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Microfabrication in Metals, Ceramics and Polymers

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Abstract—In this overview report fabriation of microconstructed devices and systems out of metals, ceramics and polymers will be presented briefly as well as bonding, sealing and packaging of devices. The main objective for the systems and divices covered here is chemical process engineering, thud, metal, ceramic, and polymer manufacturing techniques are considered, while silicon technology is itentionally left out, because this technology is normally not used in this tipoc. The text will only provide a rough overview onto the numerous techniques and technologies.

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The present review considers briefly the issues associated with fabrication of microstructured devices and systems from metals, ceramics, as well as bonding, sealing, and packaging of devices. The main objective for the systems and devices covered here is chemical process engineering, and, therefore, the author focuses on metal, ceramic, and polymer manufacturing technologies and intentionally passes over the silicon technology, since it is normally not used in such applications. The paper will only provide a rough review into the numerous techniques and technologies. More detailed information can be found in [1–5]. Details on the most common bonding and sealing techniques are reported in [4, 6].

Selection of Materials and Manufacturing Technique

The material used to manufacture microstructure devices depends strongly on the application. Process temperature and pressure range, corrosivity of the applied fluids, necessity of catalyst integration or potential catalytic blind activities, thermal conductivity, specific heat capacity, as well as electrical and other properties are predefined by the intended application. Moreover, the very design of the microstructures is also relevant. Not all designs and respecttive geometries can be realized with any manufacturing technique. Depending on the throughput of the application, a certain number of devices may be necessary, which also has an impact on the manufacturing technique. Some suit only for making one or a few devices, while others are applicable to mass production.

The manufacturing technologies can be divided into two main classes of processes given for all materials, specifically abrasive and generative manufacturing processes. Technologies like selective laser melting, embossing or molding belong to generative techniques, while precision machining or electro discharge machining are erosive techniques. However, in any case the surface quality generated by the manufacturing process must also be considered. This is quite obvious from the observation that a relative roughness of some ten micrometer does not highly affect the standard geometry with hydraulic diameters of some ten millimetres. The same roughness inside a micro channel providing a hydraulic diameter of about 300 μm is drastically influenced (cf. [4]).

Metal Devices

Like conventional process engineering, micro process engineering most commonly uses devices fabricated from metals and metal alloys. The material range involves noble metals, like gold, silver, platinum, rhodium, or palladium, other metals, from stainless steel to copper, as well as aluminium and Fe-Al-alloys or Ni-based alloys [1, 4–6]. The origins of most manufacturing technologies for metal microstructures are either in silicon device production or in conventional precision machining. The processes have been adapted and improved to reach the desired precision and surface quality necessary for microstructure devices. In most cases, more or less strong changes in the design of the device, the methodology of the process, and the manufacturing process itself were necessary to provide the accuracy and quality needed for microstructure devices suitable for process or reaction engineering. Rarely the same manufacturing process for macroscale could be directly used for microscale devices.

Almost all techniques used for microstructures in metal are abrasive. Techniques like punching are considered to be both non-abrasive and non-generative: Their function consists exclusively in forming a material.

Etching

Etching technologies are well-known processes from semiconductor production. Etching is also a cheap and well-established technology to obtain free form structures with dimensions in the submillimeter range for numerous metals. The technology is well-documented [1–5, 7]. A photosensitive polymer mask material is applied onto the metallic surface to be etched. The mask is exposed to light, UV light, or similar via a structure primary mask. Then the polymer is developed, the parts to be etched are removed, and, finally, the metal is etched. When etching techniques are used, two main points have to be considered. First, the aspect ratio (the structure width-to-depth ratio) can only be lower than 0.5 at the optimum for wet chemical etching. The minimum width of a structure is two times the depth, plus the width of the mask openings due to the isotropy of the etching in metal. This is not the case for dry etching technologies like RIE (reactive ion etching) or RIBE (reactive ion beam etching), where aspect ratios larger than 0.5 can be reached. But these techniques are only rarely used in machine metal microstructures, because they are quite costly and time- consuming. More details on the principle processes can be found in [1], where the processes are described within the silicon (or semiconductor) fabrication section; however, they can also be applied to other materials, if the etchants are chosen accordingly.

