Tooling for Micro- and Nano-Imprinting and its Consequences for Manufacturing

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ABSTRACT

Imprinting processes show remarkable potential for manufacturing devices with micro- to nanometer-scale features and planar 2-D geometry for a variety of applications. This work specifically considers tooling for thermoplastic imprinting processes such as hot micro-embossing (HME), and applications where the imprint process produces the actual functional features, such as microfluidic channels or optical elements (rather than, for example, imprinting an etch barrier). Tooling is critical for manufacturing by HME, since the tool is in intimate contact with the part and defines its geometry. Tool-to-tool variation also has a direct impact on the overall quality of imprinted parts. Tool life contributes to overall cost, as well as reducing quality if a large number of tools must be used and tool-to-tool variation is large. Various materials and techniques for producing tools are reviewed, and the unique characteristics of each process are considered with regard to their consequences for mass-manufacturing of polymer devices by HME.

INTRODUCTION

Imprinting processes have attracted increasing attention for their potential as manufacturing techniques for micro- to nanometer-scale features in polymers. Such processes include hot micro-embossing (HME), nano-imprint lithography (NIL), UV-embossing, UV-NIL, and others. Broadly, these processes involve use of a tool to replicate micro- to nanometer-scale features in a substrate. Substrates include thermoplastics, pre-cured thermoset and UV-curable polymers, and even metals.

Replication fidelity is often high, so the geometry, surface texture, and other characteristics of the finished part are largely determined by the tool. Tooling is a critical factor for the overall success of imprinting processes, not only in terms of the quality of individual parts, but also for realizing the potential of imprinting as a mass-manufacturing technique.

This work will specifically consider the impact of tooling on manufacturing concerns for thermoplastic imprinting and applications in which the imprint process produces the actual functional features of the product. In thermoplastic imprinting or hot micro-embossing (HME), features are produced in a thermoplastic polymer workpiece by deformation at elevated temperature (Fig. 1 and Fig. 2). In the typical HME process cycle, the workpiece is first heated above the glass transition temperature. Then, the structured tool is brought in contact with the workpiece and a forming pressure is applied. Next, the forming pressure is maintained at elevated temperature for a pre-determined time to allow for adequate replication of the tool features. The pressure is then further maintained as the workpiece is cooled below the glass transition temperature. Finally, the tool and workpiece are separated.

The HME process has been used to produce microfluidic devices such as a polycarbonate lab-chip for PCR in Hashimoto *et al* [1], optical interconnects and couplers in Frese *et al* [2], and a microfluidic lab-chip with integrated dye laser, lenses, and waveguides for optical sensing in Hansen *et al* [3].

These papers exemplify the majority of published work on the HME process, in that they are primarily concerned with demonstrating the feasibility of producing functional prototype devices. Relatively less work has been done on HME as a manufacturing process [4]. The manufacturing perspective introduces concerns for production rate, consistency and quality of the finished parts, and durability and reliability of the production tools and machinery. The production rate, quality, and cost of tooling have direct and important consequences for the overall production rate, quality, flexibility, and cost of the HME process.

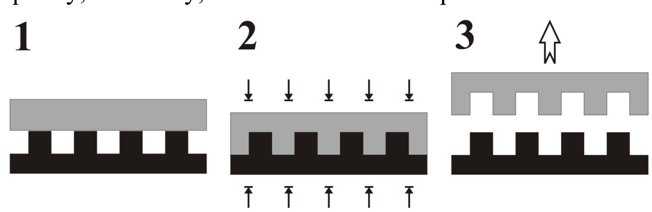

Fig. 1 Process schematic for thermoplastic imprinting, or hot micro-embossing

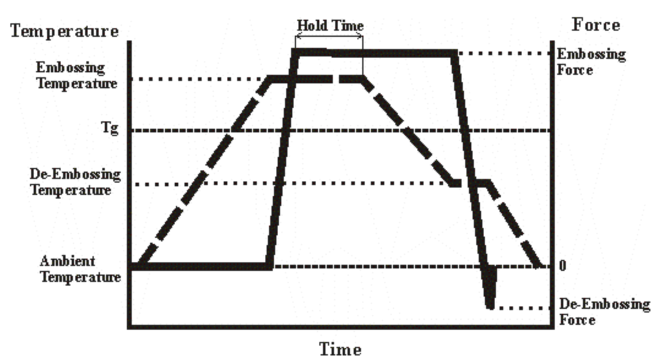

Fig. 2 Process parameters for typical thermoplastic imprinting, or hot micro-embossing. Tg=Glass transition temperature

KEY CHARACTERISTICS OF IMPRINT TOOLS

Before discussing the various materials and techniques for producing tools for HME, it is worthwhile to consider which characteristics of tools and production techniques are of interest in the context of manufacturing. We consider characteristics that affect rate, quality, flexibility, and cost in turn.

A. RATE

Production rate in HME is determined by the time to fixture and remove the part, the time required to heat and cool the part, and the time required for the workpiece material to conform to the tool features. These parameters are set by the capability of the equipment used and by the mechanics of deformation. Tooling has little or no direct impact on these factors, though Ye *et al* [5] report that when embossing polycarbonate with a silicon wafer, the part must be cooled slowly or thermal contraction mismatch will fracture the brittle tool. When heating and cooling are rate-limiting, forming at lower temperatures improves production rate. A stronger, more durable tool would be better suited to lower-temperature forming, since the pressures required are higher.

B. QUALITY

The quality of the resulting part may be the area where tooling has the greatest impact. Dimensional inaccuracy in the tool will be transferred to the part. Even worse, variation between tools will exacerbate the natural variation of the imprint process. Accuracy and consistency are thus critical factors in the evaluation of a tool production technique. The latter of these is especially important, since repeatable error can often be compensated.

Geometric characteristics of the tool such as sidewall draft angle are also important for quality. If a protruding feature on the tool is wider at its tip than at its base, demolding becomes extremely difficult. Thermal contraction mismatch between the tool and part during cooling can lead to substantial forces on feature sidewalls, potentially distorting features and making demolding difficult even when no undercut is present [6,7]. Fig. 3 illustrates the results of both thermal contraction distortion and undercut tool features. Localized defects in the tool will also adversely impact quality, since these defects will very likely be transferred to the parts.

Chemical interaction between the tool and part can also impact quality. Adhesion or friction caused by chemical bonds, van der Waals interactions, or mechanical interactions between asperities will increase demolding forces and may lead to damage of the part or tool. Hirai *et al* [8] found that sidewall friction, stress concentration at feature corners, and thermal contraction cause damage to raised features during demolding. Higher aspect ratio features are more susceptible to damage. Various anti-adhesive surface treatments have been applied to mitigate problems associated with demolding. This subject is discussed in section E.

The physical properties of the tool material also influence the quality of imprinted parts. The stiffness of the tool material will determine the amount of elastic deformation under forming pressure. This elastic deformation will affect the part dimensions. The brittleness or toughness and overall strength of the tool material will help determine tool life. As the tool degrades over its life, the quality of imprinted parts will also degrade. A short tool life will also mean that many different tools must be used to produce a large number of parts, so tool-to-tool variation becomes more important. Tool materials with thermal expansion behavior similar to that of the workpiece will reduce the various adverse effects of thermal stress caused by mismatch.

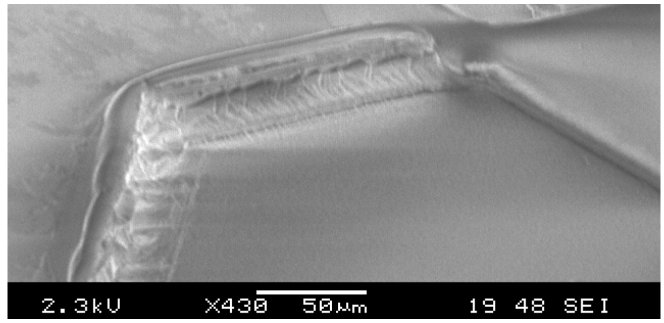

Fig. 3 Oblique SEM micrograph of a hexagonal feature on a PMMA part embossed with a silicon tool. The part was formed at 120°C under 0.6 MPa maintained for 3 min. The tool and part were separated at 50°C. The severe distortion of the left edge of the feature is attributed to thermal contraction. The edge of the part is toward the upper left. This image has been lightened and cropped from the original.

C. FLEXIBILITY

Flexibility can be defined as the ability to respond to changes in demand or customer needs [9]. The flexibility of a manufacturing process is related to the range of parts that can be made, and the amount of "pain" associated with changing from one product to another.

HME is capable of working with most thermoplastic materials, though limitations of temperature or force may reduce the range of possible materials that can be processed on a particular machine. Similarly, the temperature stability and chemical and mechanical properties of a tool material will limit its use to compatible workpiece materials.

