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Design of experiments in electrochemical microfabrication

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ABSTRACT

Electrochemical material etching techniques have attracted a significant amount of attention in the "wet" metal etching arena, as the process typically involves neutral salt electrolytes and is relatively safe to operate. There are also economical and environmental advantages associated with these techniques compared with competing etching methods.

A new concept of electrochemical microfabrication on substrates has been developed. In the technique the workpiece, which is the anode in the electrochemical reactor, is placed closely to a tool, which is the cathode containing the micro-pattern. Selective pattern transfer results in a higher etching rate on the areas opposing "exposed" regions of the cathode, and lower etching rates in the areas directly opposite to the areas, on the cathode, covered by an insulator.

In this investigation the electrochemical micro-patterning process has been evaluated and characterised in a vertical flow system described previously in literature. The experiments were carried out using copper disk anodes and patterned cathodes in a 0.1 M copper sulphate electrolyte. A 2^4 factorial experimental design procedure was adopted to determine the influence of process parameters on the electrochemical microfabrication process in terms of variability in pattern transfer over the electrode's surface area.

1. Introduction

Microfabrication processes have long been employed to make two- and three-dimensional structures in the micrometre scale [1]. It is also widely employed in the production of components in the microelectronics, optoelectronics, biomedical, and automotive industries. A typical microfabrication process to produce metallic microstructures on a substrate involves several processing steps. Typically, these are (1) the transfer of a master pattern onto the surface of the substrate by photolithography, to define the shape; (2) the metal, oxide or semiconductor is plated onto or etched into the substrate by additive or subtractive processes. In an additive process, material is added to the non-photoresist-coated areas of the wafer by metal-deposition. In a subtractive process, material is removed by wet chemical etching, dry chemical etching or electrochemical etching of the exposed areas of the wafer. Finally, the remaining photoresist can be stripped off [2,3].

In this paper we describe the application of design of experiments to a novel microfabrication method which eliminates the steps of photolithography on each substrate. The method is called EnFACE–*E*lectrochemical Patterning by *F*low and *C*hemistry. The technology's core concept is that it employs a single electrochemical dissolution step to transfer the pattern onto the substrate. This method utilizes the possibility of localising metal dissolution on an unmasked anode substrate in a suitably designed reactor with controlled fluid dynamics and electrochemistry [3–5].

In the EnFace technique, the pattern is defined on the surface of the tool using photolithography. The tool acts as the counter electrode in the electrochemical reactor, and is placed in close vicinity of the substrate, which is the working electrode. The schematic of the concept is shown in Fig. 1. A direct or pulse current is then imposed through an external circuit. As a result the metallic substrate is etched selectively opposite the exposed areas of the patterned cathode, hence replicating the pattern on the tool [4].

The electrochemical reactor, which can allow such pattern transfer processes, is based on a design which was previously constructed for wafer plating [6]. The electrochemical reactor consists of a rectangular flow channel, which houses two electrode holders, the electrolyte reservoir and a magnetic pump. The flow is controlled and monitored with a manual valve and flow meter, respectively. The anode and cathode are mounted onto the holders and electrolyte is circulated upwards through the channel. The channel gap is 3 mm everywhere except at the electrodes where it is 0.5 mm or less. This close proximity is achieved via chamfered electrode holders.

In an earlier work [5] the chemical and physical parameters, the combination of which, allowed for pattern transfer to occur

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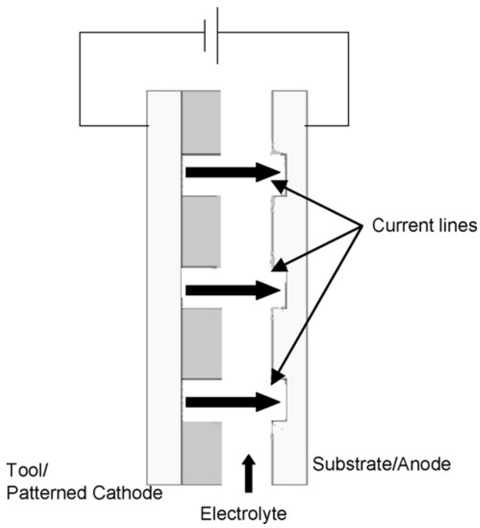

Fig. 1. Schematic representation of the EnFACE process. The patterned cathode carries the micro-patterns to be transferred onto the metallic substrate.

were found. These parameters were electrolyte composition/pH, total charge applied and electrolyte flow rate. However, it was found that etch depth as well as the area of the electrode where a pattern was transferred were a function of processing parameters. In particular, the reaction at the counter electrode, which was hydrogen evolution, caused gas bubbles to accumulate and cover the cathode surface. Since gas evolution is a function of applied current as well as the flow rate it is difficult to minimise it using the one factor at a time (OFAT) approach.