Second, wet chemical etching always results in semi-elliptic or semi-circular structures with a relatively high surface roughness (about a few microns), again due to the isotropic etching. Details of the etching processes and etchants can be found in [1, 4, 7, 8].

The so-called lamination process is another application of etching [9, 10]. Here, thin metal plates are chemically etched to form patterns. The thin strips are then stacked in a specific arrangement to form the desired microstructure, and bonded. This technology is well adapted for mass production and very cost effective. A detailed description can be found in [11]. This shim- or sheet-technology has become very popular for its easy application and many possibilities for more or less free design. Aside of chemical etching, spark erosion techniques (see below) and even punching was tried to manufacture single lamination sheets. Details on these and other manufacturing processes can be found in [12, 13]. Figures on laminated micro reactors are given in [12].

Machining

Not all materials can be etched. For example, most noble metals are resistant to this corrosive structuring processing. Therefore, precision machining may be used to generate microstructures of noble metals, as well as standard metal alloys like stainless steel, Ni-base alloys, or other metal materials. Precision machining can be performed by spark erosion (wire spark erosion, countersunk spark erosion), laser machining, or mechanical precision machining. The applied technology is strongly dependent on the material, costs arising from the process, and the required precision and surface quality. Mechanical precision machining involves milling, drilling, slotting, and planing, comparable to the techniques well known in conventional dimension machining.

Spark erosion and laser machining is suitable for any metal or conductive material. Mechanical precision machining and the tools suitable for this work depend on the stability of the alloy. Natural diamond micro tools are suitable for brass and copper, while for stainless steel and Ni-base alloys hard metal tools are needed.

The surface quality reached with these techniques is quite different, depending on the material, as well as on the machining parameters. Spark erosion techniques lead generally to a relatively rough surface. The surface quality obtained with laser ablation heavily depends on the material to be structured and on correct parameter settings.

With copper or brass as base material, the best surface quality is reachable with mechanical precision machining. An electro polishing step has to be used after machining to provide very good surface properties. Then, the surface roughness of down to 30 nm and below can be reached. Figure 1 shows an example of micro channels obtained by micro machining and wet chemical etching (see above). While the machined channels show a rectangular cross section, the etched channels are semi-elliptic. For all techniques, details can be found in [1, 4, 14–20]

Selective Laser Melting: An Example of the Generative Technology

A very special method to manufacture microstructures is selective laser melting (SLM). Named to be a rapid prototyping technology, it is one of the rare generative methods. The technique is completely different from the above-described abrasive techniques.

On a base platform made of the desired metal material (in many cases, stainless steel), a metal powder is distributed, e.g. by spraying the powder onto the platform. A focused laser beam is computer-controlled running along the structure lines given by a 3D CAD model. The metal powder is melted by exposure to laser, forming a welding bead. After the first layer of beads in the desired scheme has been generated, the platform is lowered by a certain value (i.e. about half the thickness of a single layer), new powder is distributed, and the process repeated. Thus, microstructures are generated layer by layer.

In principle, any metal powder can be used for SLM, as long as the melting temperature can be reached with the laser focus. Details on this relatively new technology can be found in [21–23].

Metal-Forming Technologies

Almost all technologies described above are suitable for prototyping or small series production only. Thus, a single device or a very small number of devices can be manufactured in this way. It simply takes a lot of time to manufacture large numbers of microstructures by laser ablation, wire erosion, milling, or SLM. This does not relate to etching: Here, large numbers of microstructure devices are very easy to generate. Therefore, this technique is also suitable for large-scale production of micro devices.

Another possibility to obtain large numbers of microstructures is embossing. As shown in [24], even microstructures with dimensions of a few tens of micrometers can be readily produced by the embossing technology. It must be taken into account that the material is pressed out of the generated microstructures and has to be pushed somewhere inside the complete design. Thus, the microstructure design to be generated should be appropriate to leave enough room for the material to be moved to.

