

Moulage par injection avec un moule en silicium à haute résolution

High resolution injection molding using silicon mould

K. METWALLY^{a,b}, R. SALUT^a, G. MICHEL^b, B. GAUTHIER-MANUEL^a, T. BARRIERE^b and C. KHAN-MALEK^a

a. FEMTO-ST Institute - UMR CNRS 6174, Dpt. Micro Nano Science & Systems, 25044 Besançon, France

b. FEMTO-ST Institute - UMR CNRS 6174, Dpt. Applied Mechanics, 25044 Besançon cedex, France

Résumé :

Le moulage par injection est un procédé de fabrication mature utilisé pour produire en masse à moindre coût des pièces pour de nombreuses applications. L'objectif de ce travail est l'utilisation du moulage par injection pour produire des pièces en polymères avec des motifs de taille micrométrique à submicrométrique. Un moule en silicium avec des motifs de largeur allant de 5 µm jusqu'à 300 nm a été fabriqué en utilisant la lithographie par faisceau d'électrons et la gravure sèche. Des répliques ont été injectées en polypropylène. Des expériences en fonction de la température d'injection et du volume injecté ont été réalisées pour optimiser le remplissage des microcavités du moule. Le moule et les répliques ont été caractérisés par profilométrie mécanique et microscopie électronique à balayage.

Mots clefs: moulage par injection, haute résolution

Abstract :

Injection molding is a mature manufacturing process used to mass produce cost-effectively parts for a variety of applications. This work focus on using injection molding to produce polymeric parts with micrometric to sub-micrometric features. A dry-etched silicon mould insert with ridges of width from 5 µm to 300 nm has been manufactured using electron beam lithography and dry etching. Replicas have been made in polypropylene. Experiments as a function of injection temperature and injected volume have been conducted to optimize the filling of microcavities of the mould insert. Both mould and replicas have been characterized using mechanical profilometry and scanning electron microscopy.

Keywords : submicrometric, injection molding process, thermoplastic

1 Introduction

Polymer replication in sub-micrometric scale gets attention of manufacturers in numerous industries according to the new applications demands, particularly in bio-analysis, photonic devices, anti-reflection polymer displays, as well as solar cells to increase the efficiency. Replication in such scale can be carried out by various processes such as hot and UV embossing [1], nano-imprint lithography [2], soft lithography [3], and injection molding [4-6].

The combination of the injection molding process and adoption of new tooling had shown strong potential to produce components with nano or micro features. Tooling for injection in micrometric scale down to nanometric scale differs from that at the macrometric scale as the influence of a number of effects like interfacial effect, tool surface effect, machine response time and high shear stress caused by thin walls with high filling pressures. Generally, metallic tool inserts are manufactured using mechanical micromachining, micro electro-discharge machining (µEDM) or a combination of lithography and electroplating. It is also possible to use silicon wafers [7] or polymeric micro tool insert [8] or a combination of both [9].

Thin walled plastic parts are defined as parts that have a nominal wall thickness less than 1 mm and a surface area of at least 50 cm² with a flow length/thickness ratio above 100 or 150 [10]. This appears in micro-molding where the challenge lies in reducing the size, weight and cost of components, as well as increasing their functionalities, trying to avoid that the frozen layer thickness could be comparable to part thickness during filling [11].

In this work, a micro-injection molding process will be described using a submicronic-structured silicon wafer as a replaceable mould insert for prototyping and manufacturing small series of polymer-based microfluidic devices. A process based on well-established clean room technologies had been used, which is generally used for micro-electronics and MEMS applications enabling the collective manufacture of micro/nano-structures in a silicon wafer. The mould insert has been then replicated in polypropylene (PP) using a commercial injection machine.

Experiments as a function of injection temperature, injection rate and injected volume have been conducted to optimize the filling of microcavities of the stamp. Both mould and replicas have been characterized using optical microscopy, profilometry and scanning electron microscopy (SEM).

2 Experimental

2.1 Materials and methods

Molding experiments have been performed using an Arburg[®] Allrounder 220S/150-35 (figure 1a). The molding press is equipped with a plastification screw of 15 mm diameter and allows injection rate up to 32 cm³/s and a clamping force up to 150 kN. The thermoplastic polymer has been injected in the mould through an injection sprue diameter of 3 mm. A mould adapter has been designed to hold the 4" wafer and allow venting (figure 1b). Vacuum venting was applied to get out the trapped air from the mould cavities and provide evacuation of gases produced by low molecular weight polymers and additives, which subsequently leads to increase the fidelity of replica especially at the edges [12].

