Chemical Machining of Monel

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Abstract: This study shows the effect of etching condition on microchannel of 100 µm width. Microchannels were fabricated on Monel 400 substrate by using different concentration of Ferric chloride ($FeCl_3$) and nitric acid (HNO_3) in water. Effect of exposure time on channel width and peel off resistance of photoresist is also studied. It is found that etching temperature has significant effect on undercut and depth of etch.

Keywords: ***Chemical machining, depth of etch, undercut, Monel 400***

1 Introduction

In recent years, chemical industry uses microchannel reactors for catalytic and non-catalytic reactions [1]. Currently, the microchannel devices are manufactured on materials like silicon, glass, copper etc. which cannot withstand a temperature above 300 °C. It is essential to fabricate these devices which can withstand higher temperature. Monel can be used as a substitute material for fabrication of microchannel reactors because it has better mechanical and thermal stability. Therefore, Monel 400 is chosen for this study. Generally, micromachining, laser machining, dry machining and wet chemical machining have been used for fabrication of microchannel [2-6].

PCM is a production technique for the manufacturing of stress-free, flat, mechanical components by selective chemical etching through a photographically produced mask. PCM process is used for fabricating thin gauge metal, ceramic or glass parts. Thickness of the part ranges from 0.025 mm to 2 mm depending on the type of metal. In PCM process, a stencil called photo tool, is used to expose multiple images of the parts to be made on a sheet of raw material that has been coated with a light sensitive and acid-resistant material, called resist. After the images of the parts have been developed, and the uncured “resist” is washed away, metal around the part is dissolved using an etchant [7].

Resulting metal from PCM has no burrs or deformations of the raw material. PCM provides a fast, flexible and relatively inexpensive way to produce a wide variety of precision metal parts. Phototool replaces conventional steel tools and dies. These tools can be generated rapidly and can be inexpensively regenerated. The photo tool operates like a stencil, so there is no “tool wear”. PCM is an attractive alternative for stamping, punching and machining of thin sheet metal parts [8].

Allen et.al. [9] Studied the characterization of aqueous $FeCl_3$ etchants used in industrial PCM. $FeCl_3$ is the most commonly used etchant. But there are a variety of grades of $FeCl_3$. P. Nageswara et al. [10] reported the effect of various concentrations of $FeCl_3$, HCL and HNO_3 in water for etching stainless steel. Davis et al. [11] used a PCM for precise etching of precipitation hardened stainless steel.

All the above studies focused on etching of different metals by using various combinations of aqueous solutions. In this study, $FeCl_3$ is used as an etchant and the effect of different etching concentration, etching temperature and etching time has been investigated.

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2 Experimental Work

The film strip phototool, as shown in Fig. 1, was used in the experimental study which consists of channels 100 µm width, and spacing between two channels was 1.5 mm. Phototool was printed on 4800 dpi by using offsetprinter.After the preparation of artwork, the widths of slots were measured by using Veeco white light interferometry.

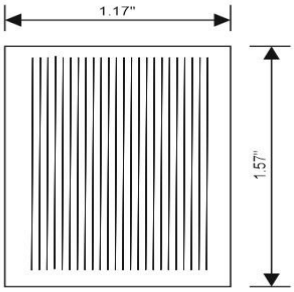

Fig. 1. Schematic diagram of experimental phototool

The specimen chosen for this study was Monel 400. Table 1 gives the chemical composition of Monel 400. The specimen is cut to size of 30 × 30 mm from 0.1 mm thin sheet. Initial roughness (Ra) of substrate was 183 nm. Before coating with photo resist, all specimens were cleaned. Each 30×30×0.1mm3 specimens were cleaned in ultrasonic cleaner. According to literature, cleaning was carried out at 80° C for 50 min.

Table 1 Chemical composition of Monel 400

Element	C	Fe	Mg	Ni	S	Si	Cu
% wt.	0.3	2.5	2.0	63	0.024	0.5	32

After ultrasonic cleaning, all the specimens were rinsed using dry air. The specimens were coated with LPR 1020 liquid film PR (negative type) by using a spin coater (Spectron-India). Specimens were coated at different spinning speed to achieve a specific coating thickness. Coating thickness was measured by using a contact type thickness gauge (Mextech CM 8823). Ultraviolet (UV) exposing was carried out by using six UV tubes of 365 nm wavelength.