To create not only channels, but also holes and throughputs, punching is a good technique even for microstructures. The key to this technology is a precise negative model which can be manufactured by, e.g., precision machining or spark erosion. In such a model, structures of different heights can be included to allow a very efficient mass production of microstructured metal sheets with holes, slits, and openings, combined with more conventional structures like channels and voids within a single working step. Thus, punching and embossing are gaining more attention now for cheap and

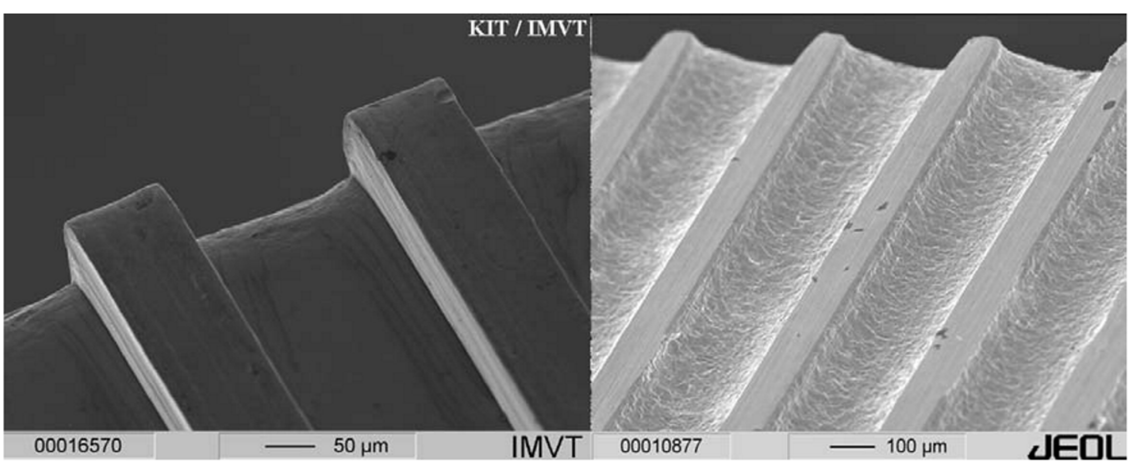

Fig. 1. Different cross-sections of microchannels obtained by (left) mechanical micro machining and (right) wet chemical etching.

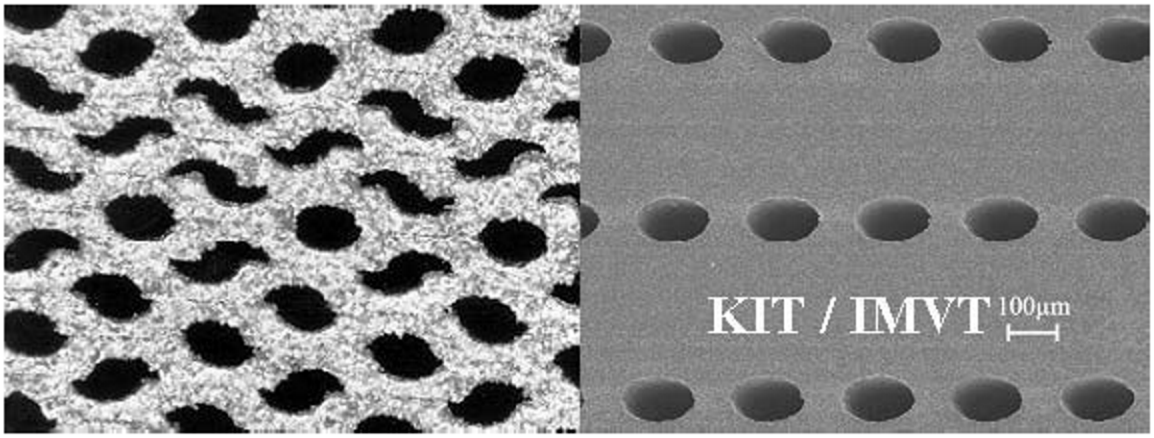

Fig. 2. Single channels generated by wet chemical etching in stainless steel: (left) incorrect alignment of etched microstructure foils, leading to non-elliptic microchannels after diffusion bonding and (right): correct alignment of etched foils, leading to fully elliptic microchannels.

easy serial production of device parts. The techniques can easily be combined to provide even more possibilities to generate microstructure foils or devices made of those.