The minimum feasible feature size, maximum aspect ratio (feature height/width for a raised feature), and feature density will vary among tooling production techniques. These tooling limits will define the variety of parts that can be produced. Demolding problems associated with material interaction or thermal contraction will impose further limits for certain combinations of tool and part materials and geometries. Certain processes may also limit the range of part characteristics that are producible. For example, some processes may only be able to produce a limited range of sidewall draft angles or a certain fineness of surface finish.

Process flexibility is also influenced by tooling costs, since high tooling costs must be amortized across many parts. Cheaper tools are better suited for rapidly changing, low-volume products.

D. COST

Tooling costs contribute directly to the overall per-part cost of the HME process. Tool life is another important consideration, since a low-cost, short-lived tool may have a similar per-part cost to a more expensive and more durable tool. The cost of rejected parts must also be offset in the price of good parts, so the quality of finished parts is another important consideration in the overall cost of the HME process as it relates to tooling.

TOOL PRODUCTION TECHNIQUES

A wide variety of tool materials and production techniques has been explored. Most any process capable of producing the desired 2-dimensional inverse features at the desired scale in an appropriate material is a candidate. Selecting a specific process and material for tooling depends on the required characteristics of the tool in terms of feature size, aspect ratio, precision, and durability.

The various processes available for the production of imprint tooling can be classified according to their mode of operation. The major distinction is between processes that produce the final tool directly and those that rely on an intermediate template. For the latter, the template itself must be produced somehow, and the quality and other characteristics of this template are just as critical to the final tool as the tool is to the imprinted part.

E.LITHOGRAPHY AND ETCHING

Photolithography and bulk-etching techniques borrowed from the fields of semiconductor processing and micro-electromechanical systems (MEMS) allow imprinting tools to be fabricated with sub-micron lateral feature dimensions and surface roughness as small as a few nanometers. For features deeper than a few micrometers, deep reactive ion etching (DRIE) is typically used [10]. DRIE can readily provide features with depth-to-width aspect ratios greater than 20, and does so by alternating every few seconds between the mildly anisotropic fluorine-based etching of silicon and the plasma deposition of an organic sidewall passivation layer. By varying the etching parameters, the taper angle of sidewalls can be controlled, providing for the exquisite refinement of tool design [11,12]. Meanwhile, quartz molds made with DRIE can be used for the casting of polymers that are subsequently UV-hardened by illuminating the resin through the mold.

Tool fabrication must ensure that demolding stresses after imprinting will not exceed the tensile strength of the substrate or tool material [13,8]. To this end, it is important to control sidewall roughness. The time-multiplexed nature of DRIE leads to a 'scalloped' profile on sidewalls with a characteristic dimension of tens to hundreds of nanometers. An advantage of DRIE is that its passivation material has a low coefficient of friction, so that an extra polymer deposition step after etching [14] leaves a useful surface coating on the tool (discussed further in section J).

Spatial variation of the etch rate in DRIE can be as great as 10% across a 150 mm-diameter silicon wafer, depending on the density and distribution of the pattern being etched [15]. The depth uniformity of features embossed with a silicon tool thus depends on careful control of the etching process.

The anisotropic wet etching of silicon is also common, using potassium hydroxide or tetramethylammonium hydroxide solutions [16,17]. In these cases, a silicon nitride or silicon oxide mask must usually replace the photoresist mask. Anisotropic wet etching restricts the tool geometries that are possible: potassium hydroxide, for example, will etch trenches in silicon with sidewalls sloped at 54.7° to the plane of a (100) wafer. The surface roughness of wet-etched tools is dependent on processing conditions such as etchant concentration.

Silicon tools are usually straightforward to fabricate and provide for high-quality prototypes. Considering the short tool lifetimes that this brittle material can offer, however, the fabrication of silicon tools is both energy-intensive and slow from a manufacturing perspective. Silicon is therefore not widely viewed as an appropriate material for heavily used imprint tools, although it may successfully be used to make master molds against which metallic or thermosetting polymer 'child' molds can, respectively, be plated or cast.

Polymer-on-silicon tools have been produced by spinning on to a silicon substrate, and then patterning, a layer of ultraviolet-curable thermosetting polymer such as mr-L 6000-XP (Micro Resist Technology, Berlin) [18] or SU-8 (Microchem Corp, Newton, MA) [19]. Features patterned in this way could approach 100 μm in depth. The lifetimes of photoresist-on-silicon tools are typically short because of the low adhesive strength of the photoresist-silicon interface.

