

Uniformity Control of Ni Thin Film Microstructures Deposited by Through-Mask Plating

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Abstract

The thickness uniformity within a specimen and the cross-sectional profiles of electroplated individual Ni-microstructures have been investigated as a function of the electroplating conditions. It was found that the uniformity and profiles of microstructures could be controlled by varying the process conditions. A uniform thickness distribution and microstructures with flat profiles could be obtained at optimal plating conditions of $8\text{mA}/\text{cm}^2$ and 60°C . Above this optimal plating current density, the microstructure has a rabbit-ears profile and the thickness of a narrow microstructure is thicker than that of wide ones. While below this current density, the microstructure has a cap-like cross-sectional profile, and a narrow structure is thinner than wide ones. Increasing the plating temperature enhances the non-uniformity, whereas other process parameters have insignificant effects on it. The current crowding observed in patterned specimens is responsible for the rabbit-ears profile of individual microstructures, while a combination of the fluidic friction on the sidewall of the photoresist and the electrophoresis of the ions in the solution are believed to be responsible for the abnormal cap-like profile of individual microstructures and the thinning effect on narrow microstructures.

Introduction

Electrodeposition (or electroplating) has become one of the most important technologies for microelectronics and microelectromechanical systems (MEMS), as it produces high quality metal films in a simple way at a low-cost. Various metal and alloy films such as Ni, In, Cu, Au and NiFe, NiP, CoFe have been formed by electrodeposition.¹⁻³ A combination of photolithography technology and electrodeposition (so-called through mask plating⁴) makes it possible to fabricate microstructures suitable for microsensors and microactuators.⁵⁻⁷ However one of the problems associated with electrodeposition is the uniformity control of the electroplated films both in thickness and composition.^{8, 9} This affects the material properties and resulting performance of micro-components.

The thickness uniformity of electroplated metal-films was intensively studied during the 90's, especially for the microelectronics industry as Cu became the standard for interconnects in integrated circuits and also for the MEMS society as plated metal microstructures became popular microcomponents for sensors and actuators. The thickness variation of a Cu film on a Si-wafer typically has a "rabbit-ears" shape distribution (it is also called a nodule shape), i.e. it is thinner in the centre of the wafer and becomes thicker towards the edge.^{9, 10} The "resistive seed layer" model has been developed to explain the thickness variation on the wafer which is due to the voltage drop from the edge terminal contact to the centre of a wafer.^{9, 10} For patterned wafer plating, the uniformity has an effect not only on the thickness variation across a wafer, but also on the thickness of the individual microstructures, which develop rabbit-ears (or nodule) cross-sectional profiles.¹¹ The "active-area density" model has been developed to explain this behaviour, which it ascribes to current crowding at the edge of photoresist patterns.¹¹ Little attention has been paid to the effects of the process conditions on the profiles of microstructures and their uniformity across a wafer. In this paper we report on the optimisation of electroplated Ni microstructure profiles and thickness uniformity as a function

of plating conditions. A previously unreported abnormal cap-like cross-sectional profile for microstructures is found when they are plated at low current density, and the thickness of a narrow structure is much less than that of a wide one for the same plating conditions, which is significantly different from those structures with rabbit-ears shape profiles.

Experiment

Plating bath and solution----- The plating solution was commercially purchased from Celtic Chemicals Ltd, and consisted of nickel sulphamate (300 g/l), nickel chloride (10 g/l) and boric acid (40 g/l). The pH value of the as-made plating solution was 3.8, which gradually increased to around 5.0 after plating up to approximately one hundred times. A Ni plate with an area of $\sim 4 \text{ cm}^2$ was used as the anode. A magnetic pellet was used to stir the solution to keep the concentration uniform and to prevent the formation of hydrogen bubbles on the sample surface. The plating temperature was varied in the range 20 - 80 °C with $\pm 2 \text{ °C}$ accuracy, and the current density was varied in the range 1 - 30 mA/cm².

Sample preparation----- the through-mask plating technique was used to fabricate the Ni-microstructures. The process flow to fabricate the photoresist micromoulds was as follows: a Si-substrate was used as the plating base. It was first cleaned using a solvent in an ultrasonic bath, and was then rinsed in DI-water. A thin seed layer was deposited on a Si-wafer substrate by sputtering (CCR Sputtering system). The seed layer consisted of a combination of $\sim 5 \text{ nm}$ Cr and $\sim 50 \text{ nm}$ Cu. The thin Cr layer is to improve the adhesion between the Cu seed layer and the Si-substrate. The seed layer was then coated with multiple layers of photoresist (AZ5214) to obtain a thick photoresist micromould structure. Each coating was baked at 100 °C for 10 min. This was followed by exposure using an EVG620 aligner. Developing and rinsing steps followed. The final photoresist micromoulds were soft baked at 110 °C for ~ 10 min to minimize the delamination of the photoresist from the seed layer. Micromould structures of bars with various widths from 2 to 200 μm were used. The typical photoresist

thickness of micromould was $\sim 4 \mu\text{m}$. After electroplating, the photoresist was removed by immersing in acetone. The seed layer outside the plated Ni-microstructures remained on the substrates as the acid used to remove the seed layer also attacks the Ni microstructures and this would modify the profiles of the microstructures. More details of the process can be found in ref.12.

Measurements---- The microstructure profiles and the thickness were measured using a DekTak 8 profiler, which has a resolution of 1nm in thickness. A Digital Instrument AFM DI3100 was used to measure the roughness of the Ni-microstructures.