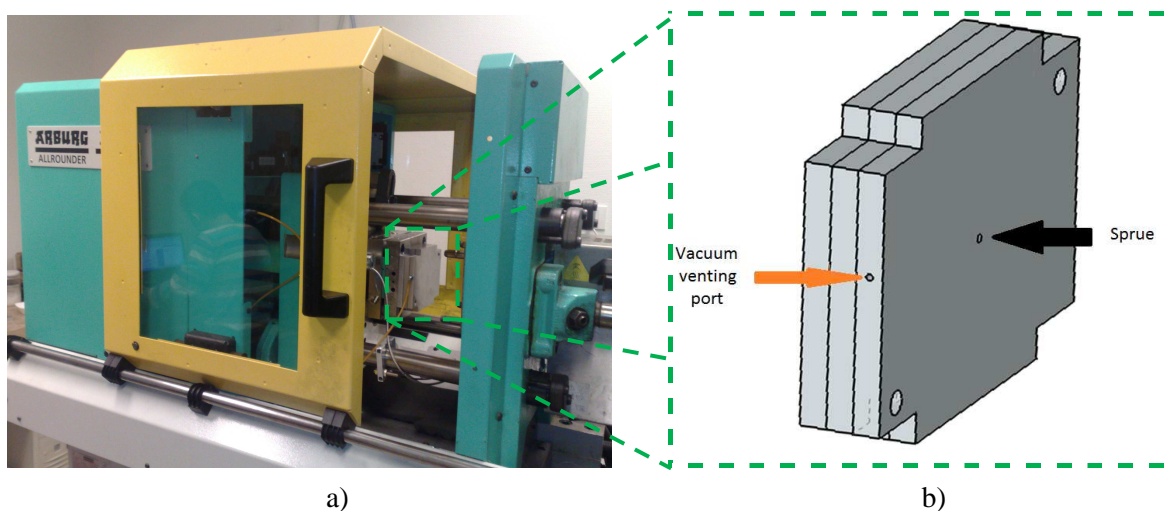


FIG. 1 – a) Optical image of experiment setup; b) Isometric view of mould holder with vacuum venting.

A polypropylene (PP-RA12MN40) from Sabic[®], a random copolymer modified by ethylene, has been selected for the injection molding experiments. This PP grade offers good fluidity which reflected on its melt flow index (MFI) equal to 40 g/10 min at [230 °C and 2.16 kg] in agreement with the ISO 1133 norm. It is also amenable to be replicated easily. It is characterized by high stiffness, good transparency and high gloss. It is typically used in houseware applications and thin walled packaging.

2.2 Mould manufacturing

Structured ridges of nominal lateral dimensions of 5 μm down to 300 nm with a fixed pitch of 5 μm have been manufactured by electron beam lithography (Raith E-line pattern generator) in an electron sensitive polymer, ZEP520A (Zeonrex Electronic Chemicals) followed by dry etching in a fluorine plasma.

2.3 Replication process

The dry-etched silicon mould insert has been then mounted in the holder of injection molding equipment. The tests have been carried out starting with the standard conditions recommended by the PP manufacturer. The parameters varied to optimize injection molding of the parts such as injection temperature, injection rate and injected volume. The injected volume has been first determined to fill the volume of the mould cavity. Several trials have been carried out to optimize the injection parameters. The injection temperature varied in range from 165 °C to 230 °C stepwise in steps of 10 °C to optimize the filling of the features of the mould insert cavity before the solidification of the melted polymer occurs. In order to overcome flow resistance, high injection rate, since the resulting high shear rate is good for viscous heating and decreasing polymer viscosity. The injection rate has been increased progressively up to its final value set close to the maximum value. An appropriate holding time at constant pressure has been selected to ensure proper packing of the polymer in the submicronic cavities of the mould insert. Good filling has been obtained for PP with a mould at room temperature. After solidification of the polymer part inside the mould upon cooling, the replicas have been manually separated from the mould using compressed air.