F.MICRO-MILLING

Micro-milling can be considered to be a miniaturized version of classic macro-scale machining. Micro-milling is capable of producing tools from most metals, so material variety is high and tool durability is extremely good. Lee *et al* [20] report machining grooves with sub-micrometer variation. Mecomber *et al* [21] report machining an embossing tool from 7075 aluminum to produce 50 μm wide and 150 μm and 200 μm deep channels.

One important drawback for micro-milling is the presence of burrs and cutting tool marks. Wang [22] machined an embossing tool from commercially pure copper on a conventional 3-axis CNC milling machine using a 508 μm diameter flat end mill. Raised features on the tool were 500 μm wide, 50 μm tall, and 5000 μm long. Burrs and cutting marks were readily apparent, and were replicated very well in the embossed parts as can be seen in Fig. 4.

Various methods have been employed to reduce or eliminate burrs and machining marks in imprint tools. Dimov *et al* [23] compared various pocket milling strategies and found that the best results were obtained when the toolpath maintained a constant load on the cutter. Heaney *et al* [24] found that cutting tools coated with nano-crystalline diamond reduced burr formation and improved surface finish relative to un-coated cutters. Heaney *et al* also note that a sharp cutting tip radius is critical for good results. Jun *et al* [25] evaluated an atomization-based system for cutting fluid application and found that the system reduced cutting force variation and tool wear, and prevented burr formation. Horsh *et al* [26] found that micro-peening with 10-20 μm silicon carbide, alumina, or glass beads effectively removed cutting tool marks in aluminum and homogenized the tool surface finish.

Although minimum feature size is limited to tens of micrometers and surface finish is typically poor compared to other techniques, micro-milling of metals is a fast and cost-effective method for producing metallic imprint tools with aspect ratios of 3 or more [21]. For applications where poor surface finish is tolerable, micro-milling presents the most flexible, lowest cost option for producing durable metal tools.

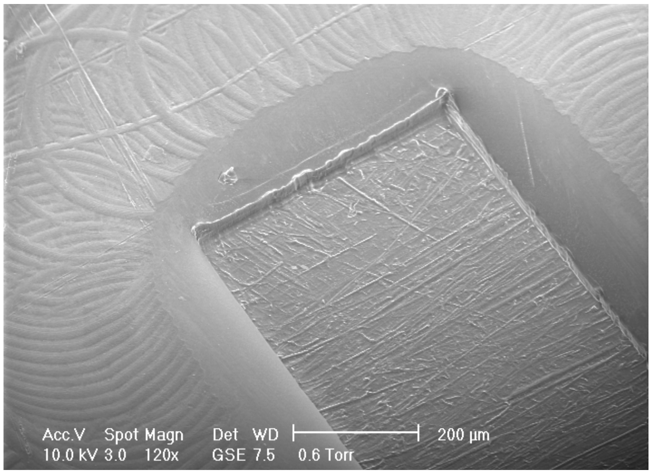

Fig. 4 Oblique SEM micrograph of PMMA embossed with a machined copper tool, reprinted from [22]. Note the replicated machining marks.

G. NON-TRADITIONAL MACHINING TECHNIQUES

At the macro scale, electric discharge machining (EDM) is well known for its ability to machine hard materials with precision and good surface finish. The two main candidates for embossing tool production are EDM contouring and die sinking. In EDM contouring, a pin-shaped electrode is moved through a three-dimensional path as it removes material. Although electrode wear can be a problem, this method is able to generate any three dimensional structure without undercuts, limited only by the minimum electrode diameter, which is currently around 5 µm [27].

In die sinking EDM, the main limitation is the process used to produce the electrode dies. Stampfl *et al* [28] report two processes by which they produce EDM dies from lithographically patterned silicon. In the first, a silicon wafer is patterned and etched to produce the desired features. A thin layer of titanium followed by electroplated copper is deposited. Finally, the silicon is etched away using KOH, leaving the desired copper electrode. In the second process, the silicon patterns with copper seed layers are further coated with a thin tungsten layer, which serves as a diffusion barrier. The silicon parts are then used as molds in a hot pressing operation to form a mixture of powdered tungsten and silver. This mixture is then sintered to produce the finished electrodes.

Uhlmann *et al* [29] review the various applications of micro EDM to micro-molding tools. They report that the minimum achievable surface roughness for contouring and die sinking micro-EDM are 0.2 µm and 0.1 µm respectively. Flushing of ablated particles and contaminants from the gap between the die and the workpiece becomes increasingly critical as feature size decreases.

For many EDM processes, as much effort is expended in producing the electrode as would be spent on the embossing tool itself in any other process. Micro-EDM is likely to be chosen only when embossing tool hardness is critical.

Imprint tools can also be produced by laser ablation machining. Pfleging *et al* [30] employ an excimer laser to machine tools from steel, alumina ceramic, and high-temperature polymers polyimide (PI), polysulfone (PSU), and polyetheretherketone (PEEK). They report surface roughness of 200-300 nm for steel, 180-215 for alumina, and less than 100 nm for PI. The polymeric tools must be coated with a thin layer of metal to prevent chemical interaction with the workpiece. This barrier layer delaminates after a few embossing cycles. The laser machining process was able to produce features in the tool less than 10 µm across with aspect ratios up to 10.

H. ELECTROFORMING (LIGA)

Electroforming metallic tools for manufacturing microstructures in polymers has a long history of use, from phonograph records [31] to holograms [32] to compact discs [33]. A more recent application is the LIGA process described in [34] and [35]. In this process, a deep layer of photoresist such as PMMA is applied to a substrate and patterned by synchrotron X-ray lithography. After the resist is developed, a metallic seed layer may be applied, and the template is placed in an electroforming bath. Metal is deposited onto the photoresist template to build up the tool features. The photoresist is then removed and the substrate with deposited metal features serves as the embossing tool. Alternatively, the plating may be continued until a thick layer of metal has built up over the whole template. The deposited metal is then removed from the template.

Electroforming relies on a template, so the minimum feature size, maximum aspect ratio, surface roughness, and dimensional accuracy depend on the process used to produce the template. This is generally some sort of photolithography, with the patterned resist serving as a template, producing excellent surface finish.

Griffiths used simulated X-ray dose and PMMA photoresist development to assess the limitations of the LIGA process [36]. He reports that the minimum feature size increased with resist thickness to the power of 0.61, and maximum aspect ratio is proportional to resist thickness to the power of 0.39. For instance, reported minimum producible feature width for positive and negative features is 1.3 µm and 1.9 µm respectively with a PMMA resist thickness of 500 µm. Sidewall offset (the amount of undercutting of mask features) is proportional to resist thickness to the power of 0.61 for positive features and large negative features, or the power of 0.68 for small negative features. Minimum offset for a resist thickness of 500 µm is 0.53 µm for positive features and 0.63 µm for negative features. Sidewall slope is found to be inversely proportional to resist thickness to the power of 0.46, but is insensitive to aspect ratio.

Advances in UV photolithography techniques are permitting higher aspect ratios without the need for synchrotron X-ray sources. Lorenz *et al* reported electroformed nickel tools with thickness of 450 µm using a SU-8 template, and other SU-8 features up to 1.2mm thick [37]. Zhang *et al* varied processing parameters such as baking, exposure, and developing times for SU-8 photolithography to find the optimal conditions for high quality microstructures which could then be used as a template [38].

The electroforming process itself presents some hurdles for high-quality tooling. The high temperatures and harsh chemical environment of the electroforming bath can cause distortion in the template. Griffiths *et al* used numerical simulations to predict the effect of thermoelastic strain and swelling due to water absorption for PMMA templates in aqueous nickel electroforming baths [39] They found that while the bottom of a PMMA feature is constrained by the substrate, the top will tend to expand due to temperature increase and water absorption. The relative error in structural width was found to be proportional to aspect ratio. For an aspect ratio of 10, the relative error magnitude could be as high as 14% due to water absorption for room temperature electroforming, and could rise to 28% at 50°C. Similarly, Luo *et al* have investigated the effect of template deformation for SU-8 [40]. They varied the electroforming time with all other parameters held constant, and measured the width of the resulting nickel features. They found that the width at top of a positive nickel feature was as little as 82% of the width at the bottom. They note that the resulting sloped sidewalls could be beneficial for de-molding during hot embossing.

A wide variety of transition metals may be successfully electroformed with specialized bath chemistries, including iron, nickel, copper, tin, lead, gold, silver, chromium, rhodium, indium, and many alloys [41]. Stein *et al* report electroforming an alloy of nickel and cobalt that has superior strength and hardness compared to pure nickel [42]. Zhang *et al* report using Argon plasma to change the wettability of SU-8 to permit electroforming with copper [43]. Tian *et al* have fabricated composite tools by adding PTFE to the nickel electroforming solution [13]. The PTFE is incorporated into the deposited nickel, reducing its adhesion to PMMA during embossing.