Results

a. Variation of microstructure profiles

Current density effect---- It is well known that electroplated microstructures tend to have rabbit-ears cross sectional profiles. For the Ni microstructures fabricated here, similar rabbit-ears profiles were observed when they were plated at a current density of $J > 12 \text{ mA/cm}^2$. However the profiles of microstructures can be controlled by varying the current density. **Figure 1 a-c** shows the profile variation of microstructures as a function of the current density. The thickness h of a microstructure has been normalized to h_{mid} , the thickness of a wide structure in the centre. It is apparent that the nature of the uniformity varies with current density, and that the width of the features affects the uniformity. The term of “a wide microstructure” used hereafter means that a structure has a typical width of $\geq 100 \mu\text{m}$; similarly a narrow microstructure means a typical width of $\leq 10 \mu\text{m}$. Those with a width between them typically have transient structures, and we will not discuss them here in details. At a plating current density $J = 30 \text{ mA/cm}^2$, the profile of a wide microstructure shows a typical rabbit-ears shape. The thickness of the plated microstructure at the middle is thinner than that at the edge. The typical variation in thickness across a wide structure is around 10 - 20 %. As the width of a given microstructure becomes smaller, the rabbit-ears profile gradually disappears, and the microstructure becomes thicker. However when the specimen was plated at a current density of

8 mA/cm², the wide microstructure becomes flatter with a uniform thickness from the edge to the centre of the microstructure. The thickness of a narrow microstructure is close to that of a wide microstructure. Further reducing the plating current density to 4 mA/cm² leads to a change in the cross-sectional profile and the thickness ratio. The profile of a wide microstructure becomes cap-like such that the middle of a wide microstructure is thickest, and becomes thinner towards the edge. Meanwhile the thickness of a narrow microstructure becomes much less than that of the wide ones with a value of typically ~ 80 % of the wide microstructures. It is therefore clear that the uniformity of microstructure profiles and the thickness can be altered by varying the plating current density.

In order to quantify the variation of the non-uniformity η upon the process conditions, we define the non-uniformity of a microstructure as follows

$$\eta = \frac{h_i - h_{mid}}{h_{mid}} * 100\% \quad (1)$$

Here h_{mid} is the thickness of a wide structure in the centre, and h_i is the thickness at the edge of a wide microstructure or the thickness of a narrow microstructure. **Figure 2** shows the non-uniformity of plated Ni microstructures as a function of the plating current density at T = 20 °C and 60 °C respectively. A negative non-uniformity means that the thickness of a narrow microstructure or the edge of a wide microstructure is less than the thickness of a wide microstructure in the middle, while a positive non-uniformity means the opposite. At low plating current density, the non-uniformity is negative where the film is thinnest for a narrow microstructure, and it becomes positive as the current density is increased. A near zero non-uniformity regime (a uniform thickness) for those plated at 60 °C exists at a current density of $\sim 8 \pm 2$ mA/cm². The non-uniformity over the measured current density range is ± 20 % of the thickness.

Temperature effect---- It was found that the uniformity and profiles of microstructures are strongly affected by plating temperature. **Figure 3 a & b** show the profiles of microstructures plated at $J = 28$ and 2 mA/cm^2 at $20 \text{ }^\circ\text{C}$ respectively. Comparing these profiles with those plated at $60 \text{ }^\circ\text{C}$ and similar current densities as shown in **Fig.1 a & c** (30 and 4mA/cm^2), it is clear that low temperature plating leads to a more uniform profile. The thickness of a narrow structure plated at $20 \text{ }^\circ\text{C}$ is only $\sim 10\%$ less than that of a wide one when plated at $J = 2 \text{ mA/cm}^2$; this difference is much smaller than those structures plated at $60 \text{ }^\circ\text{C}$. Similar results were also obtained for high current density plating. **Figure 2** also summarised the non-uniformity of microstructures plated at $20 \text{ }^\circ\text{C}$ at different current densities. Although the non-uniformity follows the same trend with the current density at different temperatures, i.e. it changes from a negative to a positive non-uniformity; non-uniformity becomes stronger when plated at a higher temperature. The thickness uniformity and the profile of a microstructure plated at $20 \text{ }^\circ\text{C}$ is generally better than those plated at $60 \text{ }^\circ\text{C}$ over the whole range of current densities used here. A higher temperature corresponds to a higher diffusion rate of Ni ions in the solution. This may enhance the current crowding effect in patterned microstructure plating, thus enhancing the non-uniformity of plated microstructures as was observed.

Other effects---- The effects created by other process parameters such as stirring speed, pH value and aging of solution on the uniformity of plated microstructures have also been investigated. It was found that the effects from these process parameters are not as significant as the effects of plating current density and temperature when the pH value is lower than 5. For a newly made solution, there was no visible difference in the microstructure profiles for plating with or without magnetic pellet stirring, when other parameters were fixed. The profile and the thickness variation were determined principally by the current density used, but when it was plated in an aged solution, which had a pH value of > 5 , the profile of the microstructure was typically asymmetrical as shown in **Fig.4**. The film on the side facing the on-coming flow

was typically thicker than that away from the on-coming flow, and the average plating rate was lower than those when a solution with a low pH value was used. Stirring the solution increased the diffusion of Ni-ions near the cathode, leading to a higher plating rate on the on-coming side. In an aging solution, Ni-ion concentration is lower as they are consumed gradually though there is supplier from anode, while hydrogen inclusion in cathode electrode gradually consumes acidic ions, leading to an increase in pH value (approaching neutral point pH~7). These factors may be responsible for the slow down in plating rate.

The uniformity of the microstructures as a function of the film thickness has also been investigated in the range of 0.5 - 5 μm . The uniformity does not show a clear dependence on the film thickness.

b. Thickness distribution within a specimen

The distribution of the thickness across a plated wafer had been studied by many groups.⁸⁻¹⁰ The thickness of plated microstructures on a wafer has been seen to have a distribution characterized by a rabbit ears shape. It is thinner in the middle of a wafer, and becomes thicker towards the edge. We have conducted similar experiments on specimens with much reduced areas of 2 x 2 cm^2 . For a specimen plated at high current density, a similar rabbit-ears thickness distribution was observed. However, when it was plated at low current density ($J = 2 \sim 4 \text{ mA/cm}^2$), the thickness variation across a specimen was reversed, and it was thicker in the middle of a specimen and became thinner towards the edge. **Figure 5** shows an example of the thickness distribution within a specimen plated at 4 mA/cm^2 . It is obvious that the thickness distribution is cap-like, significantly different from the rabbit-ears thickness profile. Similarly, an optimal condition should exist for a uniform thickness distribution within a wafer. The result also indicates that the cross-sectional profile of an individual microstructure is strongly correlated to the shape of the thickness distribution within a wafer. When a thickness distribution has a rabbit-ears shape within a wafer, the individual microstructure also has a

rabbit-ears profile. Similarly a cap-like shape in thickness distribution corresponds to a cap-like profile for individual microstructures. The correlation between the profile of microstructures and the thickness distribution in a specimen therefore may originate from the same source.

c. Surface morphology and roughness

AFM has been used to investigate the surface morphology of plated Ni films. **Figure 6** shows the topology of Ni films plated under various conditions. The surface of a Ni film plated at low T is very rough with RMS of 20 nm, but it becomes smoother with RMS of 11 nm as the temperature is increased to 60 °C. Further increasing the temperature to 75 °C leads to a dramatic increase of the roughness to an RMS value of 25 nm. **Figure 7** shows the roughness variation as a function of the temperature at a fixed current density of $J = 2\text{mA}/\text{cm}^2$. The increase in the roughness at 75 °C is due to the high plating rate induced by electrolysis process at this temperature.¹³ **Figure 8** shows the dependence of the roughness on the plating current density at a fixed temperature of 60 °C. The surface roughness of Ni films increases with increasing plating current, up to $J \sim 15\text{ mA}/\text{cm}^2$; further increase of the current density causes it to decrease. The initial increase in the surface roughness with current density is due to the increased plating rate in this small current range, where grain sizes grow bigger as current density increases. Further increasing the current density leads to a rapid increase in the plating rate; so there is insufficient time for grains to grow big. Thus the film becomes a fine-grained structure with a smooth surface.¹⁴ Similar effect was observed for those plated at different temperatures with the peak current shift to low side of current density when plated at a lower temperature. Similar surface roughness effects have also been reported by other researchers.¹⁵

Discussion

The active-area density model for 2-dimensional specimens has been developed to explain the profile of patterned microstructures plating.¹¹ The mechanism of this model is illustrated in **Fig.9**. Regions that are more densely populated with photoresist tend to attract a high current

density, and hence tend to grow a thicker film. This model can be used to explain the observation of rabbit-ears profiles of microstructures and the thickness distribution within a specimen when a high plating current density is used. For a wide microstructure, significant current crowding occurs at the edge, but not in the centre as shown on the left side of **Fig.9**. As the electroplating is proportional to the current density, a thicker film is deposited on the edge of a wide structure due to the increased current density, whereas the middle of the structure is thinner. As the microstructure becomes narrower, the current crowding becomes severer across the whole width of the patterned microstructure, and the effective current density becomes much higher than that in the wide structure. This leads to a thicker film for a narrow microstructure as shown on the right side of **Fig.9**. As to the thickness variation within a small specimen, the voltage drop due to resistive seed layer from the edge to the centre is negligible. The current crowding at the edge of the small specimen is believed to be the major cause of the thickness increases at the edge when it is plated at high current density.

The active area density is a measure of the physical layout of the photoresist patterns, so it is not associated with the potential strength applied. Current crowding should always exist for patterned wafer plating. Clearly this model is unable to explain the cap-like microstructure profiles and the thickness distribution within a small specimen, so there must be other mechanisms that are responsible for the slower plating on the edge of microstructures.

A combination of fluidic friction and electrophoresis effects^{16, 17} is believed to be responsible for this abnormal profile and thickness distribution. **Figure 10** schematically shows the effects of fluidic friction and electrophoresis on the profile of microstructures. The fluidic friction between the solution and the solid surface produces a thin boundary layer, which does not move freely. Ions in the boundary layer have a very small mobility, and the mobility of ions near the solid surface is almost zero, and gradually increases as they move away from the interface. As a consequence, there is insufficient supply of ions for plating in the boundary

layer regions. However ions outside the boundary layer can move towards the surface of the cathode under an electrical field (electrophoresis effect), providing a continuous source of ions for plating. Consequently for a wide micromould, the existence of the boundary layer only affects the plating on the edge of a wide microstructure, however for a narrow micromould, the boundary layer thickness is comparable to the dimension of the narrow micromould. Ions are unable to be transported to the cathode surface and the plating rate in this narrow structure is therefore affected significantly, leading to a much-reduced thickness.

The thickness of the boundary layer can be varied by applying an external potential and varying the ion concentration.^{16, 17} At a low current density (a low electrical field), the friction effect is dominant and a lightly crowded current density is not sufficient to compete with the friction effect, and so the plating rate on the edge is still small. As the current density increases (the field increases), the thickness of the boundary layer reduces, and the electrophoresis effect becomes stronger even for those ions in the boundary layer. A highly crowded current density leads to much faster plating at the edge; hence a thicker film is formed on the edge. These two effects are superimposed, and eventually form a rabbit-ears profile on the microstructure. A close examination reveals that a structure with rabbit-ears profile does not have a 90-degree sidewall, but has a slope that may correspond to the fluidic friction effect. A detailed theoretical study to develop a model is in progress to explain this abnormal behaviour.

So far, only the rabbit-ears profile structure and thickness distribution have been noticed and studied. The cap profiles of individual microstructures have actually been shown in many publications but without comment. For example, the cap-like profiles and cap-like pillars in electroplated microstructures were shown in refs.1 and 18, significantly different from those rabbit-ears profiles reported by others. The reason for not noticing this abnormal behaviour is that most of the electroplating was conducted at a relatively high current density, where the rabbit-ears profile dominates the microstructure profile.

The combination of these two models can also explain the effects of other process parameters qualitatively. As long as the ion concentration is sufficient to provide the plating reaction, the effect on the profile of individual microstructures and the thickness distribution is insignificant, as the profile is determined by the thickness of the boundary layers and the degree of the current crowding. Other causes that may be responsible for the abnormal profile include the potential perturbation by the photoresist sidewall and the impurities absorbed.

The thickness variation across a wafer and for microstructures with different widths is undesirable for device applications, as it would lead to a variation in device performance and characteristics. For Cu plating used in CMOS, the problem is less critical as the Cu film on a wafer is planarized by the CMP process. However the thickness variation across a wafer and for microstructures with different widths will cause problems for MEMS applications, as electroplating often is employed to construct active components in MEMS devices without planarization. A variation in thickness for narrow components of $\pm 20\%$ can lead to a large variation in MEMS performance. This is particularly important when the active structures are used as mechanical components where the spring constant has a strong dependence on the thickness of the structures.

Conclusions

The uniformity of electroplated microstructures has been investigated as a function of plating conditions. The following conclusions can be drawn;

- The uniformity and profiles of electroplated microstructures are controlled mainly by plating current density and temperature. Optimal conditions of 8mA/cm^2 and 60°C were obtained to fabricate flat cross-sectional profile microstructures and a uniform thickness distribution across a specimen.

- Plating at a higher current density produces microstructures with rabbit-ears shape profiles, and a narrow microstructure is much thicker than a wide structure when plated at the same conditions.
- Lower current density plating creates microstructures with cap-like shape profiles, and a narrow structure is much thinner than that of a wide microstructure when plated at the same conditions.
- Plating temperature has a significant effect on the profiles and the uniformity of microstructures, whereas other process parameters have little effect on them.
- The active area density model can be used to explain the rabbit-ears profile, while a combination of fluidic friction and electrophoresis is believed to be responsible for the abnormal cap-like profile of microstructures and the thinning effect for narrow structures.

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Figure Captions

Figure 1. Normalized microstructure profiles plated at a) $J = 30$, b) $J = 8$ and c) $J = 4$ mA/cm² at a fixed temperature of 60 °C. The wide microstructure has a rabbit-ears shape cross-sectional profile when it is plated at a high J , and the film of a narrow structure is thicker than that of a wide one. The profile and the thickness variation are reversed when it is plated at a low J . The wide structure has a cap-like profile, and the thickness of a narrow structure is thinner than that of a wide one.

Figure 2. The non-uniformity of microstructures as a function of the plating current density at plating temperatures of 20 and 60°C. The profile of a microstructure has a cap like shape (negative), and gradually shifts to a rabbit-ears shape (positive) as J is increased. The non-uniformity is more pronounced when plated at high temperature than those plated at low temperature.

Figure 3. The profiles of individual microstructures plated at $J = 28$ and 2 mA/cm² and 20 °C. Comparing with those plated at 60 °C with the same current density shown in Fig.1, it is clear that low temperature plating produces microstructures with better uniformity.

Figure 4. Asymmetric profile of a microstructure plated using an aged solution with a pH value > 5 . The film on the side facing the on-coming flow is thicker than that away from the on-coming flow.

Figure 5. The thickness distribution within a specimen with an area of 2×2 cm² plated at $J = 4$ mA/cm² and $T = 60$ °C. The thickness distribution has a cap-like shape, significantly different from those with rabbit-ears shape reported by others.

Figure 6. AFM topologies of Ni films plated at various current densities and temperatures, a) 2 mA/cm² and 20°C, b) 2 mA/cm² and 60°C, c) 15 mA/cm² and 60°C and d).

31mA/cm² and 60°C. All pictures have the same scale, x = 1μm/div, and z = 200nm/div.

Figure 7. The roughness of Ni films as a function of the plating temperature at a fixed current density of 2 mA/cm². The roughness decreases with increasing temperature up to 60 °C; then it increases at a temperature of 75 °C, a high plating rate is observed due to electrolysis.

Figure 8. The roughness as a function of the plating current density at a fixed temperature of 60°C. The roughness increases with the current density firstly, and then decreases as further increasing the current density due to the growth of fine-grained structure.

Figure 9 The “active area density” model to explain the thickness non-uniformity of a microstructure. The current crowding occurs at the edge of a wide microstructure and in the whole area of a narrow structure, leading to higher plating rates and thicker films in these areas.

Figure 10 The fluidic friction and electrophoresis effects model to explain the slow plating rate at the edge of a wide microstructure (left) and in a narrow structure (right). This boundary layer leads to insufficient supply of Ni ions, thus a slow plating rate and thinner film at the edge and in a narrow structure.

