Photoelectrochemical Fabrication of Stainless Steel Filter Elements*

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Precision fabrication of stainless steel filters is described. Controlled depth (10 µm) of metal removal was achieved by an anodic dissolution technique. In addition, this technique gave a very smooth surface finish. The limitations encountered in chemical milling methods were largely overcome by this method. Unlike the conventional ECM technique, a plane cathode (instead of a square cathode) is used in conjunction with photolithographic masking because it is not possible to get the accuracy needed. Photolithographic masking and control of electrochemical parameters and proper choice of electrolyte operating conditions, and cell geometry.

Metal forming techniques usually involve physical action such as milling, gouging, drilling, grinding and punching. These techniques sometimes yield unsatisfactory results, especially when very thin, very brittle or very soft materials are being processed. Considering the effects of punching a piece of metal, it is seen that strains are caused when the die passes through the metal introducing changes in the grain structure. In soft and thin samples this leads to low yield.

Photochemical fabrication also known as photo-fabrication or chemical milling is used for accurate reproduction of components of metals and alloys. This method makes use of the art of photolithography and chemical milling.

Photochemical machining is primarily employed in the manufacture of flat components such as diaphragms, gaskets, shims, etc., where the process offers a faster and more economic alternative to fine blanking for prototype or low volume work.

It is commonly believed that the technique of chemical milling is not precise and is a second rate alternative to pressing or fine blanking and that production rates are unacceptably slow. Such opinions are probably formed from an experience of the chemical etching methods.

In this paper we describe the precision fabrication of stainless steel filters with controlled depth of dissolution, making use of photolithography and electrochemical dissolution with a specially formulated bath. In this method, limitations of the chemical milling methods are largely overcome by

* Dedicated to Professor K S C Doss on his eightieth birthday.

Stainless steel filter element for hydrazine filter

Hydrazine, the rocket fuel for altitude control of satellites, needs filtering like any other fluid to remove the possible entry of dust particles into the nozzle. Hydrazine, an extremely reactive fluid, corrodes the conventional organic or inorganic filter materials. This has necessitated this special filtering technique where stainless steel is milled (10 µm depth) to provide filter elements. Hydrazine is allowed to flow in an assembly of these elements which entraps particles above 20 µm (Fig. 1).
The specifications require that the channel depth be controlled to 10±2 μm. After etching the channel, the separations of elements from the stock sheet is not carried out by punching operations as it is likely to leave burrs larger than 10 μm on the circumference. Hence both the operations of partial metal removal and separation of stock are carried out by electrochemical dissolution.

Dimensions of the filter element are given below:

- Outer diameter = 18 mm
- Inner diameter = 12 mm
- Thickness of element = 70 μm
- Depth of channel = 10 μm ± 2 μm

Fabrication process

The following steps are required for precision fabrication of filter element by the photoelectrochemical method.

1. Preparation of an enlarged art work of the component.
2. Preparation of photographic masks for channels and for separating the filter element from the stock sheet.
3. Surface cleaning of stainless steel sheet to remove oil, grease, etc.
4. Sensitizing the clean sheet with photosensitive material (photoresist).
5. Exposing the sensitized surface in contact with the photomask (for channels) to UV radiation.
6. Developing the exposed surface in a suitable developer.
7. Selective anodic dissolution of the channels.
8. Stripping the photoresist.
9. Repeating steps 4-6 with the photomask for separating the filter element from the stock sheet.
10. Selective anodic dissolution.
11. Stripping the photoresist.

Why choose electrochemical dissolution?

Chemical etching produces at best surfaces with roughness of the order of 3-5 μm depending upon the choice of the etchant, the rate of the spray of the etchant, etc. The dissolution takes place by Preferential chemical attack of the etchant on the grain boundary or defect sites. The surface appears coarse grained or fine grained depending on the metallurgical status of the metal. In electrochemical dissolution a surface finish of better than 1 μm can be achieved. Hence the latter process is adopted.

Critical parameters in photoelectrochemical fabrication

The prime requirement for successful fabrication of Precision filters are:

1. The suppression of preferential dissolution of grain boundaries which leads to enhanced roughness.
2. No new irregularity dictated by the crystallographic factors should occur. This can be achieved by making use of electrolytes which are used for chemical/electropolishing studies.
3. The photoresist should not be chemically attacked by the solution that is used for electrochemical dissolution. Otherwise, there may be defects arising from peeling, lifting, exfoliation or pitting of the photoresist.