Experiments were conducted to find the correct exposure time because over exposure and under exposure would result in different line width [12]. The exposure time for the proper polymerization of photoresist depends on thickness of resist coating and type and intensity of the light source used in exposure. Seven levels of exposure time 30, 60, 90,120,150, 180 and 210 seconds were used to investigate their effect on image width. The correct exposing time was established by measuring the line width under Nikon microscope. Scratch tester (Ducom India) was used to check the peel-off resistance of the photoresist. In the scratch test, a diamond stylus of 200 µm tip radius is drawn over the sample surface under a continuously increasing normal force until the coating detaches. Fig. 2 shows the set-up for scratch testing.

Fig. 2. Photographic view of scratch tester set up

Exposed specimens were developed for 40-70 sec and subsequently rinsed with clean water. Unexposed area of the photoresist is dissolved in the developer. After development of photoresist, the photoresist film was washed with distilled water to wipe-off residual developer solution and then dried by filter paper.
Experiments were carried out with different concentration of etchants in the range of 450 g/l to 650 g/l. To determine the effect of temperature, different etchant temperatures were used between 50 to 70°C. Also, the effect of addition of HNO_3 (30%) to $FeCl_3$ was investigated. Table 2 shows the experimental data of exposure test.

Table 2 Channel width and strength of photoresist obtained by different exposing time

Experiment No	Exposure time (sec)	Image width (µm)	Photoresist strength (N)
1	30	120.34	18.85
2	60	116.78	19.33
3	90	108.00	19.79
4	120	99.72	20.00
5	150	98.25	20.12
6	180	97.66	20.76
7	210	96.38	21.06

3 Results and Discussion

Fig. 3 shows the effect of exposure time on line width and strength of photoresist. With the increase in exposure time, the width of pattern initially decreases at faster rate because of the scattering and diffraction effect of light. It is observed that, for line width of 100 µm and 30 sec time, the blur image is observed and for 120 sec image with very fine feature was obtained. This is due to the fact that negative photoresist contains cross linkers which are activated during exposure. As exposing time increases, degree of cross-linking also increases. While developing the substrate, in case of lower exposure time, erosion of photoresist occurs which results in larger pattern width. As the cross-linking of negative photoresist increases with exposure time, the peel-off resistance of photoresist also increases.
Fig. 3 indicates that 120 sec exposure time is enough. When the exposure time is less, photo mask pattern has not absorbed sufficient UV energy. Hence, a developed photoresist pattern is more than desired one.

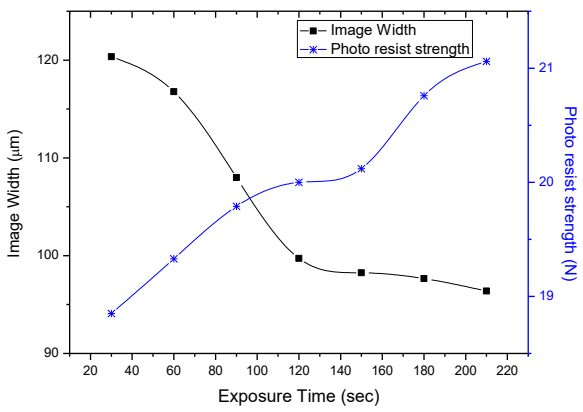

Fig. 3. Effect of exposure time on image width and photo resist film strength

3.1 Effect of Concentration on Depth of Etch and Undercut

Effects of concentration on depth of etch and undercut is shown in Fig. 4 If the concentration of FeCl3 increases, depth of etch and undercut also increases. Variation of undercut from 12.5 to 39 µm with increasing concentration from 450 to 650 g/l along with depth of etch variation from 12.73 to 17.13 µm is observed.

Therise in concentration of etchant favours the etching. But for higher concentration of etchant undercut is more and it affects the quality of etching.

