultrasound assisted plating because cavitation bubbles can stick to the surface. Furthermore, collapsing cavitation bubbles generate strong localized jets close to the substrate surface. However, the effect can be minimized by substrate movement.

The other type of ultrasound used in the present study is a setup generating standing waves parallel to the substrate by medium frequency ultrasound. The two frequencies used are 100 kHz and 400 kHz. A steel plate is used as reflector, and the distance between transducer and reflector as well as the angle are adjusted carefully for establishing a strong standing wave field.

Optimum reflection at the plate is achieved when its thickness L is an uneven multiplication of a quarter of a wavelength λ_{steel} in the plate, i.e., Equation 1 for natural n reads

$$L = (2n - 1) \lambda_{\text{steel}}/4 \quad <1>$$

In [11] it was shown that standing waves generate near-boundary acoustic streaming because of the interaction with a solid boundary. Near-boundary acoustic streaming differs from ordinary hydrodynamic flow as it originates from boundary attenuation. Figure 1 shows a schematic of near-boundary streaming for a standing wave (modified *Rayleigh* streaming), which proves most relevant in describing the experiments presented. The acoustic standing wave generates vortices on both sides of the particle velocity nodes. The width of each vortex is equal to one quarter of a wavelength $\lambda = cf$, where c is the speed of sound and f is the frequency. Movement of the substrate inherits a wave pattern of the deposit (varying thickness) that else wise can be observed

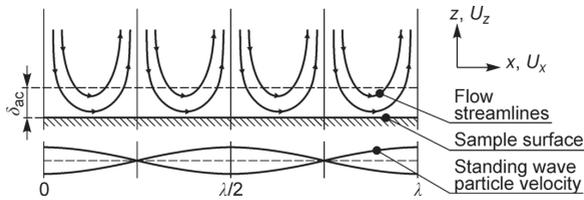


Fig. 1: Near-boundary streaming for a standing wave (modified Rayleigh streaming), after Pocwiardowski [11]

due to the presence of particle velocity nodes and anti-nodes. The near-boundary streaming acts as a local mass transport phenomenon.

The *Rayleigh* streaming pattern shown is slightly different from *Schlichting* streaming where vortices in the boundary layer are decoupled from the outer flow and hence decrease mass exchange to the surface. *Schlichting* streaming is only valid for obstacles much smaller than the wavelength, which is not the case here. When vortices appear close to holes or grooves, it is expected that the viscous forces exerted by the rotating fluid will cause fluid movement into the grooves. Though the width of substrate grooves in these experiments is 0.3 to 1 mm, modified *Rayleigh* streaming caused by an ultrasonic frequency with a quarter of a wavelength between 0.3 and 1 mm is expected to have the highest impact on filling properties during electroplating.

For the two frequencies used, a quarter of the wavelength is approximately 0.94 mm and 3.75 mm, respectively, which are values that should be comparable with the width or other characteristic dimensions of the grooves. The agitation obtained by modified *Rayleigh* streaming is well controlled and relatively uniform. Moreover, the acoustic streaming adjusts its shape to the surface interacting with the sound waves.

3 Experimental

3.1 Laboratory Setup

Figure 2 shows the tank used for electroplating, with a total volume of 160 liters. The tank has an overflow between a main tank and a reservoir with heating and filtering equipment. Filtered and circulated electrolyte is pumped in at the bottom of the main tank

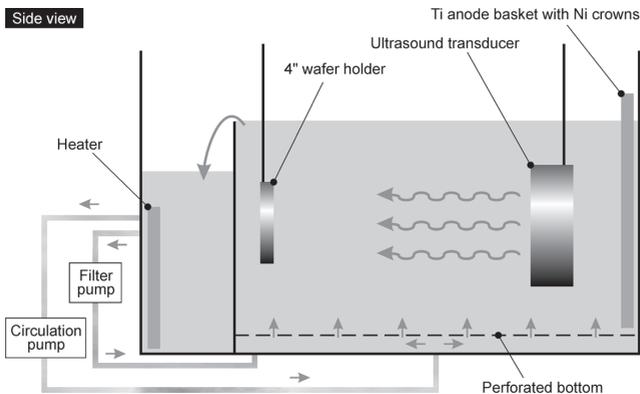


Fig. 2: Setup of ultrasonically assisted plating bath

through a perforated double bottom giving laminar vertical flow and good temperature control. During experiments, the filter pump was turned off, and the circulation pump was varied from a minimum flow of 0.3 m³/h to a maximum flow of 6 m³/h.

To enable simultaneous control of current and ultrasonic agitation versus time, an upgraded pulse plating control system (*TCD Teknologi*) was used. 20 A and 100 A pulse rectifiers were used for the experiments.

Figure 3 is a schematic of the 25 kHz ultrasonic transducer with adjustable power in the range of 0 to 225 W used in *LF*-mode (built for the purpose, *Reson A/S*). The transducer features 8 transducers placed in an array for the ultrasound to protrude homogeneously and primarily in one direction. The transducer was placed between the cathode and the anode, fronting the cathode, when plating in *LF*-mode (Fig. 2). For conventional- and *MF*-mode plating, the *LF*-transducer was removed from the tank. The distance between transducer and substrate could be varied from 10 to 40 cm in the plating tank (Fig. 2).

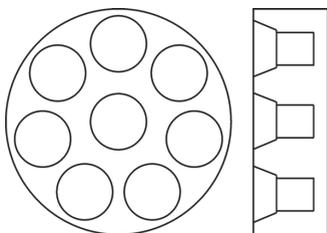


Fig. 3: Schematic of 25 kHz ultrasonic transducer made up of 8 small transducers working together as one

Figure 4 shows *MF*-mode with a setup consisting of an ultrasonic transducer facing a stainless steel plate, with a sideways movable cathode. The transducer used in *MF*-mode was operated at 100 kHz and 400 kHz with 4 W during the experiments. A small hydrophone was used to adjust the parameters in order to produce a standing wave in front of the substrate in the electrolyte between the transducer and the ultrasonic reflecting stainless steel plate. To avoid a wave pattern formed in the Ni electrodeposit caused by the standing wave [16], the cathode was moved sideways during electroplating.

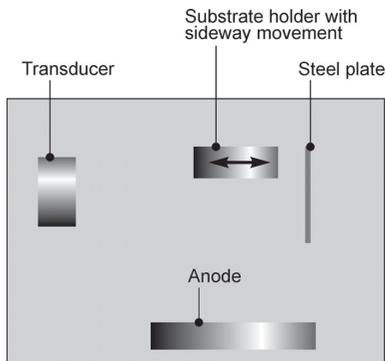


Fig. 4: Schematic setup of parallel ultrasonic standing wave viewed from above, with ultrasonic transducer facing a reflector in front of sideways movable cathode

Tab. 1: Composition and parameters for the sulphamate electrolyte used in the present study

Substance/parameter	Value
Ni(NH ₂ SO ₃) ₂	504.5 g/l
H ₃ BO ₃	40 g/l
NiBr ₂	6.97 g/l
Wetting agent: Ankor [®] F (2.5% Bayer FT248)	10 ml/l
pH (adjusted with sulphamic acid)	3.8±0.2
Temperature	45–50 °C

3.2 Electrolyte

The composition and parameters for the sulphamate electrolyte used in the present study is shown in Table 1.

3.3 Substrates

Discs of brass (SS 5168-06) with milled 3D pattern slits and holes were used as substrates. The pattern is shown in Figure 5. The thickness of the disc was 3.5 mm and the diameter 105 mm, with the pattern placed inside a diameter of 89 mm. This was to ensure that edge effects would not affect the plating results.

Table 2 shows the approximate width, depth, and aspect ratios of the 3D pattern. The patterns were oriented differently in each quadrant of the discs in order to study possible effects of the orientation.

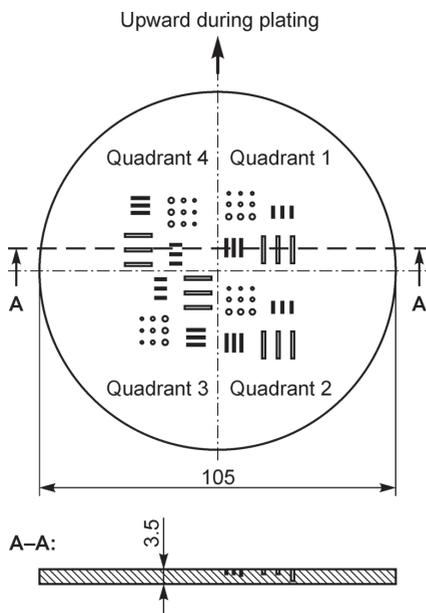


Fig. 5: Design of experimental 3D disc with grooves and blind holes

Tab. 2: Approximate aspect ratios as a function of depth and width of grooves

Width (mm)	Depth (mm) at aspect ratios of		
	0.65	1.2	2.9
0.35	0.25	0.45	0.90
0.55	0.35	0.60	1.55
1.1	0.60	1.1	3.0

3.4 Process Parameters

Table 3 shows process parameters for seven experiments: one in conventional mode with agitation by pumping the electrolyte through the perforated bottom, four in *LF*-mode with ultrasonic agitation perpendicular to the substrate, and two in *MF*-mode with standing waves parallel to the substrate surface. Initial experiments with *LF*-mode showed that the ultrasound heats the cathode. In the present *LF*-mode setup, the temperature of the cathode stabilizes at 5 °C above the temperature of the electrolyte after approximately 10 min. In order to provide a temperature of 50±1 °C on the substrate in all experiments, the temperature in the electrolyte was lowered to about 45 °C when *LF*-mode was applied. Plating current was set to 0.4 A/dm² during the first 720 s to avoid corrosion of the substrate while preheating the cathode in *LF*-mode experiments, and for uniformity reasons also in *MF*- and conventional mode. No heating of the cathode was observed when using ultrasound orientated in parallel. The temperature of the electrolyte was set to 50 °C in conventional and *LF*-mode.

For conventional and *LF*-mode, actual current density was approximately 12.9 A/dm² for 10 600 s in the patterned region of the substrate, resulting in approximately 455 µm of nickel. For *MF*-mode, the actual current density was approximately 5.5 A/dm², yielding 194 µm of nickel at the substrate center. In the case of conventional and *LF*-mode, a current thief was used to avoid large variations in current density. When using *MF*-mode, however, the current thief was removed in order to not disturb the standing wave along the substrate surface. Removing the current thief resulted in increased nonuniformity of the

Tab. 3: Process parameters for experiments during electroplating

Experiment	Ultrasound frequency	Distance transducer to cathode	Direction of ultrasound related to cathode	Agitation by pump	Temperature of electrolyte	Temperature of 3D disk after 720 s
0H	off	n/a	–	6 m ³ /h	50 °C	50 °C
U40L	25 kHz	40 cm	perpendicular	0.3 m ³ /h	45 °C	50 °C
U40H	25 kHz	40 cm	perpendicular	6 m ³ /h	46 °C	51 °C
U10H	25 kHz	10 cm	perpendicular	6 m ³ /h	45 °C	49 °C
U10L	25 kHz	10 cm	perpendicular	0.3 m ³ /h	45 °C	49 °C
100UL	100 kHz	n/a	parallel	0.3 m ³ /h	50 °C	50 °C
400UL	400 kHz	n/a	parallel	0.3 m ³ /h	50 °C	50 °C

current distribution over the surface. In the absence of a current thief, the current density had to be limited to 5.5 A/dm^2 in the center of the substrate to avoid severe hydrogen development around the edges of the substrate.

4 Results and Discussion

No significant differences in the results were observed between the four quadrants of the substrates. Therefore, representative results are presented only from Quadrant 4 in the following. Due to edge effects, the area outside the patterned area of the substrate showed a thicker deposit but inside this region the thickness on the flat surface varied by less than 7 percent.

4.1 Filling of 3D-Grooves

To understand the influence of ultrasonic power on filling properties, four experiments were performed in *LF*-mode with the transducer at different distances from the substrate and with different additional agitation originating from pumping. The influence on filling properties by *MF*-mode was investigated using two frequencies. One experiment was also performed as reference with conventional mode. The filling of the grooves is presented as relative nickel thickness in the bottom of the grooves related to the average thickness on the top of the plate.

Figure 6 shows the filling of nickel at the bottom of 1 mm wide grooves with depths of approximately 3 mm, 1 mm, and 0.5 mm. It can be seen that 3 mm deep grooves were plated at the bottom when using *LF*-mode and *MF*-mode with 400 kHz. When using conventional or *MF*-mode with 100 kHz, no nickel

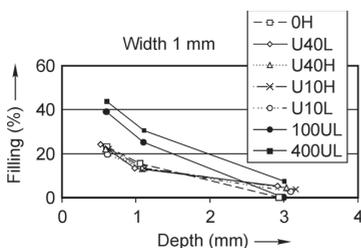


Fig. 6: Filling of nickel at the bottom of 1 mm wide grooves in percent, related to the nickel top surface thickness near the grooves

was deposited at the bottom. The difference for the *MF*-modes is probably due to differences in size of the vortices formed by the two frequencies, where the 0.94 mm vortex at 400 kHz fits the dimension of the groove perfectly. In comparison, the 3.75 mm vortex at 100 kHz is rather large. In less deep grooves (1 mm and 0.5 mm), no clear advantage of using *LF*-mode can be shown, but *MF*-mode shows improvements of the filling at the bottom of the grooves compared to conventional mode. This is due to the generated near boundary micro streaming.

Figure 7 shows the filling at the bottom of 0.55 mm wide grooves with depths of approximately 1.55 mm, 0.65 mm, and 0.35 mm. Conventional mode results in no deposition at all at the bottom of any groove, but in *LF*- and *MF*-modes the bottoms of all grooves were plated. *MF*-mode has better filling properties than the *LF*-mode, especially at the two lower aspect ratios.

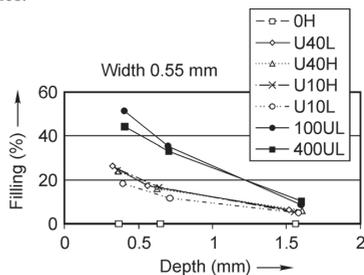


Fig. 7: Filling of nickel at the bottom of 0.55 mm wide grooves in percent, related to the nickel top surface thickness near the grooves

Figure 8 shows the filling at the bottom of 0.35 mm wide grooves with depths of approximately 0.9 mm, 0.45 mm, and 0.3 mm. Again, no nickel is plated at the bottom of the grooves by conventional mode, but the grooves are plated in *LF*- and *MF*-modes. The same tendency as found in Figures 6 and 7 is also present in Figure 8, where the ultrasonic standing wave has better filling properties than the perpendicular ultrasonic wave at the two lower aspect ratios, and where both the ultrasonic agitation techniques yield much better filling properties than conventional pumped agitation, especially at grooves with widths lower than 1 mm. The *U40L* experiment with *LF*-mode setup shows extraordinary filling properties at

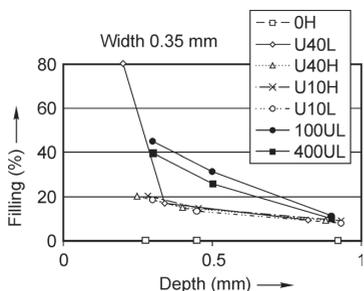


Fig. 8: Filling of nickel at the bottom of 0.35 mm wide grooves in percent, related to the nickel top surface thickness near the grooves

the lowest aspect ratio. This is due to the fact that the nominal depth is a little smaller in this case than for the compared grooves.

Figure 9 shows cross-sectional optical microscopy images of nickel plating in 0.55 mm wide and 1.5 mm deep grooves in experiments *0H*, *U40L*, and *400UL*. Experiments *U40L* and *400UL* reveal that *LF*- and *MF*-modes can give a smoothly deposited nickel at the bottom and at edges of trenches. Using conventional mode did not result in any deposited nickel at all at the bottom of the groove. This difference is likely to be caused by the ultrasonic influence of increased agitation in the grooves resulting in increased mass transport and reduced diffusion barrier thickness in the grooves.

Figure 10 shows cross-sectional optical microscopy images of nickel plating in 0.35 mm wide grooves of approximately 0.3 mm depth in experiments *0H*, *U40L*, and *400UL*. It can be seen that 0.20 mm deep grooves are totally filled when using *LF*-mode in experiment *U40L*, but when using conventional mode, the 0.25 mm deep grooves grow together in the top and encapsulate electrolyte in a cavity inside the groove.

Figure 11 shows cross-sectional optical microscopy images of nickel plating in 0.55 mm wide and 0.35 mm deep grooves in experiments *0H*, *U40L*, and *100UL*. The gaps between the plated top edges are close to equal or smaller in experiment *0H* with conventional mode compared to experiments using *LF*- and *MF*-mode. This means that gaps close at the same time or earlier during electroplating when

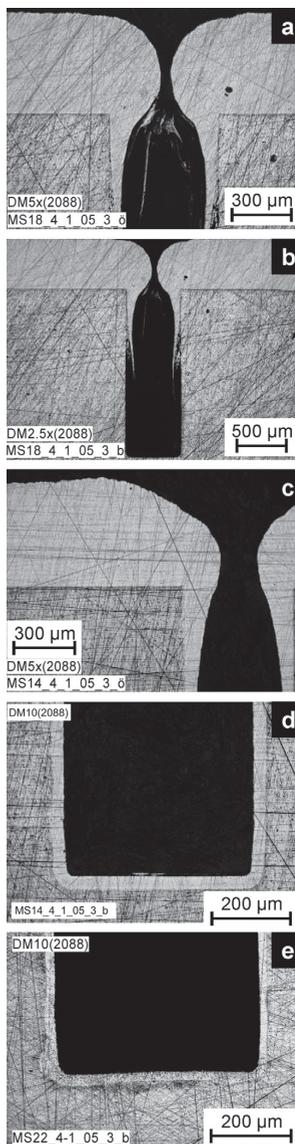


Fig. 9: Cross-sectional optical microscopy images of approximately 0.55 mm wide and 1.5 mm deep grooves in Quadrant 4 after nickel plating; a) and b): 6 m³/h flow and no ultrasound; c) and d): with 225 W ultrasonic agitation, 0.3 m³/h flow, and a distance of 40 cm; e) 400 kHz ultrasonic standing wave agitation with 0.3 m³/h pumped flow

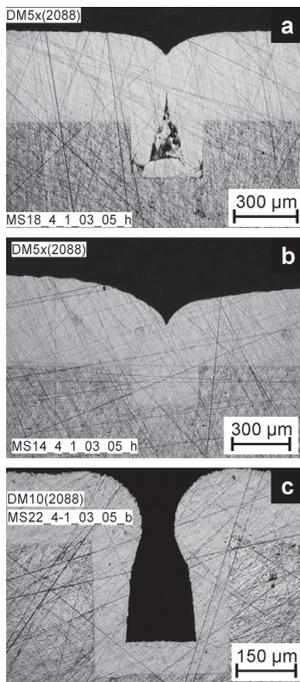


Fig. 10: Cross-sectional optical microscopy images of approximately 0.35 mm wide grooves in Quadrant 4 after nickel plating; a) 0.25 mm deep, 6 m³/h pumped flow and no ultrasound; b) 0.2 mm deep, 225 W ultrasonic agitation, 0.3 m³/h flow, and a distance of 40 cm; c) 0.35 mm deep, 400 kHz ultrasonic standing wave agitation with 0.3 m³/h pumped flow

using conventional pumped agitation compared to ultrasonic agitation, which increases the possibility to fill the grooves using ultrasonic agitation. Complete filling of grooves using *LF*- and *MF*-modes combined with pulse reversal plating or by using an electrolyte with proper leveling agents might be possible. It is clearly shown in *Figures 9 to 11* that the use of ultrasonic agitation produces far better filling of high aspect ratio holes, when compared to using conventional pumped agitation.

Additional pumped agitation as well as shorter distances from transducer to substrate seem to disturb the ultrasonic effect during plating with *LF*-mode.

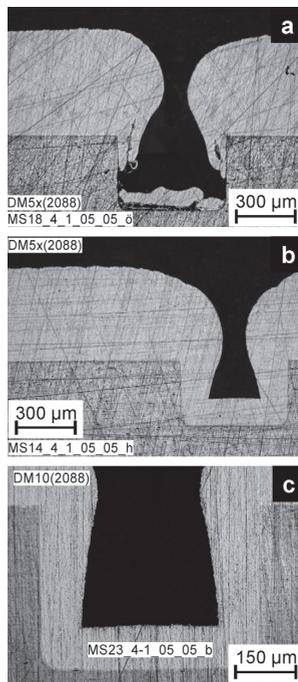


Fig. 11: Cross-sectional optical microscopy images of approximately 0.55 mm wide grooves in Quadrant 4 after nickel plating; a) 0.35 mm deep, no ultrasound, and 6 m³/h pumped flow; b) 0.35 mm deep, perpendicular ultrasonic agitation at a distance of 40 cm, and 0.3 m³/h flow; c) 0.39 mm deep, 100 kHz ultrasonic standing wave agitation with 0.3 m³/h pumped flow

When positioning the transducer too close to the substrate, the current field diverges around the transducer influencing the overall current distribution on the substrate. Higher ultrasonic power could increase the benefits of using ultrasonic agitation in *LF*-mode. However, increasing ultrasound power might also cause increased cavitation activity.