Electrolyte—The choice of the electrolyte for electrochemical dissolution is dictated by the above mentioned critical parameters. The close control of dissolution depends on current density (cd), time, temperature, addition agents, etc. Several baths were tested for their suitability for precision fabrication and it was found that phosphoric acid either alone or in combination with lactic acid, sulphuric acid or glycerol, works under a wide range of cd. Table 1 gives the composition and operating conditions for a few baths found to be useful. Since the current efficiency for dissolution under the optimum operating conditions is nearly 100%, the thickness of metal removed can be predetermined by the duration of electrolysis at constant current.

Cell design—The cell geometry is a crucial factor in achieving the desired result. Uniformity of dissolution depends on the uniformity of cd distribution which in turn is controlled by the cell design. It is found that cd distribution becomes more uniform if the electrodes fill

<table>
<thead>
<tr>
<th>Table 1—Bath Composition and Operating Conditions of Various Baths Used for Electrochemical Dissolution</th>
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<tbody>
<tr>
<td>A. ( \text{Phosphoric acid} ) = 63%</td>
</tr>
<tr>
<td>( \text{Sulphuric acid} ) = 15%</td>
</tr>
<tr>
<td>( \text{Water} ) = 12%</td>
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<tr>
<td>( \text{cd (A/dm}^2 ) = 1-4</td>
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<tr>
<td>( \text{Temperature} ) = 30°C</td>
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<td>( \text{Metal removal} ) rate at 2A/dm² = 0.4 μm/min</td>
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<tr>
<td>B. ( \text{Phosphoric acid} ) = 40%</td>
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<tr>
<td>( \text{Lactic acid} ) = 33%</td>
</tr>
<tr>
<td>( \text{Sulphuric acid} ) = 14%</td>
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<tr>
<td>( \text{Water} ) = 14%</td>
</tr>
<tr>
<td>( \text{cd (A/dn}^2 ) = 0.5-4</td>
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<tr>
<td>( \text{Temperature} ) = 30°C</td>
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<tr>
<td>( \text{Metal removal} ) rate at 1A/dm² = 0.2 μm/min</td>
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<tr>
<td>C. ( \text{Phosphoric acid} ) = 62%</td>
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<tr>
<td>( \text{Glycerol} ) = 27%</td>
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<tr>
<td>( \text{Water} ) = 10%</td>
</tr>
<tr>
<td>( \text{cd (A/dm}^2 ) = 1-3</td>
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<tr>
<td>( \text{Temperature} ) = 30°C</td>
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<td>( \text{Metal removal} ) rate at 2.5 A/dm² = 0.5 μm/min</td>
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The polishing rate cd

Electroplating of Both

Mc
electrochemical

counter electrode (in our case, cathode). It has been established that the entire cross-section and if holes are drilled on the counter electrode (in our case, cathode). It has been established that for the cell of size $500 \times 120 \times 120\text{mm}$ an interelectrode distance of $10\text{mm}$ and a number of equispaced $1/8\text{ inch}$ holes on the cathode give best uniformity in cd distribution.

The cell is made of fibre reinforced plastic (Fig. 2). Each cell could accommodate 20 anodes and 21 cathodes. The cathodes are lead sheets with holes drilled in it to improve cd distribution and permit mixing of electrolyte. The anode is $70\mu\text{m}$ stainless steel sheet covered with photolithographic masking. Each anode gives 16 filter elements. Both the surfaces of the anode face the cathode and hence dissolution takes place on both the faces. A schematic diagram of the cell is given in Fig. 2.

Photolithography and electrochemical dissolution— In each circular filter element (Fig. 1) the areas which should not be dissolved are masked precisely by photolithography. The channels on either side of the filter element are to be identical. This demands that masking should be done with appropriate registry marks so that the channels are identical on both sides. Kodak or Way coat photoresist is found to be satisfactory.

Masking ensures that channels of the required geometry will be created if the dissolution rate is uniform in the exposed areas. The cell geometry and the cathode (with holes) establish uniform cd distribution. Since the electrolyte and the operating conditions have been so chose to effect uniform and smooth dissolution at constant current density, anodic dissolution of the photolithographically masked sheet in the baths given above resulted in filters with good accuracy of channel depth.

The filter elements are fabricated in a two-step operation. The first step creates channels of $10\mu\text{m}$ depth by anodic dissolution. The second step which is also electrolytic dissolution helps to separate the filter element from the mother sheet. This two-step operation is necessitated by the fact that the material removal in each step is quite different. In the first step it is $10\mu\text{m}$ per side, whereas in the second step it is $35\mu\text{m}$ per side.

The cell described here produces 320 filter elements per batch. Total operation time per batch is typically 90 min. Employing a constant current powder supply and bath 1C (Table 1) we have produced by the process described in this paper about four thousand filter elements. Based on the metrology of these pieces, we find that the accuracy obtained for the depth of channels is better than $\pm 2\mu\text{m}$. Accuracy obtained in chemical milling of this job is $\pm 5\mu\text{m}$.

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