3 Results and discussion

3.1 Mould cavity observation

A SEM image (figure 2a) displays a 5 µm-period array of 500 nm wide, 50 µm long and 386 nm deep features obtained by EBL patterning and transferred in Si wafer by dry etching. Exposure dose varies between 12-22 µC/cm² for features width of 300 nm till 5 µm. In submicronic structure arrays, optimum dose modulation depends on pattern size and period.

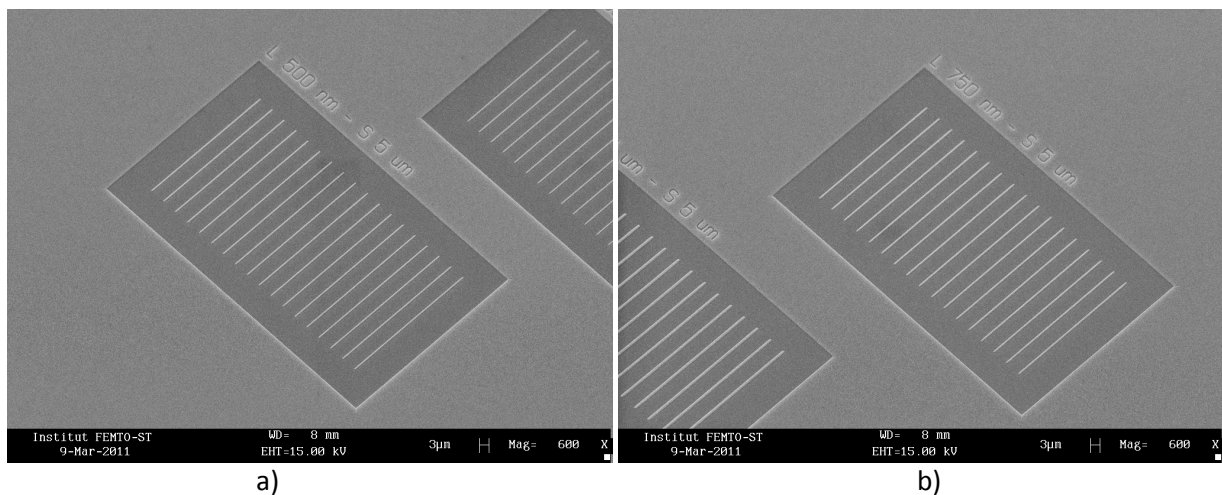


FIG. 2 – SEM images of 400 nm till 1 µm wide, 386 nm height and 5 µm pitch etched in Si: a) 500 nm wide features @ center, 400 nm features @ right. b): 750 nm wide features @ center, 1 µm features @ left.

3.2 Replication results

The projections of the tooling have produced depressions or features in the molded part. These traces give the depth of the molded part as a function of scanned distance. When feature dimensions have been measured, the replication of the tooling features was quantified using the depth ratio and feature definition. The depth ratio, DR , is defined as:

$$DR = \frac{d_p}{d_t} \quad (1)$$

where d_p is the depth of the depression in the molded part and d_t is the tooling depth (figure 3a, i.e., 386 nm).

By calculating the depth ratio (1), it was found to be 75.13%. This difference between replicated feature depth (figure 3b) and tool depth can be explained by the shrinkage of the polymer. Usually, semi-crystalline materials shrink more than amorphous materials due to the repeated units along the molecular chains being of such a form that they can pack very closely together in an ordered manner.