Assembling and Bonding of Metal Microstructures

While in the macroscale world assembling a number of device parts is not really a problem, this step is more delicate to handle on the macroscale. Along with sealing, fixing, and bonding technology problems, the main point is the adjustment and alignment accuracy of the single parts of devices. Aligning errors may reach dimension values similar to those of the microstructures themselves, depending on the surface quality and the bonding technology applied. An example is shown in Fig. 2. Here, a number of microstructure foils with wet chemically etched micro channels have been aligned to form fully elliptic channels. The semi-elliptical shape of the channels is well shown (see above).

Misalignment will lead to non-regular channels, and, therefore, may interfere with bonding. Only small deviations from the desired elliptical shape will arise in the case of correct alignment, and the distortion on bonding will be low. The alignment techniques used to avoid errors can be simple mechanical methods (e.g. use of alignment pins), edge-catches in a specially designed assembling device, or optical methods like fully automated alignment by light diodes or laser. Most of these methods are coming from the silicon processing technology, where precise alignment of multiple mask layers is needed to guarantee functionality of the manufactured devices [1, 3].

On the microscale, burr formation in mechanical micromachining or laser machining may entail significant problems with assembling device parts, as well as with bonding. A burr on top of the structure edges will lead to slight deformations or an uneven contact of the single layers. Thus, special attention has to be paid to burr microstructures or to avoid burr formation.

Connection and bonding of metals can be accomplished by numerous techniques. Common for microstructures is either low-temperature or high temperature soldering, welding (laser, e-beam, WYG), and brazing or diffusion bonding. Even gluing and clamping, including different sealing techniques and gaskets, may be options, if the temperature and pressure range of the application allows these techniques. Details on the processes can be found in [1, 2, 4, 5, 25–33]. It is obvious that the choice of the bonding technique has to be made with a view to the process parameters the device should handle later on. A device bonded by low-temperature soldering is impossible to run at some hundred degrees Celsius, and, likewise, a glued device will not withstand an absolute pressure of some hundred MPa. For high-temperature and high-pressure applications, mostly welded, brazed, or diffusion- bonded devices are suitable. Therewith, diffusion bonding is a preferred technique, because it creates a monolithic device, if done correctly. Devices obtained by this method can withstand very high pressures of up to some hundred MPa [4, 5, 9, 10].

Ceramic and Glass Devices

Microstructure devices made from ceramics and glass can be applied at process parameters reachable neither with metals nor with polymers. High temperatures up to 1000°C and more, no catalytic blind activity and easier integration of catalytically active materials in the case of porous ceramics makes ceramics a very interesting material. Glass is chemically resistant against almost all chemicals and also features a good resistivity at elevated temperatures. In addition, the optical transparency of glass leads to a number of very interesting possibilities, like UV-intensified or photo chemistry. A closer look inside several fluid dynamic and process parameters by online analytical methods using optical fibres or high-speed visualization, μ PIV or fluorescence photometry is also possible. However, micro fabrication of components made from glass and ceramics is limited to some known technologies and is generally not very cost-efficient.

Ceramic Devices

The conventional way to obtain ceramic microstructures is to prepare a feedstock or a slurry, fluid or plastic molding, injection molding or casting (CIM, HPIM, tape casting), demolding, debinding, and sintering. Most ceramic materials will shrink, while the sintering process takes place. Therefore, a certain dimensional tolerance should to be included. Solid freeform techniques like printing, fused deposition, or microstereolithography are also possible with ceramic slurries, but will, in most cases, lead to a single prototype device only. There are certain ceramic materials which can be mechanically machined. Details on these manu-facturing processes can be found in [6, 34–46].

Selective laser melting (SLM) was also tried recently to comply with ceramics, and proved to be working well. The principle of this technique is similar to stereolithography which was described before. First experiments have shown promising results [23].

Independently of the manufacturing process, the grain size of the ceramic powder used to generate the precursor or the slurry has to be small enough to reproduce precisely all details of the desired microstructure. Even after sintering which is normally accompanied by grain coarsening, the grains should be at least one order of magnitude smaller than the smallest dimension of the device. This means that for a minimum dimension of about 50 μm, the grain size should be below 5 μm. Additives, too, play an important role in the manufacturing process. Removing additives in a wrong way may lead to distortions and cracks, or even to delamination of microscopic parts of the desired microstructure device. This is also true for the speed of additive removal. If it is done too fast, cracks and ruptures in the structure will most likely occur. Densification of the material is achieved by sintering, for alumina e.g. at a temperature of about 1600°C, while zirconia will need temperatures around 1500 C only. The temperature depends strongly on the grain size. It can be reduced drastically by using nanoparticles.

However, the microstructure design is the most critical point. Due to the specific properties of ceramics, it is not suitable simply to transfer the design of metallic or polymer devices to ceramics. Special needs for sealing, assembling and joining, as well as for interconnections to metal devices have to be considered. Especially critical is connection to metal devices, because ceramics is a brittle material and has a thermal expansion coefficient, in most cases, different to that of metals. Therefore, thermal cracks may be generated at the interconnection. Guidelines for the microstructure design in ceramics are still missing, and experiences obtained with macroscopic devices cannot be transferred directly down to microscale [6].

Another possibility to apply ceramic materials is the use of coatings and foams inside of, e.g., metal microstructure devices. Well-known technologies like CVD processes, sputtering, electrophoretic deposition, sol–gel methods, or generation of open foams in combination with spin coating, dip coating, or wash coating methods are feasible here. If the base material of the devices is an aluminum alloy, anodic oxidation will lead to either dense, protective ceramic coatings or porous layers which can serve as catalyst support. Ceramic foams can be inserted into metal or polymer microstructure devices to enhance the surface area, act as catalyst supports, or even work as heaters, if the electrical resistance of the ceramics is modified by, e.g., incorporated metals, or by the use of SiC as base material. Details for these processes can be found in [34–47].

Joining of ceramic materials should involve materials with similar properties as those of the base ceramics. Especially the thermal expansion coefficient is a crucial point while joining either ceramic materials

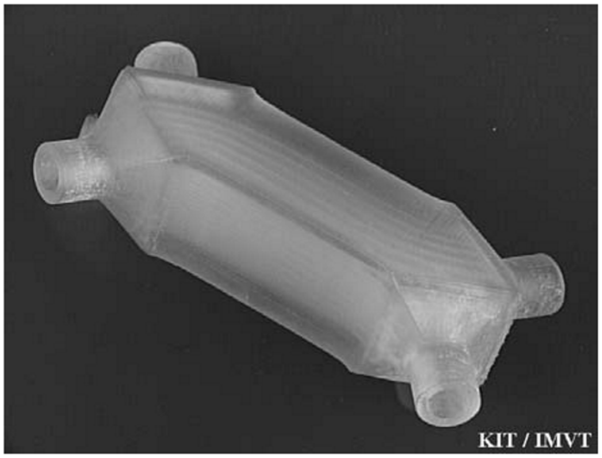

Fig. 3. Counterflow-design microstructure heat exchanger made by microstereolithography.

to each other, or, even worse, joining ceramics to metals.

Ceramic materials are ideally joined to each other in the green state before firing. While the firing process takes place, the ceramics is bound together tightly to form a single ceramic body from all parts. The pre-condition for this is that the parts are made from the same or similar ceramics, providing the possibility to be joined without cracking.

Another possibility is the soldering with, e.g., glass-ceramic sealants or glass-metallic sealants. Here, the working temperature of the device is limited by the melting temperature of the sealant. Reversible assembling and sealing with clamping technologies or gluing is also possible. Conventional seals like polymer rings or metal gaskets may be used as well as in metal technology. The adaptation of ceramic microstructure devices to metal process equipment should be done as far away from high temperatures as possible. Due to the very different thermal expansion coefficients of both material classes, problems will most likely occur here. If the connection has to be made close to thermal gradients, the sealing used should be designed to minimize tensile stresses as good as possible, as it is done for SOFC applications. For more details, see [1, 6, 34–47]

Glass Devices

Glass is an especially interesting material for microstructure devices due to its high chemical resistivity and optical transparency. The techniques available to microstructure glass are few in number [6, 48]. The most common are wet chemical (isotropic) etching using hydrofluoric acid and sand or powder blasting. Laser micromachining is also possible: In this case, evidence has to be obtained that the wavelength of the laser is absorbed by the glass and not simply passing through it. Aside of these techniques, a photosensitive glass is available, which is structured either by exposure to UV light followed by etching or by direct laser patterning. A photochemical reaction takes place in both cases, changing the structure of the exposed glass areas to be dissolved afterwards in HF. The exposed area is etched faster than those not exposed. While for photosensitive glasses like FOTURAN an etch mask is needed, laser patterning can be performed by pulsed UV laser without a mask but controlled by a CAD model, a process similar to stereolithography [48–50].

Bonding of glass devices is a little easier than for ceramics. Glass parts of single devices can be joined together by diffusion bonding, anodic bonding, soldering, or gluing. Soldering in this case means that the solder is glass, too. Connection to non-glass materials like ceramics, silicon or metal is done by anodic bonding, gluing, sputtering, or electroforming. Again, the most critical points here are the brittle glass material and the thermal expansion coefficient which differs between the materials to bond. Thus, the above-mentioned remarks for ceramics are also valid for glass [48, 49].

Polymer Microdevices

Polymer materials are widely used in conventional process engineering. It is, thus, an option to think about possibilities to apply polymers to microstructure devices or manufacture such devices completely out of polymers. Obviously, the use of polymer devices much more depends on the parameters of the process the device should be applied in than any other material. High temperatures normally prevent the use of polymers. There are some high-tech polymer materials to withstand temperatures up to 400°C and more, but they are costly and not easy to machine or to form.

High pressures may also be a reason not to use polymer materials, as well as the use of solvents is unsuitable with plastics. Thus, polymer materials are much more difficult to apply to process engineering than other material classes.

Most applications for polymer microstructure devices are found in the lab-on-a-chip technology, biotechnology, or medical research and development, where the low temperature and low pressure resistance is no concern, but the cheap mass production options are advantages for the generation of disposable devices. Another major advantage of polymer materials is optical transparency, useful in several analytical applications [51–55].

Various manufacturing techniques for polymers are known in conventional macroscale equipments. Again, two possibilities of processing have to be considered, abrasive techniques (machining, laser ablation) and generative techniques (embossing, injection molding, micro stereo lithography), which will not be separated strictly in this discussion.

Widely used are injection molding and hot embossing. Both technologies are also common for microscale devices. These two manufacturing techniques are very well established to mass and series production of devices [1, 6, 51–55].

Aside of the manufacturing techniques described so far, there are some others not so common, especially for mass production. Among those, the best known is laser machining. Different possibilities are existing, mostly excimer or UV-Nd:YAG lasers pulsed at high frequencies [56–58].

Another very interesting technology for generation of small numbers of prototypes is microstereolithography [59–62]. It is also considered to be among rapid prototyping technologies. Like the SLM process described above, it is based on the layer-by-layer generation of microstructures due to exposure of a liquid photosensitive monomer plastic to a focused low-power laser with a desired wavelength. The exposure leads to polymerisation and, therefore, to polymer microstructure walls. After the generation of one layer, the base platform used to build up the device is lowered by a specific value and flooded with the liquid monomer again. The next layer can be generated. The pattern of the laser focus is controlled by a computer, following the lines of a 3D CAD model. In Fig. 3, a microstructure device made by microstereolithography is shown. The polymer used here is stable up to a temperature of about 100°C and is more or less optically transparent.

Various techniques are possible to bond polymer materials. There are different opportunities for gluing and welding. While the gluing method heavily depends on the polymer used, welding like ultrasonic welding, laser welding, or solution welding can, in principle, be performed with all polymer materials. Details of the joining and sealing processes can be found in [6, 63–71].

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