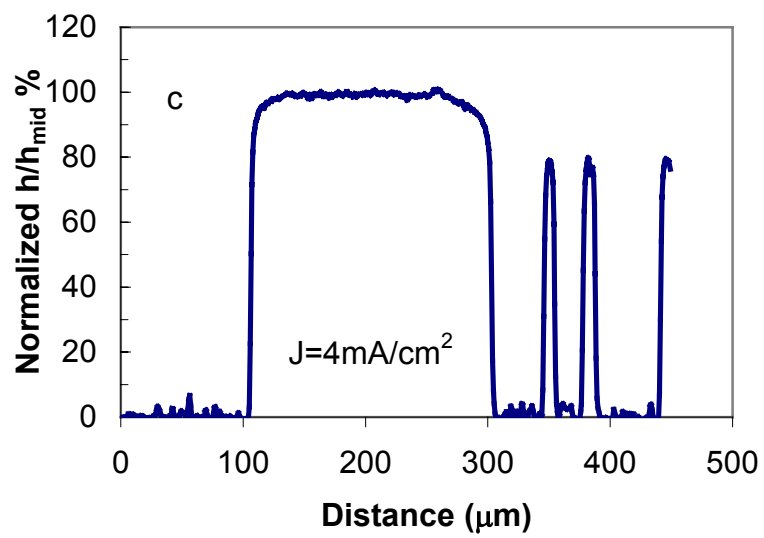
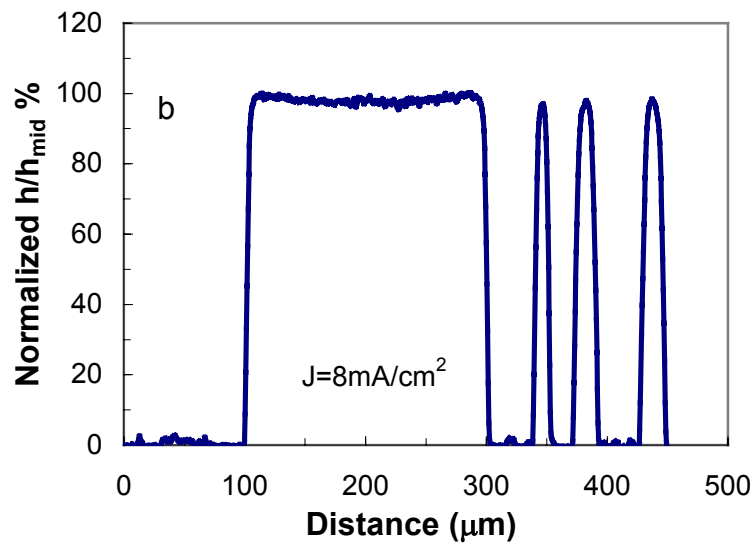
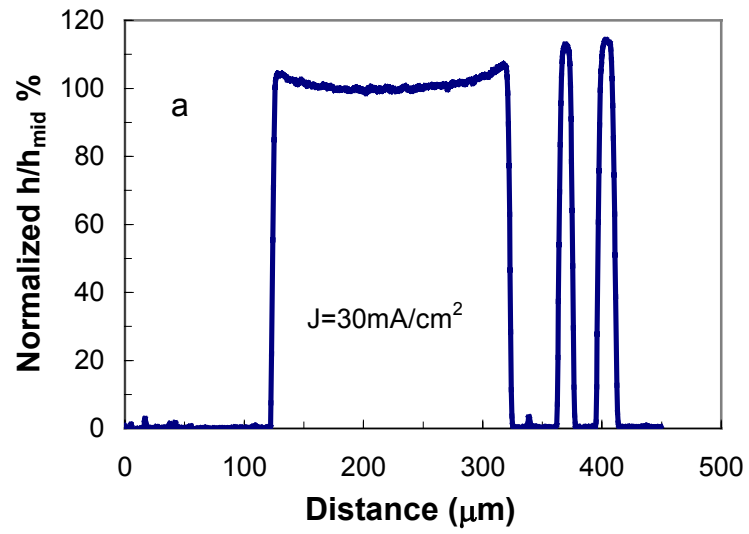


Figure 1 a-c

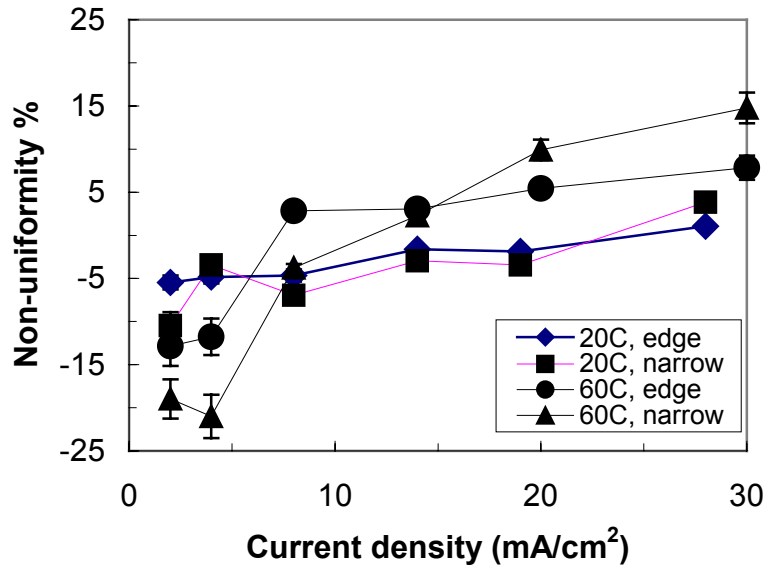


Figure 2

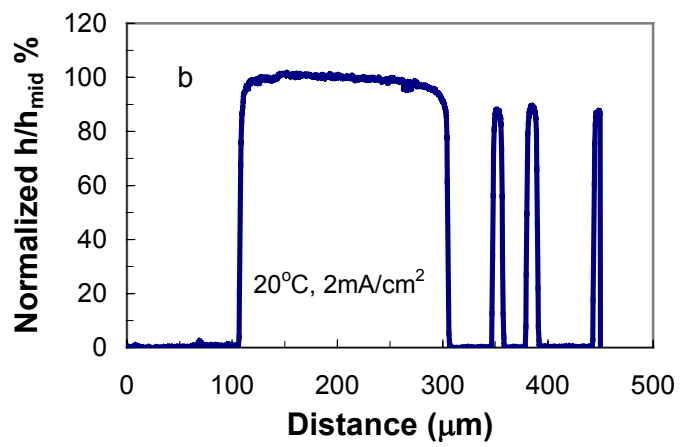
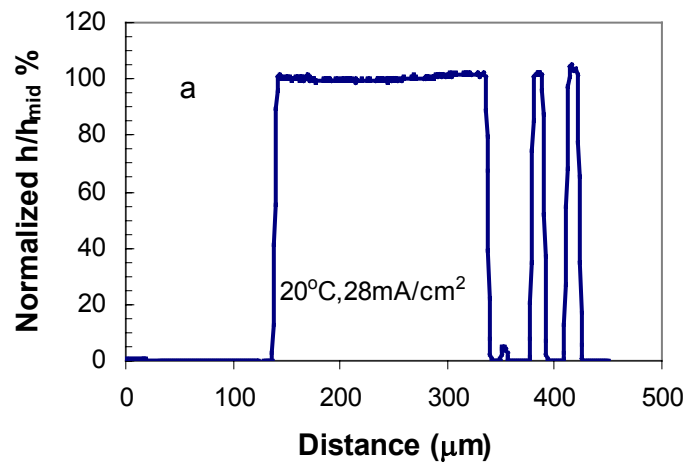


Figure 3 a & b

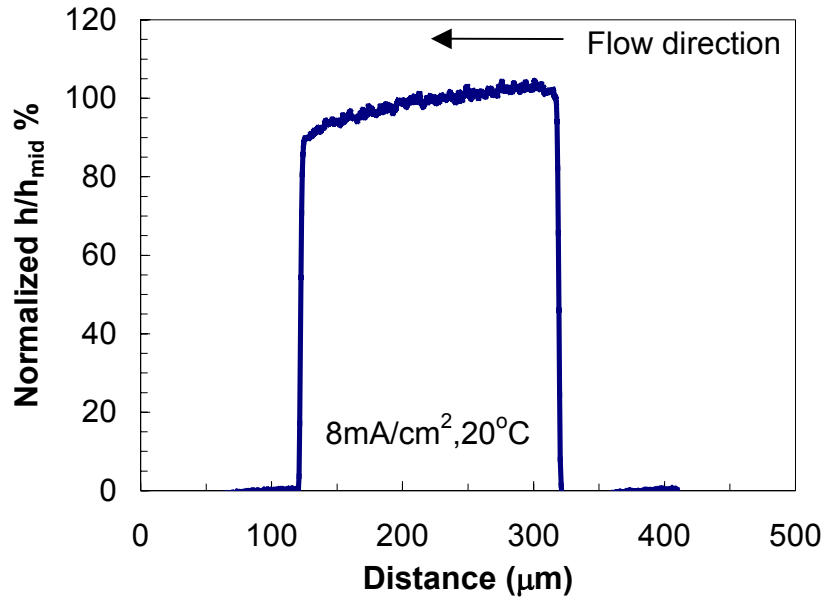


Figure 4

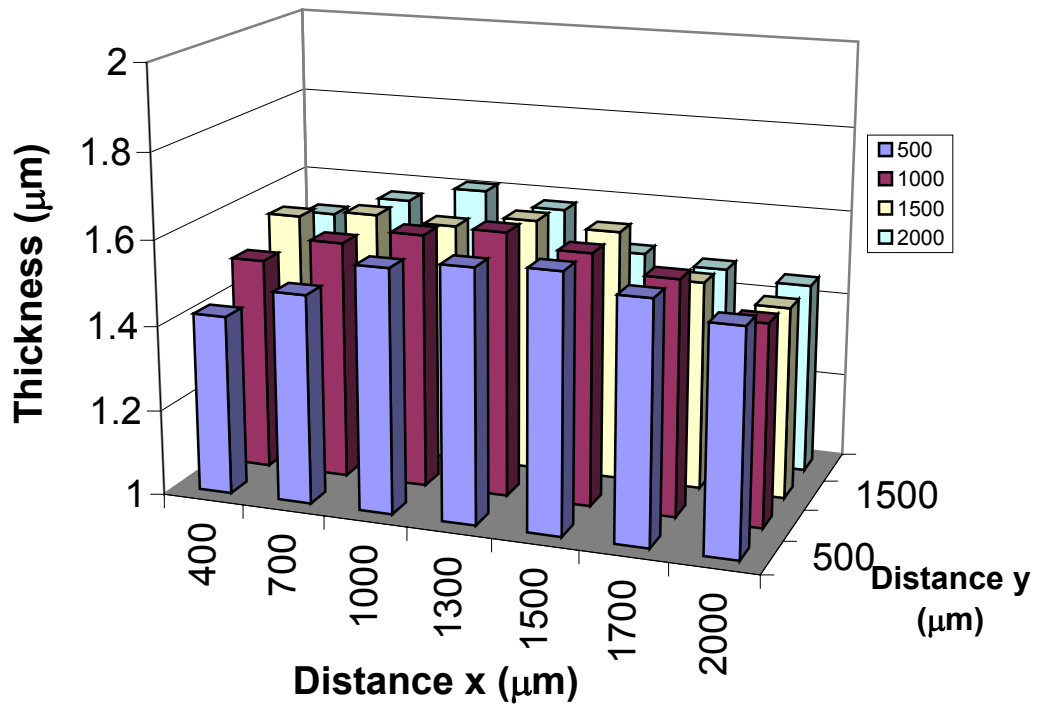


Figure 5

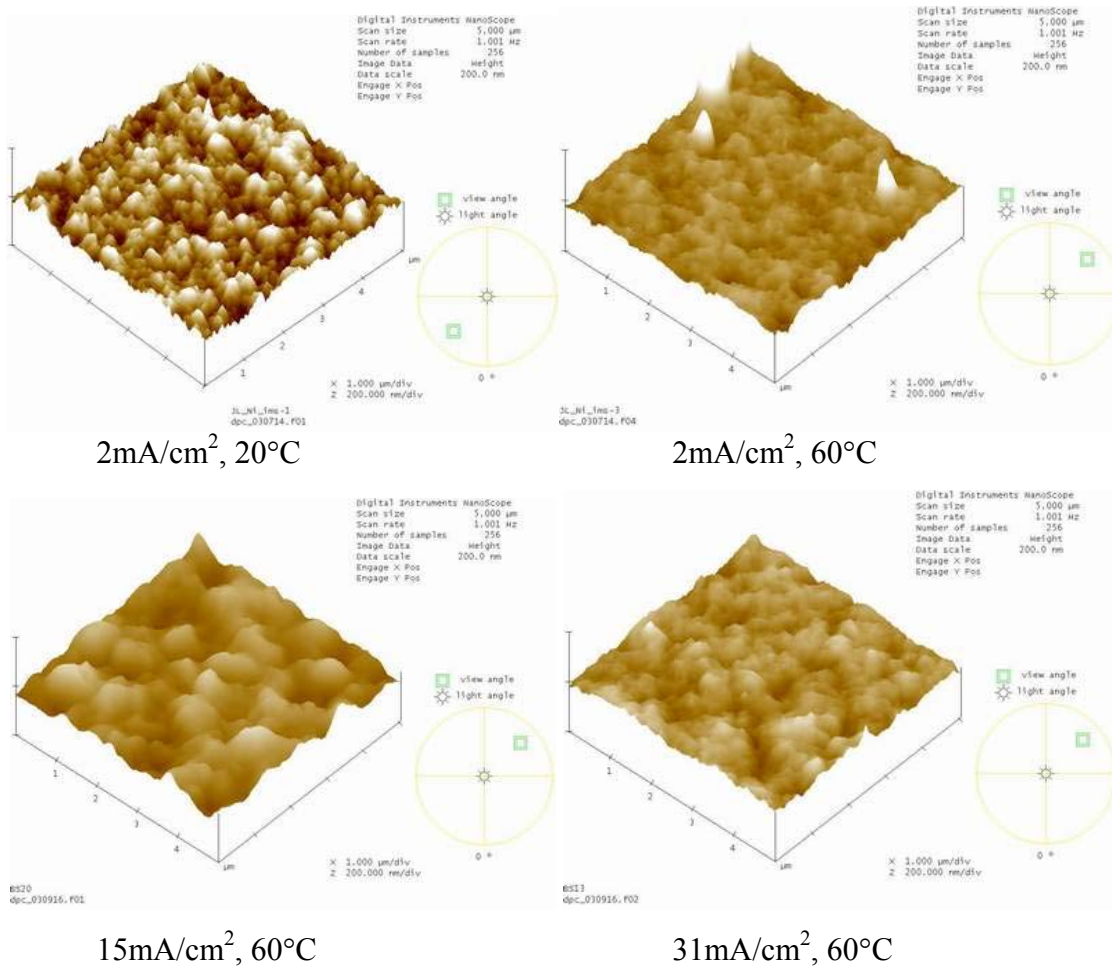


Figure 6