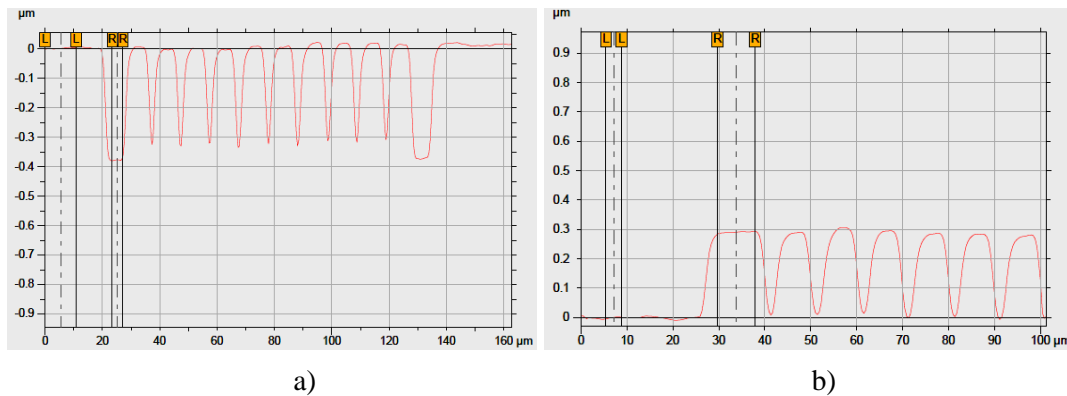


FIG. 3 – Measured depth at 5µm wide features, a) Tool depth of 386 nm, b) PP replica transferred depth of 290 nm.

In macro scale injection molding the processing shrinkage of PP is 1.3-2%, and 0.8-1.8%, in direction of flow and crosswise to direction of flow, respectively [13]. However, it is not the same in microscale, as it has been declared by Barry group that the depth ratio is extremely affected by shrinkage [14]. Depth ratio can be improved by using gas assist injection molding process to reach almost higher depth ratio.

After optimizing the injection molding parameters, results have been obtained for 500 nm wide features array (figure 4a) and 750 nm wide features (figure 4b) at injection volume of 7 cm³, while the injection temperature was 230 °C for the four heating zones and the mould temperature was kept at ambient temperature. Injection rate was set to 20 cm³/s and injection pressure was 100 MPa for a final part thickness of 0.6 mm.

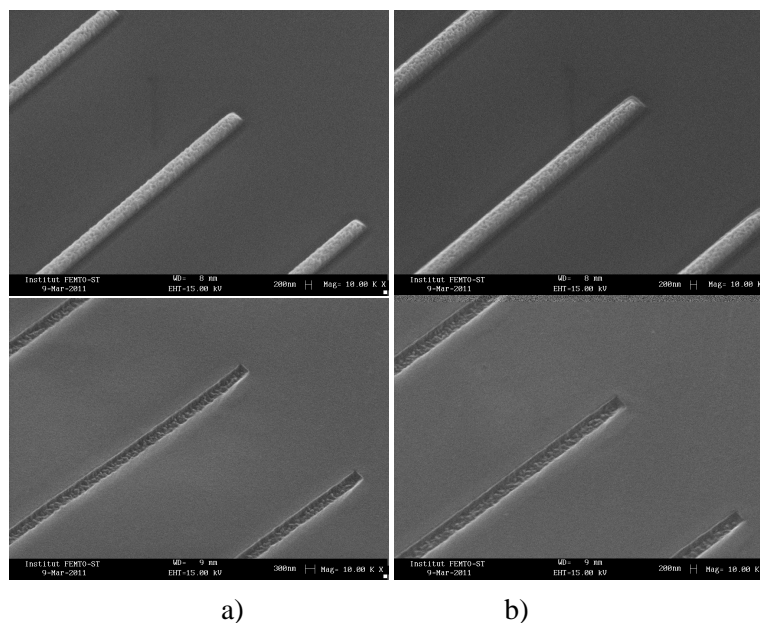


FIG. 4 – SEM images for Si mould features of 386 nm height & 5 µm pitch and replica with a transferred depth of 290 nm: a) Top 500 nm wide, bottom its replica, b) Top 750 nm wide, bottom its replica.

Theoretically, increasing melt temperature, injection rate, and resulting pressure should improve feature definition and depth ratio. At higher melt temperatures, material can flow longer to complete the part filling, while higher mold temperatures will slow the cooling and allow more material to flow to the end of the fill. The higher injection rate results in lower melt viscosity due to higher rates of shear and elongation, and to shear heating. Lower viscosity melt would be expected to improve the depth ratio and feature definition because the material can more easily flow into the features. Rapid filling also reduces cooling of the molten polymer during filling, and thus, should improve feature replication. For this mold design, packing pressure acts like a compression force and higher pressures should force the melt closer to the cavity walls. In our previous work, the mould temperature didn't make a great difference when working with PP as injected material.

Conclusion and perspective

PP replicas with 500 nm & 750 nm wide features were replicated in PP from a silicon mould insert manufactured by electron beam lithography patterning and dry-etching using a conventional micro-injection molding equipment.

The manufacture of silicon mould insert with finer feature size (less than 500 nm) is presently being investigated and replication with higher resolution will be explored.

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