In addition, pattern transfer is also a complex process involving the generation of an oxide film on the electrode surface [4], and small defects in the metal surface can change the outcome of the process. Therefore, a statistical method was employed to relate electrochemical and engineering factors and their levels to output variability using a design of experiment (DoE) approach. The design focuses on identifying the factors and interactions between factors, which influence the variability by setting the parameters at different levels.

2. Factorial design of experiment

The EnFACE process can be represented by the model shown in Fig. 2. The process is realised as anodic metal dissolution, which transforms the input, in this case the copper anode, into an output, with the transferred pattern being the observed response. The process variables can be broken into controllable and uncontrollable variables, although the uncontrollable variables can be manipulated so that they are constant throughout the experiments. The controllable parameters, x, can be set at different levels (i.e. similar to state variable), which allows one to study the effect on the process output, y. Design of experiment [7] can be used to determine the influential factors, shown as x in Fig. 2 so that the process output (y) has a small variability. It was also used to determine the most significant factors and whether or not there are any significant interactions between them.

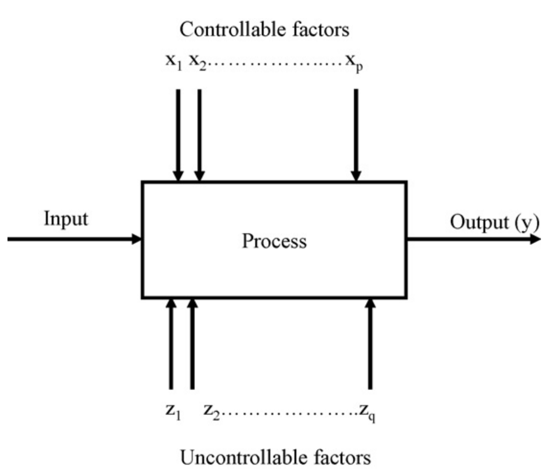

Fig. 2. Model of controllable and uncontrollable factors in a process and design of experiment.

Table 1
Factors used in the design of experiment of the electrochemical pattern transfer process.

Factor	Name	Unit	Low level (−1)	Centre (0)	High level (+1)
A	Current density	$mA\,cm^{-2}$	50	70	150
B	Time	s	30	90	180
C	Volumetric flow rate	$cm^3\,s^{-1}$	30	50	150
D	Feature density	%	30	50	70

The experimental design and the statistical manipulation of the data set were performed using a commercially available statistical package, Stat-Ease Design. The Stat-Ease package is statistical software, which allows multi-level factorial screening designs that can lead to process improvement. The software generates a randomised experimental procedure, which consists of every possible combination of factor levels. The software allows the user to set up and analyse general factorial, two-level factorial, fractional factorial and Placket-Burman designs [7]. With these designs, the user is then able to screen for critical factors and interaction. The software allows the user to layout a test plan, where each factor in the experiment is systematically changed to different levels. After the data acquisition step, the least-square regression technique is used on the randomised data set, to test the best statistical model/equation for the data set. This statistical model can then be used to draw contour plots showing the predicted values and how the process behaviour changes depending on the parameter setting.

The design of experiments procedure was adopted to determine the influence of process parameters on the electrochemical microfabrication process in terms of percentage pattern transfer on the electrode surface area. The effects of the process parameters on pattern transfer were quantified by performing 2^4 factorial experiments. In the 2^4 factorial designs, 16 experiments were carried out at upper and lower experimental levels, with an additional three experiments at centre of the factorial design. The concept of studying parameters at statistically different levels is integral to the design of experiment approach. Through the earlier studies on the EnFace process [5] we have gained an appreciation of the required "settings" for the electrochemical reactor to achieve pattern transfer, albeit with variable results. The factors were changed between three levels, high (+1), mid-level (0) and low (−1), as listed in Table 1.

The high-, mid- and low-level settings translate to controllable parameters for the etching process. A current density of 50, 70 and 180 $mA\,cm^{-2}$ normalised with respect to the apparent surface area of the electrode at the start of an etching experiment was used. A processing time of 30, 60 and 180 s corresponded to the low, mid and high levels of etching time. An electrolyte flow rate of 30, 50 and 150 cm^3/s was used in the factorial experiments. Finally, a feature density 30%, 50% and 70% of the apparent surface area was also examined. Specifically two sets of features were used in our experiments; linear features with 50 and 100 μm width and 100 μm × 100 μm square features with a pitch of 700 μm were used.