Summarizing *MF*- and *LF*-modes, both techniques appear beneficial to be developed further since both result in improved covering and throwing power in grooves. *MF*-mode yields better filling properties in lower depth grooves, but *LF*-mode is much easier to handle. The *MF*-mode technique has the disad-

vantage of needing adjustments in order to obtain a standing wave, and it is not easy to design a setup for more complex shaped substrates.

4.2 Surface Phenomena

Figure 12 shows that the nickel surface of experiment 0H plated with conventional mode is smooth and free of pinholes.

Smoothness of the substrate surface in experiments 100UL, 400UL, and U40L is comparable to 0H. Figure 13 shows the surface of nickel plated substrate in U10H. It is obvious that there are pinholes concentrated at the lower part of the substrate. Likewise, pinholes are observed to a smaller degree on samples U40H and U10L. The pinholes are caused by small gas cavities formed by the ultrasound, which stick to the surface of the cathode.

The bright patterns in Figure 13 originating from the holes on the surfaces of U10H are also observed with almost identical patterns on U40L, U40H, and U10L. They probably originate from clouds of cavitation bubbles, so-called streamer activity, a phenomenon especially pronounced at sharp edges and characteristic for gaseous cavitation. Since cavitation causes

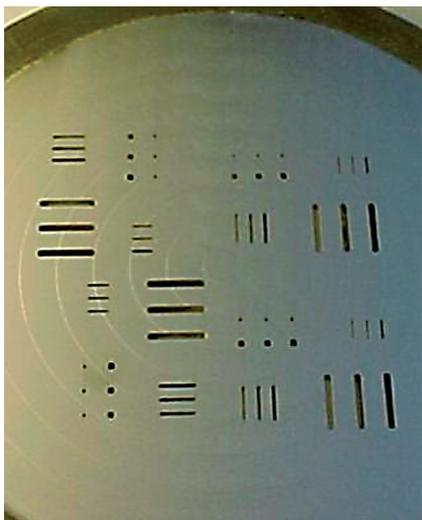


Fig. 12: Brass disc electroformed with nickel using $6 \text{ m}^3/\text{h}$ external pumped agitation

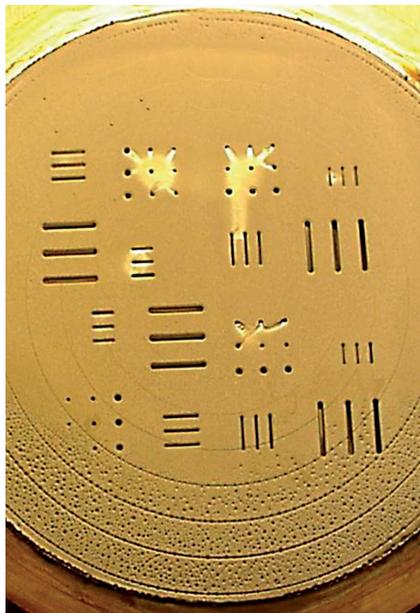


Fig. 13: Brass disc electroformed with nickel using 225 W ultrasonic agitation at a distance of 10 cm from the disc and $6 \text{ m}^3/\text{h}$ external pumped agitation

small shock waves, higher agitation occurs locally around small holes. A similar pattern is visible at the front of the 25 kHz ultrasound transducer.

5 Conclusions

Ultrasonic agitation has the ability to increase mass transport, thereby limit the depletion of metal ions in deep grooves during electroplating, and results in more uniform deposition. Compared to conventional agitation, the application of ultrasonic agitation during plating significantly improves filling in grooves of less than 1 mm width, with both the perpendicularly directed technique and the technique employing standing waves parallel to the substrate. The observed filling effect is strongest in the setup where 400 kHz parallel ultrasonic standing waves are used, even though the applied ultrasonic power is relatively low. When filling a 0.5 mm wide and 1.5 mm deep groove by electroplating using ultrasonically assisted plating, nickel is deposited all over the

walls and at the bottom of the grooves. For the case of pumped agitation, however, nickel deposition is obtained only at the top of the walls. The more powerful setup with 25 kHz ultrasonic agitation directed perpendicularly to the substrate heats the substrate to approximately 5 °C over electrolyte temperature. It might be possible to completely fill these grooves by using ultrasound combined with pulse reversal plating or with an additive containing electrolyte designed for leveling the surface.

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