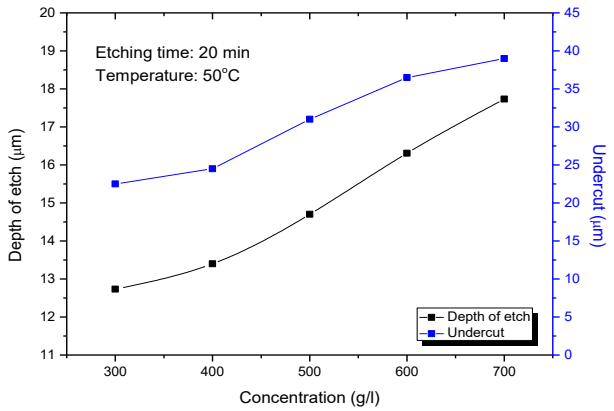

Fig. 4. Variation in depth of etch and undercut with concentration of FeCl3

3.2 Effect of Temperature on Depth of Etch and Undercut

Fig. 5 clearly indicates that with increase in temperature there is increase in undercut. There is increase in undercut from 16.5 to 149 µm at temperature variation from 50 °C to 70 °C. As the temperature increases, energy level of atoms of etchant (FeCl3) goes on increasing; hence there is increase in rate of reaction. Depth of etch also increases from 11.94 to 98 µm

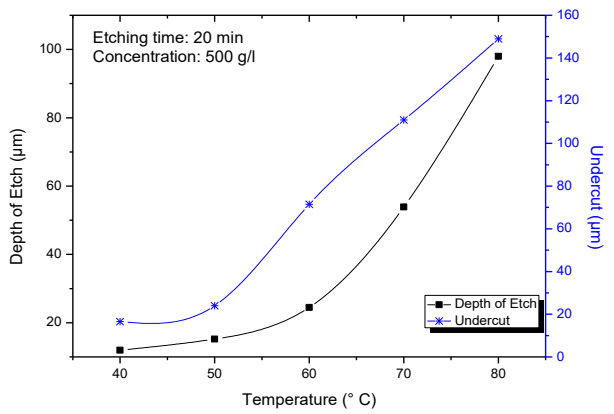

Fig. 5. Effect of temperature on depth of etch and undercut

3.3 Effect of Time on Depth of Etch and Undercut

Effects of etching time on depth of etch and undercut is shown in Fig. 6 Undercut is increased from 6 to 34.5 µm as time varies from 3 to 7 min. Also the depth of etch varies from 2.43 to 18.64 µm. with increase in time. Hence, etch rate of Monel 400 increases with respect to time.

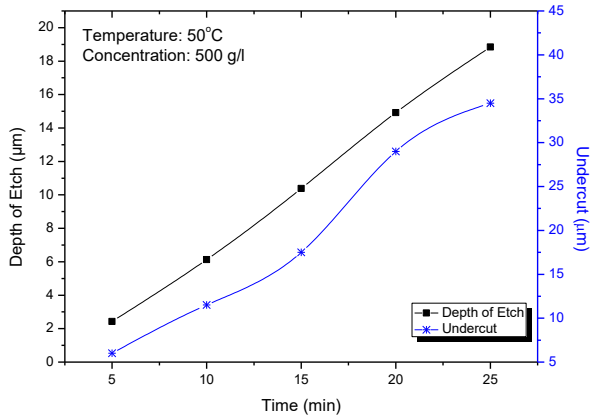

Fig. 6. Variation of depth of etch and undercut with time

4 Conclusions

Spinning time affects the photoresist film thickness, as the time increases thickness of the photoresist thickness decreases. Because of lower thickness, photoresist peels-off during the etching. Spinning time of 30 sec has photoresist thickness of 16.01 µm and goes on decreasing up to 12.89 µm for 70 sec.

- Strength of photoresist is checked and it is observed that 20 N force is required to scratch the photoresist coating for exposing time of 120 sec and image width of 99.72 µm.
- Because of the scattering and diffraction effect of light, width of pattern initially decreases as the exposure time increases.
- For line width of 100 µm and 120 sec exposure time image with very fine feature was obtained.
- Rise in concentration and time favours the etching. Increase in time and concentration increases depth of etch and undercut.
- Temperature is the major parameter which increases depth of etch and undercut. At 500 C, undercut of 16.5 µm and depth of etch 11.94 µm was observed and for 700 C an increase in depth of etch and undercut of 149 µm and 98 µm respectively is seen.

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