The long history and versatility of electroforming strongly recommend it as a tool production process for imprinting-based manufacturing. Although the process is time-consuming and expensive, history has already shown that electroforming is the most economical technique for producing precise, micro-structured metallic tools for high-volume manufacturing of polymeric products.

I. CASTING

There has been some interest in producing embossing tools by casting thermosetting polymers over templates. This has been motivated by the relative simplicity and speed of casting materials such as PDMS over SU-8 templates patterned by photolithography. The similarity of a polymer tool's thermal expansion coefficient to that of the workpiece is also expected to reduce problems caused by thermal mismatch.

Narasimhan and Papautski [44] had good results using cast PDMS tools with feature widths from 150 to 600 µm and depths from 90 to 250 µm. Long embossing times up to 1 hour were required because of the low stiffness of PDMS. The long hold time allowed stresses in the part to relax so the PDMS tool could rebound to its original size. A harder formulation of PDMS was found to reduce the required embossing time. After 20 cycles, low aspect ratio tools exhibited no signs of wear, however, higher aspect ratio tools showed some permanent deformation. Xing *et al.* [45] also had success using PDMS tools to emboss PMMA.

Roos *et al.* [46] created polymeric tools by casting a UV-curing polymer over patterned silicon wafers. The features ranged in size from 100 nm to 100 µm, and were 300 nm high. Despite anti-adhesive treatment, thermal contraction mismatch resulted in damage to the tools when they were cooled while still in contact with the PMMA parts.

J. SURFACE TREATMENTS

In many cases, some force must be applied to separate the part and tool after embossing and cooling. These de-embossing loads have important consequences, as the part or tool may be damaged during separation [17,7], and in extreme cases, difficulty of separation will limit the types and density of features that can be produced successfully. Both chemical interaction and physical "locking" may play a role in this adhesion, but certain treatments may be applied to tools to mitigate these effects.

Jaszewski *et al.* [47] investigated PTFE layers deposited onto embossing tools by either ion sputtering or plasma polymerization. They measured the amount of fluorine transferred from the tool to the part using X-ray photoelectron spectroscopy in order to assess the wear rate of the anti-adhesive coating. They found a high amount of fluorine transfer initially, falling off to a steady low amount by the 10^{th} cycle. They attribute the high initial material transfer to the diffusion of low molecular weight species into the part, and the low steady transfer to abrasion. This hypothesis was confirmed by embossing with a pure PTFE flat sheet, for which material transfer similar to the low steady rate was observed. Their results suggest that such coatings will have a finite lifetime, though this may be hundreds of cycles.

Wissen *et al.* [48] applied ((1,1,2,2 H perfluorooctyl)-trichlorosilane to both silicon and epoxy tools by gas-phase deposition. Contact angle measurement of treated and untreated tools showed that the molecular monolayer significantly reduced the surface energy of the tool. Reducing surface energy leads to reduced adhesion. Contact angle was again measured after 6 cycles, and was found to be unchanged.

CONCLUSION

While the feasibility of HME with a variety of tools has been demonstrated, questions related to manufacturing have received less attention. The consistency and repeatability of many tooling production techniques have not been completely characterized. Each tooling material and production technique has unique fundamental limits in terms of minimum feature size, aspect ratio, surface finish, and other attributes. These limits place additional constraints on production quality and tool durability. In many cases, however, these constraints are not yet known.

Etching of silicon currently provides the smallest features. However, aspect ratio is somewhat limited and tool durability may be problematic. Micro-milling of metals is capable of producing durable tools very rapidly, but the minimum feature size is rather large, and surface finish is poor. Direct use of photoresist is feasible and provides small features and high aspect ratios, but durability is extremely limited. Template-based techniques introduce an additional critical step, since the quality of the tool is dependent on the quality of the template as well as the characteristics of the technique. Electroforming techniques such as LIGA have a long history and great potential for micro- and nano-imprinting tooling, however, template quality and dimensional stability may present obstacles. The repeatability and precision of cast-polymer tools may be limited, given the magnitude of dimensional shrinkage during curing.

Imprinting techniques have demonstrated great potential for manufacturing of polymer-based devices with features at the micro- to nanometer-scale. In order to realize this potential on an industrial scale, fundamental issues related to mass production, such as production rate and quality, must be addressed. Imprint tooling strongly influences the characteristics of the final product, and the production and quality of tooling is a vital concern for micro- and nano-manufacturing.

ACKNOLWDGEMENT

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