Quantified evaluation of machined surfaces on sintered porous stainless steel samples

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Abstract
A wide variety of shapes can be produced by powder metallurgy technology which deals with blending fine metal powder to produce low cost, near net – shape parts in large quantities. Most metallic and alloy materials can be used to manufacture elements with controlled porosity of various degree.

As described in this paper, sintered porous stainless steel filters, with different bulk porosity and void diameters, were used in machining experiments. Three different approaches were implemented: 1) micro electrical discharge machining (µEDM), 2) micro milling and 3) milling followed by chemical etching. The goal was to find out how much the surface open pore fraction is reduced by different machining techniques. Therefore, quantified evaluation of every process was done using 3D optical surface metrology equipment (Alicona Infinite Focus) and SPIP surface analysis software. Results presented in this paper are useful to understand how the porous material functionality (eg. fluid flow permeability through the surface) can be influenced when machining of a porous metallic material is needed.

1 Introduction
Generally, powder metallurgy (PM) includes metal injection moulding, high pressure powder compaction and sintering processes which are already highly industrially developed. However, industrial methods are likely to be too costly for prototyping purposes when only few parts and rapid geometry design changes are needed. In this case, machining the necessary shape straight out of the porous material piece provides faster results. However, the surface porosity depends on machining processes and therefore can be very different from the bulk porosity provided by
material manufacturer. It has been already shown, that original surface is sealed because metal is smeared over the pores making it impermeable. In other words, pores are closed due to plastic deformation coming from continuous pressure exerted by cutting tool [1, 2]. However, there are not many sources available which could give a sufficient quantified evaluation of the surface sealing by different machining methods. In this paper, the authors present how optical metrology equipment and surface analysis software can be combined in order to measure the surface porosity and estimate the possible changes in the functionality (eg. surface permeability) of material. The results given provide numbers of open pore surface fraction measurements on the original and machined porous stainless steel surfaces.

2 Experimental set-up

Material samples included two sintered stainless steel (316 L) porous specimens with different grain/pore sizes and different bulk porosities. Specimen Nr. 1 was produced using 125 - 180 µm diameter metal powder and had a pore diameter range between 3 – 25 µm. Bulk porosity (measured by weighing) of a specimen was ≈ 23 % (Figure 1 a). The porous metal specimen Nr. 2 had pore size in the range from 3.5 to 9 µm and 30 % bulk porosity, again measured by weighing (Figure 1 b).

![Figure 1](image)

**Figure 1.** Original surfaces of a) specimen Nr. 1, b) specimen Nr. 2.

To compare original and machined surfaces, it was necessary to perform experiments followed by analysis and calculations of surface coverage fraction by pores. After that three different process sequences, listed in Table 1, were explored. As mentioned before, surface sealing occurs due to the mechanical force coming from the cutting tool. Keeping this in mind, experiments included also “zero mechanical force” machining processes. Equipment with some important process parameters are listed in Table 1. In the first case, both material samples were machined by electrical
discharge milling process using energy index 105 and tool diameter of 300 µm. In the second case a 3 axis milling machine with 1 mm diameter end-mill tool was used for milling the surfaces of two samples. In the third case chemical etching by means of a solution consisting of 5 ml HCl acid, 5 ml nitric acid and 100 ml H₂O was applied after milling operation in order to re-open pores, but not as a tool to machine the surfaces of samples.

Table 1: machining processes and parameters.

<table>
<thead>
<tr>
<th>Process</th>
<th>Equipment, parameters</th>
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<tbody>
<tr>
<td>1) µEDM</td>
<td>Energy index: 105, electrode diameter: 300 µm</td>
</tr>
<tr>
<td>2) micro milling</td>
<td>3 axis milling machine with Ø 1 mm end-mill tool</td>
</tr>
<tr>
<td></td>
<td>spindle speed: 5800 rpm</td>
</tr>
<tr>
<td>3) µMilling / chemical etching</td>
<td>3 axis milling machine with Ø 1 mm end-mill tool</td>
</tr>
<tr>
<td></td>
<td>spindle speed: 5800 rpm</td>
</tr>
<tr>
<td></td>
<td>Etching bath: 5 ml HCl acid, 5 ml nitric acid and 100 ml H₂O</td>
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</tbody>
</table>

Open pore fraction calculations were done by analyzing the optical microscope pictures with SPIP software’s built-in ‘Particle and Pore analysis’ module. To make measurements reliable, the same rules were applied to the analysis and evaluation of the different surfaces. Machined geometries and accuracy control of equipment is not presented in the paper as the main interest was to look into specimen surface pore sealing and re-opening issues.

3 Discussion and results

Different results were achieved while performing techniques described in Table 1 under the same conditions. For the “specimen Nr. 1”, optical measurements and software analysis showed that ≈ 36 % of the material surface area was covered by pores. Micro milling operation almost sealed the surface by reducing pore coverage down 4% (Figure 2 b). After 1h of chemical etching, the surface open pore was increased to ≈ 10% (Figure 2 c).
After µEDM milling pore coverage was reduced to ≈ 11% (Figure 2 d). For the less dense “specimen Nr. 2” analysis showed that ≈ 56% of the original surface area was covered by pores (Figure 3 a). Micro milling operation almost sealed the surface by reducing pore coverage down 2% (Figure 3 b). After 45 min of chemical etching, the surface open pore was increased to ≈ 19% (Figure 3 c). After µEDM milling pore coverage was reduced to ≈ 24% (Figure 3 d). It was noticed for both samples that, longer etching time resulted in more open pore surface. However, etching (material removal) rate was not calculated during the experiments.

4 Conclusion
As shown by this experimental investigation, mechanical machining considerably reduced the surface porosity due to material smearing. However, the combination of milling with chemical etching allows re-opening a considerable fraction of the pores, which depends both on the etching time and on the powder size and bulking porosity. Alternatively, micro EDM milling was shown to maintain a sufficient degree of surface porosity. These process combinations can effectively be used to prototype porous metallic structures in a faster and simpler way than using conventional PM processes.

References: