

EXPLORING VACUUM CASTING TECHNIQUES FOR MICRON AND SUBMICRON FEATURES

M. Denoual^{*}, P. Mognol^{**}, B. Lepioufle^{*}
^{*}Biomis-SATIE ENS-Cachan antenne de Bretagne,
Campus Ker Lann, av Robert Schumann 35170 Bruz, France
^{**}IRCCyN Nantes, France
Reviewed, accepted August 25, 2004

Abstract

A study of resolution limits in standard rapid prototyping vacuum cast molding processes and adaptation of this technique to reach submicron accuracy is proposed. Micro-fabrication technologies are used to fabricate micron and submicron high aspect-ratio patterns on the original parts. The molding of the original parts is optimized to allow replication of submicron features. In carefully exploring materials and surface treatments, cast parts are successfully replicated with submicron and high aspect ratio micron structures. These encouraging results enable the use of such processes for micro- and nano-systems applications and open the door to development and production of low cost, high resolution biochips.

Introduction

Microsystems including Micro-ElectroMechanical Systems (MEMS), micro total analysis systems (μ TAS), micro arrays and biochips, are now well known as an emergent field in research and industry. The micro-fabrication technologies for microsystems result mainly from microelectronics technologies based on silicon processing [1] taking advantage of high volume production of microelectronics industry equipment.

However, other materials can be preferred in microsystems fabrication for specific material properties or fabrication cost. This is the case for polymers newly introduced in the microsystem field through biochemical applications. They are particularly fitted to biochemical microsystems that require easily fonctionnalisable materials. Transparency is often a critical property, particularly for biomicrosystems putting silicon out of the race. Moreover, bio-chemical microsystems that serve as vehicles for performing chemical and biochemical reactions, don't require on-board complex electronics except in some cases metallic electrodes, this eliminates silicon for those applications. And finally, the silicon micromachining techniques, such as deep-etching, are still low-volume, high-cost techniques. This need for polymer microsystems yields to the adaptation of plastic industry techniques such as hot-embossing [3-6] and injection molding [7]. The resulting techniques, micro-hot embossing and micro-injection molding, present the same characteristics as their macro equivalent. They require a large investment in time and money before the first part is realized and they become cost-effective only after several ten's of thousands parts are produced.

Thus, there is a niche prior to mass production for a low cost and low volume polymer microsystem part production technique suitable for rapid prototyping.

In this work, the vacuum casting process was adapted to the replication of structures with accuracy reaching micron and submicron features. It has been proved that this technique can be used for the micro-fabrication of microsystems. Replicated microstructures were obtained by classic micro-fabrication techniques. Successful results for the replication of microstructures have been achieved by minor improvements of the conventional vacuum

casting process. Using the improved vacuum casting techniques for biochip fabrication has shown that this technique is suitable for pilot lot production because of its low cost and fast design to product flow.

This paper aims to show the work and improvements performed in adapting the vacuum casting technique to microstructure duplication. The paper will show micro-fabrication technologies used to realize the original parts, the vacuum casting process, and the performances of vacuum casting technique for the replication of micron structures, high-aspect ratio and sub-micron structures.

Materials and Methods

The experimental step demonstrated in this paper was that : micro-structured original parts were subjected to the vacuum casting process and then duplicated parts were characterized in terms of dimensions and aspect-ratios.

1. Microstructures fabrication

In order to qualify the vacuum casting process at micron-scales, microstructures needed are obtained through microelectronics industry fabrication techniques and micro-fabrication technologies. The following are the techniques used.

a. Photolithography for micron scale structures (2 μ m to 300 μ m) with medium aspect-ratios (2:1 up to 1:6)

Standard microelectronic industry photolithography using a negative-tone photoresist (Microchem SU-8) was employed to realize the original parts. This process enables the patterning of micro-scale features by crosslinking of UV-exposed regions in the SU-8 photoresist [8]. This process, derived from microelectronics technologies, is presented in Figure 1. A standard silicon wafer is used as a substrate for the fabrication process (step 1.). SU-8 photoresist is spun on the wafer (step 2.). The photoresist viscosity and the spinning rotation speed determine the resulting thickness of the layer. In this work, two thicknesses are used: 20 μ m and 100 μ m. Once applied, the photoresist is baked to evaporate the solvents. The photoresist coating is then exposed to UV light through a chrome mask (step 3) in order to pattern the photoresist. Exposure time varies upon the photoresist thickness. Patterns designed on the chrome mask are geometrical shapes (rectangles and circles) with dimensions ranging from 2 μ m to 300 μ m. The UV-exposed photoresist is reticulated through a second bake. The non-exposed zones are dissolved in developer (step 4). Figure 2 shows the typical negative taper profile of an SU8 photoresist pattern on a 100 μ m high 20 μ m diameter tower, intentionally a broken tower is showed to facilitate the observation. This observation underlines the fabrication limitations of the negative photoresist process that enables high aspect ratio structures of up to 1:5 or 1:6.

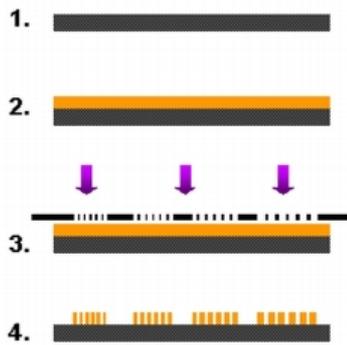


Figure 1 : Negative photoresist process.

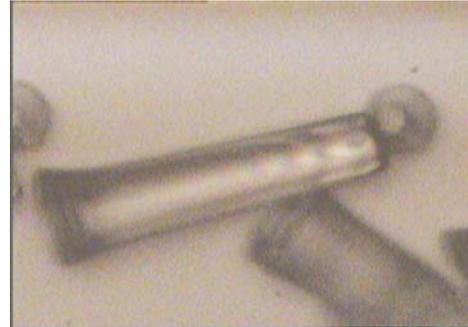


Figure 2 : Negative photoresist towers.

b. Oxide layer patterning for sub-micron features (down to 300nm)

The process used for the realization of the sub-micron features is a standard microelectronics photolithography and patterning of an oxide layer traditionally used as an insulation layer in semiconductor process. Lithography offers in-plane sub-micron precision on dimensional scales from micron to millimeter.

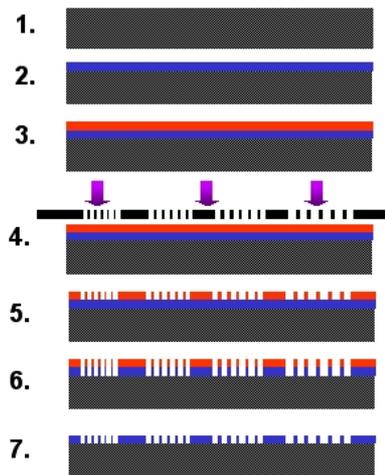


Figure 3 : oxide layer patterning process.

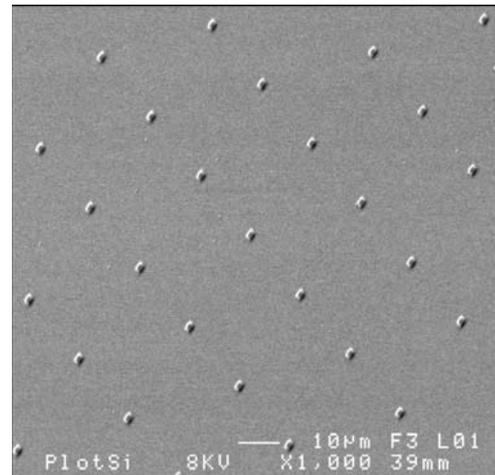


Figure 4 : oxide dots (1 μ m in diameter, 300nm thick).

The figure 3 describes the process used to obtain the high-aspect ratio structure used for duplication. A silicon wafer is first oxidized (step 2). Then a standard microelectronics photolithography process is performed. Positive-tone photoresist is spin-coated on the wafer (step 3) and exposed to UV light through a chromium mask (step 4). Exposed photoresist is then developed leaving the unexposed regions (step 5). The remaining photoresist acts as a mask during the etching of the oxide layer in acid bath or in plasma of a reactive ion etching (RIE) machine (step 6). The thickness of the oxide layer is 300nm. Photoresist is stripped (step 7).

The figure 4 shows matrix of oxide dots with 1 μ m in diameter and 300nm in height.

c. Silicon deep etching for high aspect-ratio structures (1:50)

Silicon deep-etching was introduced at the beginning of the 90's as a new technological process for micromachining of silicon. Specific tools were developed to perform this process called deep reactive ion etching (deep-RIE) because it is an adaptation of the RIE semiconductor industry process to the etching of deep structures. Since that time, deep-

etching has taken an increasing role in MEMS processing especially for actuators and sensors [9]. Deep-RIE has not yet entered high-volume manufacturing because of single wafer processing and wafer non-uniformity of the etching reaction.

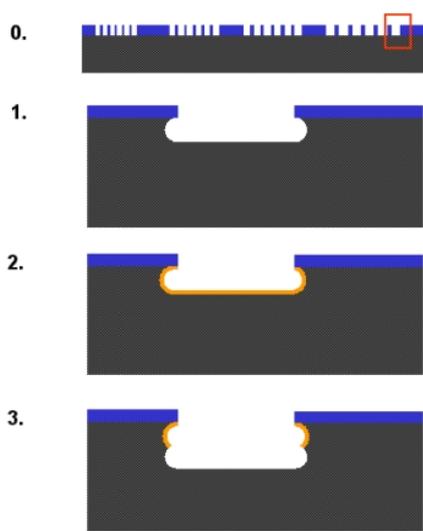


Figure 5 : silicon deep-etching Bosch process.

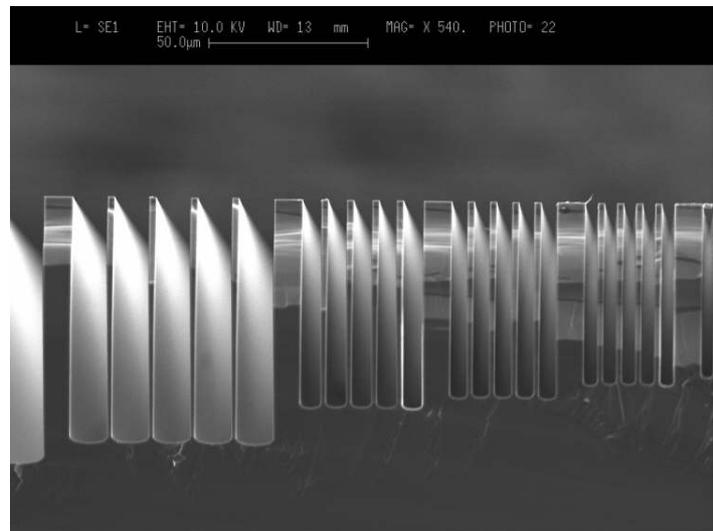


Figure 6 : SEM image of a polished microsection of a deeply etch silicon wafer.

Figure 5 describes the process used to obtain the high-aspect ratio structure used for duplication. An oxide layer acts as a mask during the deep-etching of the silicon. The oxide layer patterns are obtained through the process described in the previous paragraph. The deep-etching process developed by Bosch takes advantages of a side effect of glow discharges, the tendency to create polymeric species by chemical cross-linking. In fact, most plasma processes are a critical race between deposition of polymeric material from the plasma and the removal of material from the surface. In well-designed plasma chemistries, removal dominates. However, in the process developed for deep-etching, the deposition of polymer from the etchant is used to a great advantage. The etch proceeds in alternating steps of reactive-ion etching in an SF_6 plasma (step 3) and polymer deposition from a C_4F_8 plasma (step 2). During the etch step process, the polymer is rapidly removed from the bottom of the feature but lingers on the sidewall, protecting it from the SF_6 etchant. The etching depth is controlled thanks to process duration time, i.e. the number of etching cycles (repetition of step 2 and 3).

Figure 6 shows an example of deeply etched silicon wafer. We can observe that the depth of the trenches depends on the opening of the trench. High aspect ratio (up to 1:50) can be performed with this technique.

The deep-etching technique tends to form structure with a slightly negative taper profile with angle between 85° and 89.5° which increase the mechanical constraints during the vacuum casting process for the removal of the duplicated parts. Figures 7 and 8 show close-up views of the trenches that illustrate the dimensions of the walls and the scalloping effect due to the process steps.

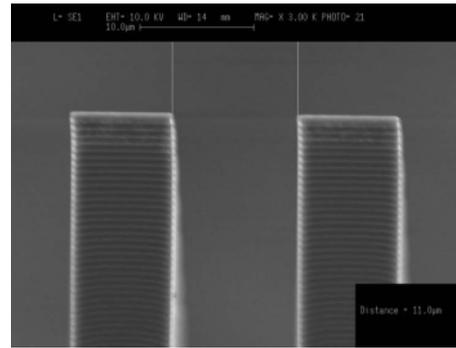
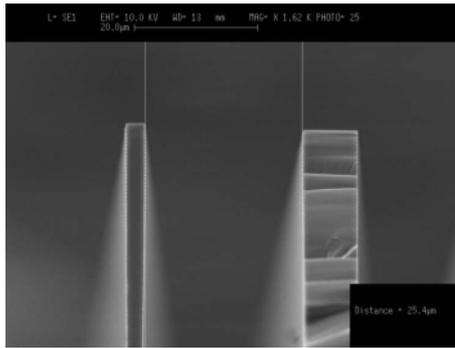


Figure 7 : SEM image of vertical thin walls with here a $3\mu\text{m}$ wide and $84\mu\text{m}$ high wall vertical pillars. (aspect ratio 1:28). Figure 8 : high magnification SEM image of vertical pillars.

2. Silicon processing limits

Microelectronics industry techniques facilitate the realization of micro-structured devices, but they are limited by three factors.

- The choice of material is limited to silicon-based materials. This is a real limitation when considering biological or chemical applications because of transparency and biocompatibility requirements. In addition, silicon is more expensive than polymers when bulky parts are concerned.
- The techniques allowing high aspect-ratios and volume structures patterning such as deep RIE techniques are still very expensive.
- Those techniques are not available for mass production because of the expensive equipment involved treating only a single wafer at a time and the processing time is long (with etching speed $\sim 1\mu\text{m}/\text{min}$).

3. Duplication process

The vacuum casting process consists in the realization of a silicon female mould with an original part followed by resin casting under vacuum to obtain the duplicated parts.

In the first experiments, the standard vacuum casting process was applied. The direct application of this process is illustrated in Figure 9. A Sprue bushing channel and vent are joined to the original part to be duplicated (step 1.). A female mould is made of silicone around the original part (step 2.). VTV 750W silicone form MCP is first mixed 2 minutes in a ratio of 10:1 with the hardener solution (CAT 750) and then degassed 15 minutes prior to pouring over the original part. Curing time is 6 h at 70°C . After the bake, the silicone female mould is opened and the original part withdrawn (step 3.). Female mould is then ready for casting. MCP 6091 resin was used for casting under vacuum in the female mould. First MCP 6091 part B is degassed under vacuum for 10 minutes. Then MCP 6091 part A and MCP 6091 part B are mixed in a ratio of 1:1.8 (A:B) during 1 minute. The mixed solution is then cast in the female silicone mould (step 4.). After 2 h bake at 70°C , the resin is hardened and the resin duplicated part is withdrawn from the mould (step 5.).

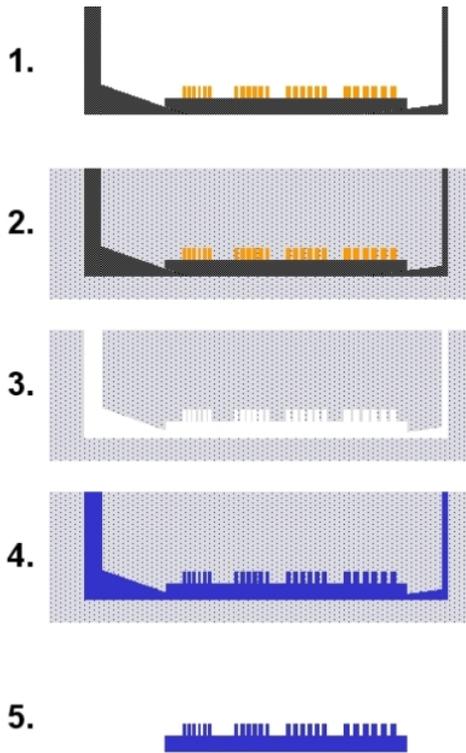


Figure 9 : Vacuum casting procedure

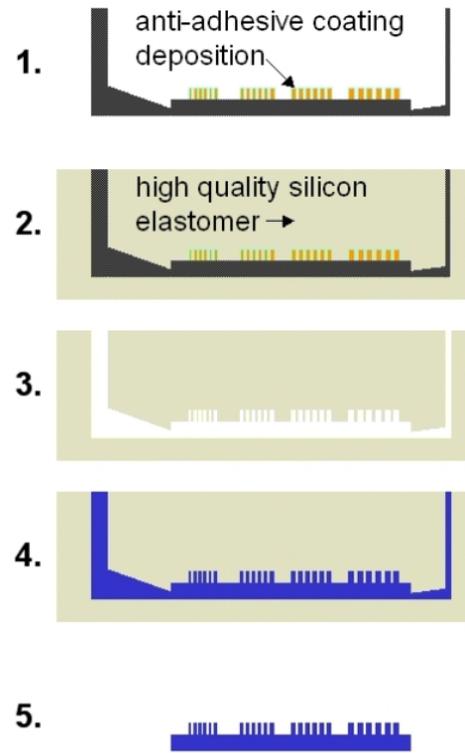


Figure 10 : improved process

It appeared that the direct application of the vacuum casting process was not good enough for microsystem applications as illustrated below. The poor surface roughness was due to sweating of the silicone used for the female mould fabrication. Hence, the first improvement was to use a high quality silicone to realize the female mould. PDMS (PolyDiMethylSiloxane, Sylgard RTV184 from Down Corning) was chosen. This silicone is often used in micro-fabrication technologies for micro-molding processes [10]. The PDMS curing process is similar to the VTV 750W. The PDMS prepolymer is mixed in a ratio 10 : 1 with the hardener solution and then degassed 15 minutes in vacuum prior to pouring. Curing time is quicker with 1 h at 70°C.

Another improvement to prevent rupture of the silicon female mould during the withdrawal of the original part, in case of high aspect ratio structure or slightly negative taper profiles, is the deposition of an anti-adhesive coating on the surface of the original part. Indeed, a “teflon”-like layer can be deposited in CHF_3 plasma by a standard reactive ion etching (RIE) machine. This treatment is particularly important for the reproduction of high aspect-ratio structures. The improved process is presented in Figure 10.

Results and performances

1. Micron features, medium aspect ratio

Figures 11.b,d and f illustrate the results obtained when the classic macro vacuum casting process is applied to micro-scale structures. The replication is not correct. Dimensions are not reproduced and shapes are altered. In addition the resin has a poor surface quality. Surface quality can be compared to the original part surface quality in Figure 11.a,c and e. Original parts have a flat surface whereas duplicated parts have variegated surfaces, and straight lines on the original part become wavy on the duplicated part. Those preliminary results were not

good enough to consider the use of vacuum casting for microsystem applications.

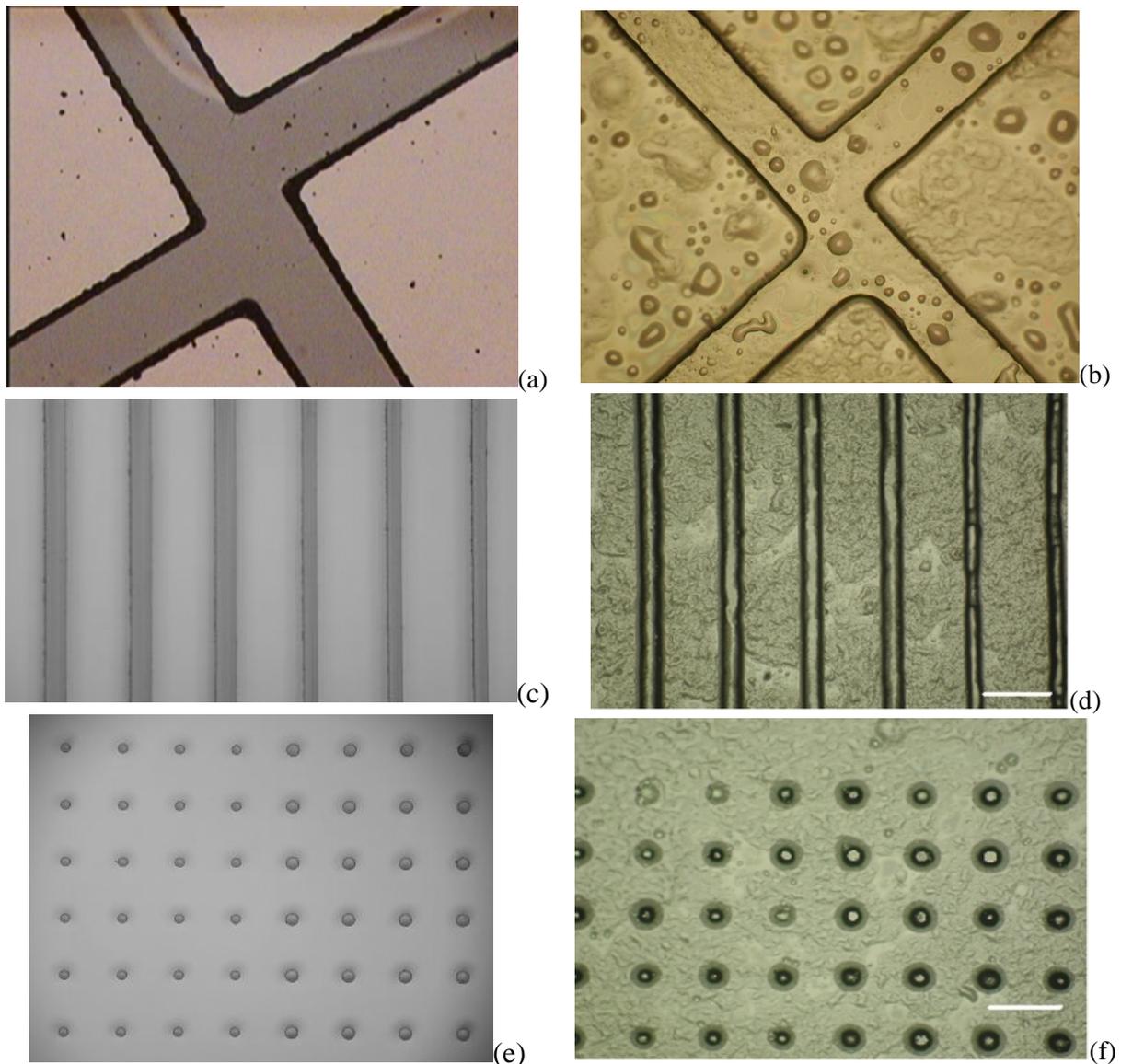


Figure 11 : The pictures on the right correspond to the original micron scale structures. On the left, the pictures show the duplication results. SU8 cross with $300\mu\text{m}$ wide and $100\mu\text{m}$ high walls (a), resin replication of the cross (b), SU8 original towers (c), duplicated towers (d, scale bar is $100\mu\text{m}$). The patterns are not accurately duplicated here and the surface quality is poor.

The improvements presented in the previous paragraph were applied (anti adhesive coating and high quality elastomer for the mould) and Figures 12.a,b,c and d show the results obtained. The surface quality is comparable to the surface of original part. Moreover, the structures are accurately duplicated.

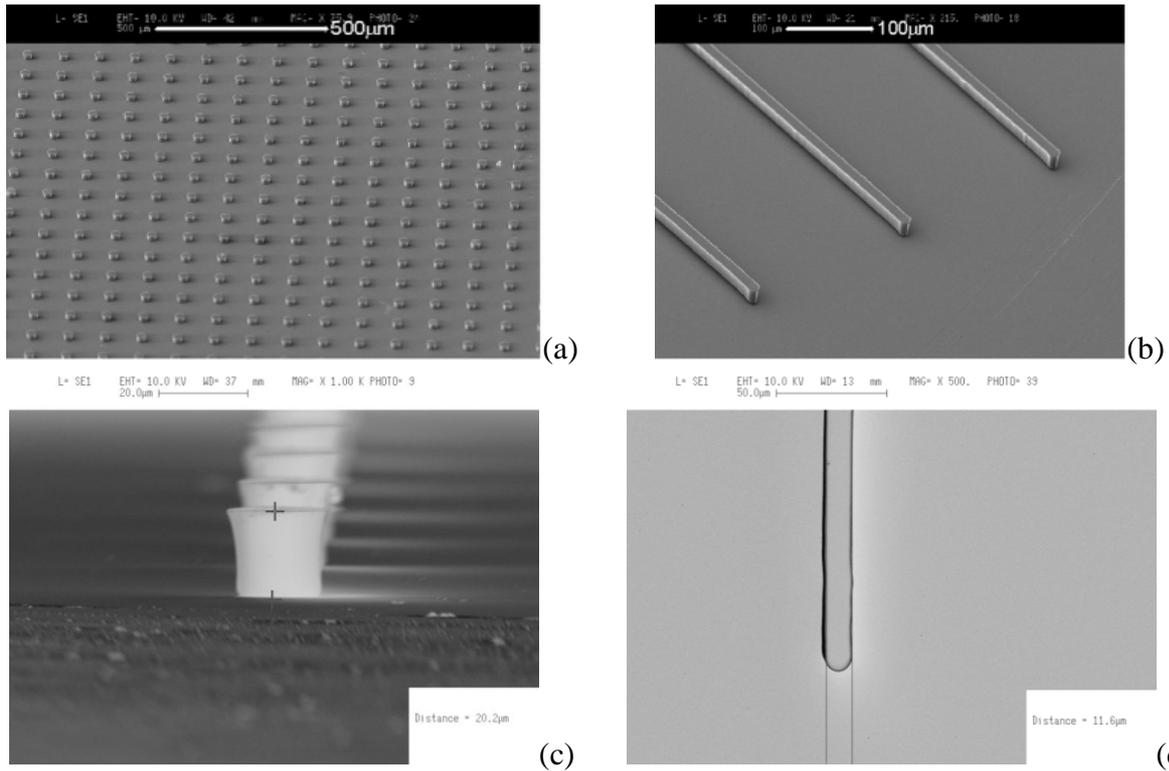


Figure 12 : SEM image of duplicated structures. Field of towers (a), set of walls (b), negative taper profile towers (c) with 10μm diameter, 20μm high, and a 11.6μm wide, 20μm high duplicated wall (d).

Using this technique, features ranging from 10 to 300 μm were successfully replicated. Aspect ratio of the realized structures varied up to 1:6. The smallest structures of the original parts were successfully duplicated and the duplicated dimensions were limited only by the dimensions of the original parts. Duplication error is estimated to be about 1%.

The slightly negative taper profile were also successfully replicated thanks to the elasticity of the mould. Such profiles are not allowed with hot embossing or injection moulding since they lead to structural breakdown of the mould or prevent part withdrawal.

2. Submicron features

To qualify the replication of submicronic features, the improved vacuum casting process has been performed for the duplication of oxide layers patterned on silicon. Figure 13 presents original oxide and silicon parts against their resin vacuum casting replication. The smallest pattern dimensions are 0.8μm and the oxide layer thickness is 300nm.

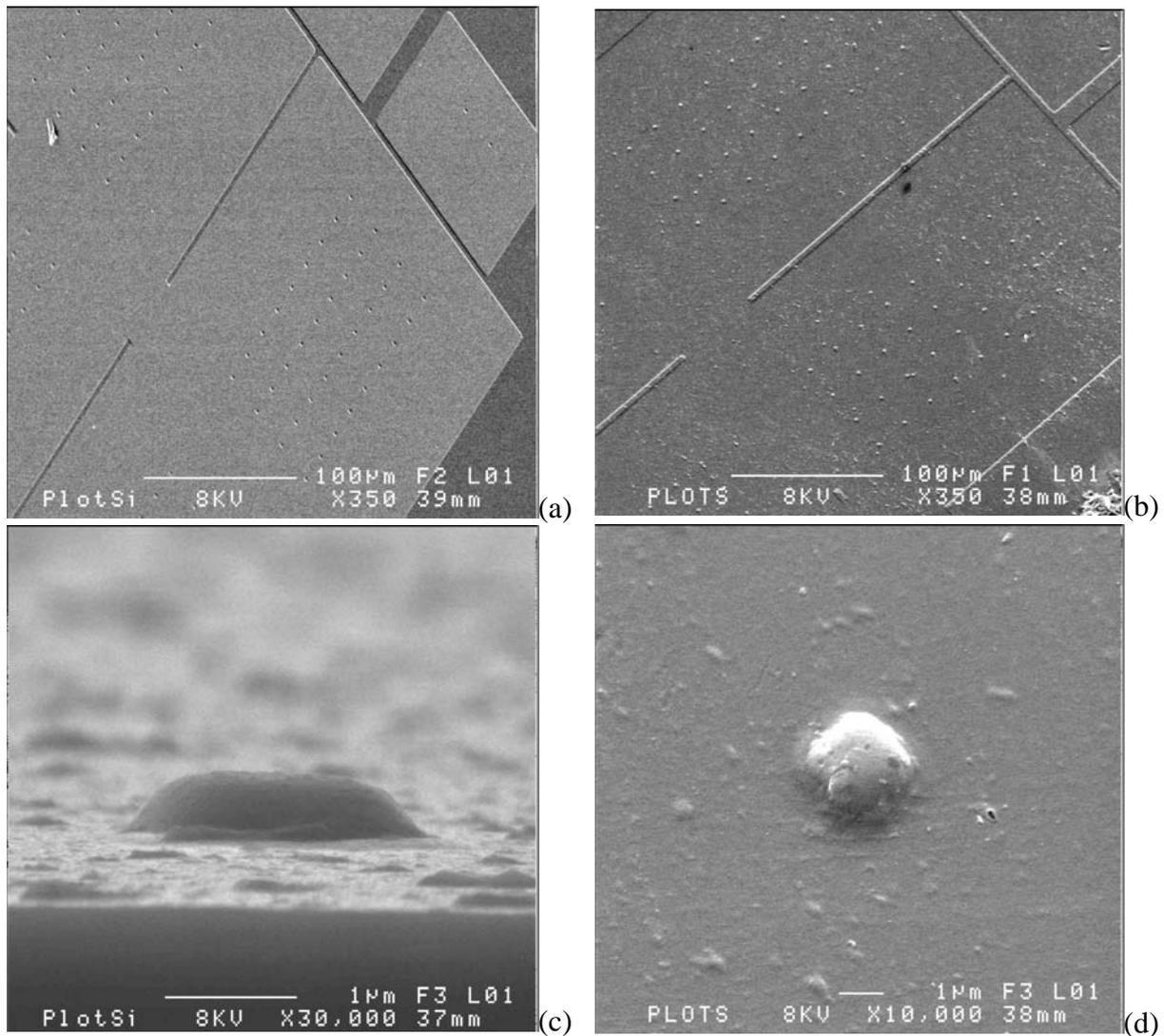


Figure 13 : SEM images of original oxide and silicon structures and their resin duplication. Geometrical shapes patterned in oxide (a), resin replicated shape (b), small oxide dot (c) and its replication in resin (d).

The results demonstrate that submicron structures can be replicated using the vacuum casting improved process. Features down to 800nm wide and 300nm high have been successfully replicated using the vacuum casting improved process. The limitation here is due to the process environment. The vacuum casting process is operated in ambient environment without any particular dust class control. Therefore some dust is visible on the surface of the duplicated structures. For nano-replication using vacuum casting technique, clean-room environment is required.

3. High aspect ratio

In order to characterize the performances of the vacuum casting process, deep-etching of silicon was used to realize high-aspect ratio (up to 1:50) silicon patterns. The structures utilized for replication were walls, trenches, cylinder and cubes, with dimensions down to 2µm wide and walls, up to 100µm in height. The results are presented in the figure 14.

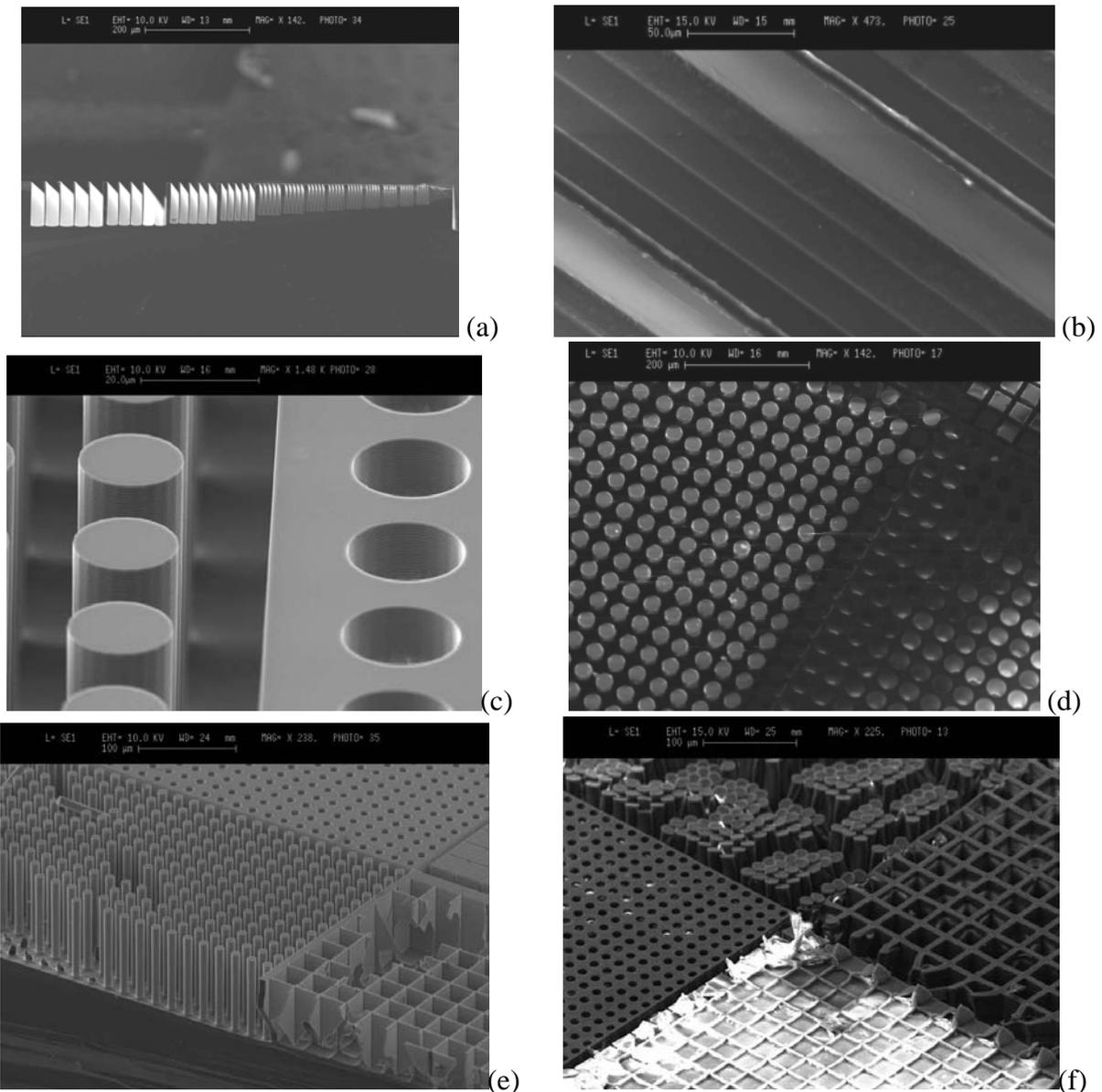


Figure 14 : SEM images of high-aspect ratio silicon structures (a,c and e) and their resin replication through vacuum casting (b,d and f). A $6\mu\text{m}$ wide and $100\mu\text{m}$ high wall (b), $20\mu\text{m}$ diameter and $100\mu\text{m}$ towers (d).

Visualization of polymer structures with a SEM requires the coating of the sample surface with a thin conductive layer (chromium-gold). However a side effect of the high aspect-ratio structures is creation of shadows during the deposition of this layer, which causes brightness variations on the images of resin structures compared to the images of silicon structures.

The maximum aspect ratio obtained in a replicated structure is 1:16. One could probably improve this aspect ratio with positive profile high aspect ratio structures.

Structures with an aspect ratio of 1:20 have been replicated as illustrated in the figure 15. Indeed the replicated cylinders are $5\mu\text{m}$ in diameter and $100\mu\text{m}$ high. But the material stiffness seems insufficient to maintain the shape. This implies future work on the material choice and processing to improve aspect-ratio.

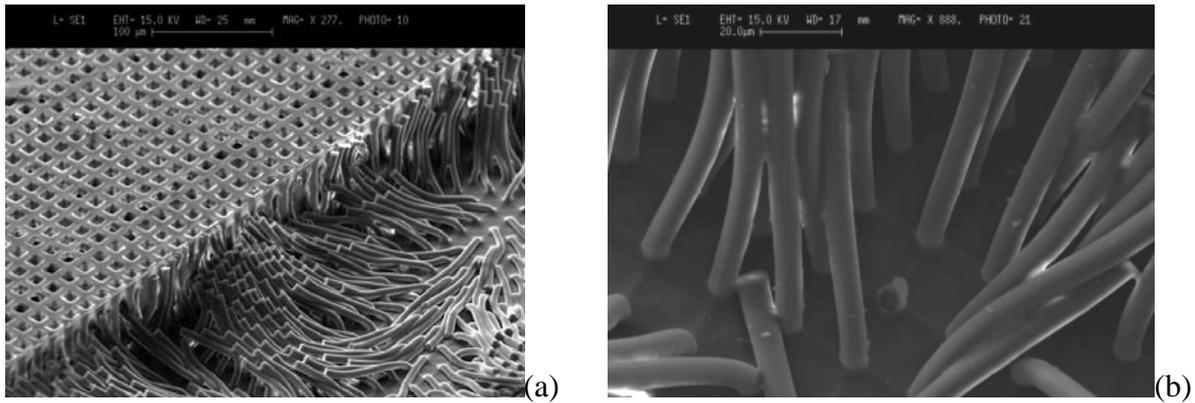


Figure 15 : SEM image of resin duplicated patterns. 5 μ m diameter and 100 μ m high cylinders. Low magnification (a), high magnification close-up view (b).

Conclusion

The adaptation to the replication of microstructures of a conventional rapid prototyping technique was investigated. According to the experimental results, a conventional vacuum casting process with minor improvements can be applied to the fabrication of microstructures. High quality materials are essential to overcome sweating and adhesion problems found at the micron scale. The duplications of classical test structures showed sub-micron (down to 300nm) and high aspect-ratio (up to 1:16) performances. The improved vacuum casting process allows accurate replication of microstructures made with standard micro-technologies.

A cost study has been performed for a specific bio-chemical microsystem application. The results of the study, illustrated in the figure 16, stress the cost-effective domain of this technique : from a few parts to a few thousand parts. Therefore, improved vacuum casting meets the need for small series of micro-structured systems made of polymers. Because of its high speed to first part and low-cost, the adaptation of the vacuum casting process to micro-fabrication will lead to a promising technology for MEMS application. This is indeed a suitable technique for rapid prototyping of polymer microsystems dedicated to biology or chemistry when compared to hot embossing or micro-injection molding techniques that require significant more time and money.

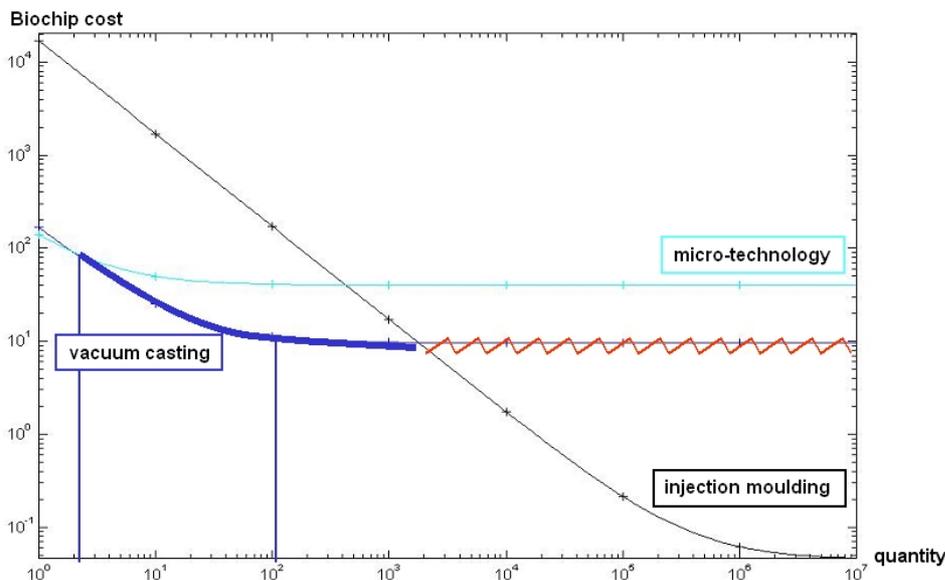


Figure 16 : part cost versus production volume compared for injection molding, micro-fabrication technology and vacuum casting process.

REFERENCES

- [1] J.W. Gardner, V.K. Varadan, O.O. Awadelkarim, "Microsensors, MEMS and Smart Devices", John Wiley & Sons, 2001.
- [2] V. K. Varadan, X. Jiang, V. V. Varadan, "Microstereolithography and other Fabrication Techniques for 3D MEMS" John Wiley & Sons Ltd, 2001.
- [3] G-B. Lee, S-H. Chen, G-R. Huang, W-C. Sung, Y-H. Lin, "Microfabricated plastic chips by hot embossing methods and their applications for DNA separation and detection", *Sensors and Actuators B*, n° 75, pp 142-148, 2001.
- [4] L. Lin, T. Cheng, C.J. Chiu, "Comparative study of hot embossed micro structures fabricated by laboratory and commercial environments", *Microsystem Technologies*, n° 4, pp 113-116, 1998.
- [5] X-J Shen, L-W Pan, L. Lin, "Microplastic embossing process experimental and theoretical characterizations", *Sensors and Actuators A*, n° 97-98, pp 428-433, 2002.
- [6] H. Becker, U. Heim, "Hot embossing as a method for the fabrication of high-aspect ratio structures", *Sensors and Actuators A*, n° 83, pp 130-135, 2000.
- [7] Y-C. Su, J. Shah, L. Lin, "Implementation and analysis of polymeric microstructure replication by micro-injection molding", *J. Micromech. Microeng.*, n° 14, pp 415-422, 2004.
- [8] E. Conradie and D. Moore, "SU-8 thick photoresist processing as functional material for MEMS applications", *J. Micromech. Microeng.*, n° 12, pp 368-374, 2002.
- [9] S. D. Senturia, "Microsystem design", Kluwer Academic Publisher, 2000, pp 67-71.
- [10] D. Duffy, C. Mac Donald, O. J.A. Schueller, G. M. Whitesides, "Rapid Prototyping of Microfluidic Systems in PDMS", *Anal. Chem.* 1998, n° 70, pp4974-4984.