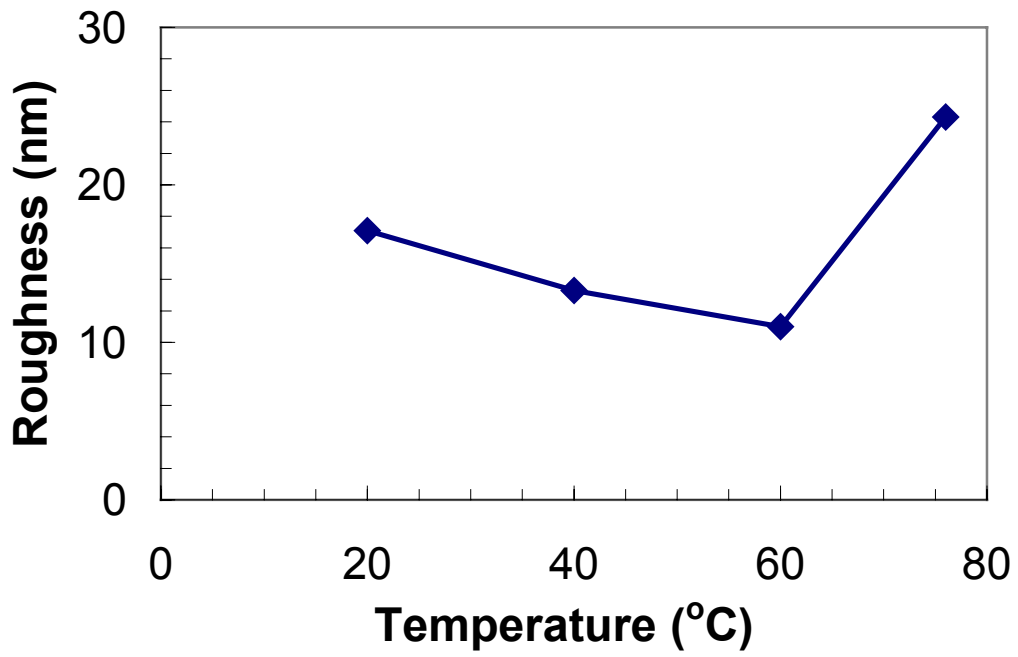


Figure 7

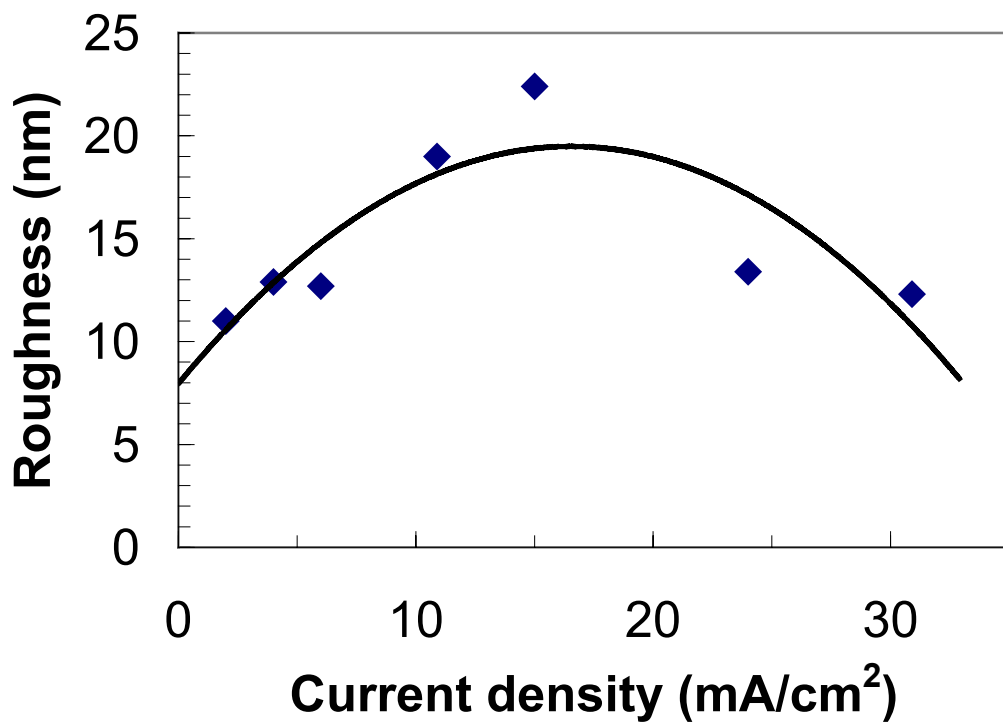


Figure 8

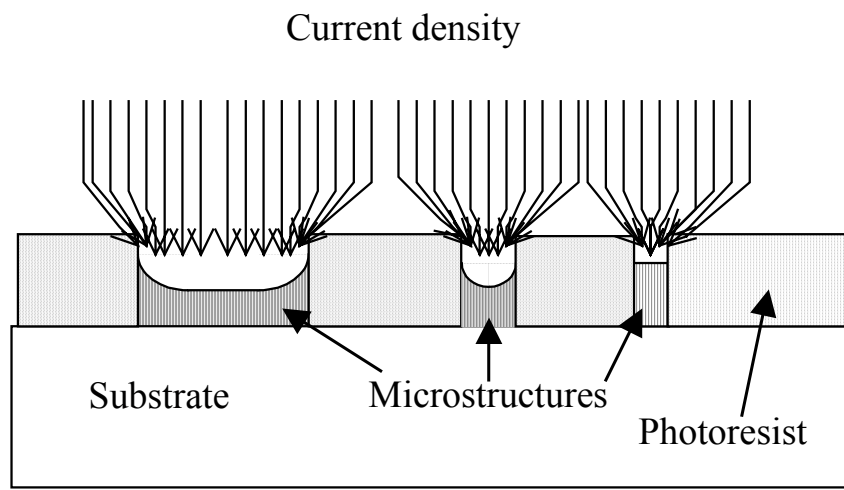


Figure 9

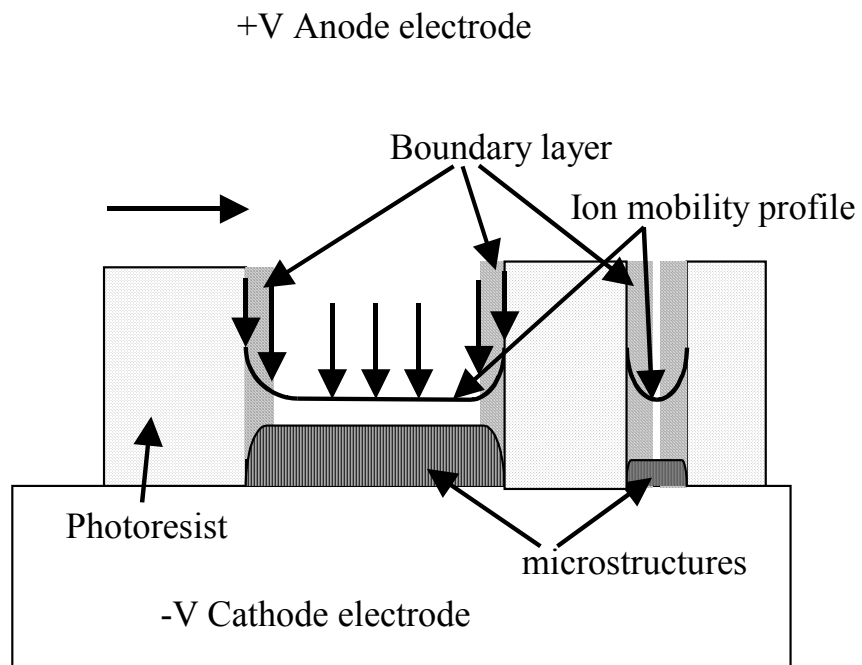


Figure 10