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## Design and validation of a novel master-making process chain for organic and large area electronics on flexible substrates

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### ABSTRACT

This paper presents a novel process chain for fabrication of replication masters for serial manufacture. The proposed process chain is validated for serial fabrication of (large area) organic electronic devices on flexible substrates. The advantages and limitations of the component technologies in the proposed manufacturing route are discussed and their interdependencies in a process chain for producing both 2.5D and 3D nano- and micro-structures are analysed. The proposed master-making route relies on using different technologies for micro-structuring and sub-micron and nano patterning that are applied to the fabrication of Ni shims incorporating different length scale features. In particular, the capabilities of photolithography as a micro-structuring technology were combined with those of FIB machining to add sub-micron and nano-features on micro patterned fused silica templates. Then, by applying UV nanoimprint lithography such templates were validated and their nano and micro-structures were consistently replicated in one step. Finally, the feature transfer of such imprints onto Ni shims was also successfully accomplished with only minor deviations from the target dimensions.

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### 1. Introduction

The most important requirement for achieving a high-throughput production of micro-components is the design of viable manufacturing platforms for their cost-effective fabrication. A number of technologies have been utilised for mass production of non-electronic micro-components. CD and DVD data storage devices, various micro fluidic devices, sensors, optical switches, micro lenses and holograms for data protection imprinted on credit cards and bills are among the most well known products that incorporate micro and nano scale functional structures and at the same time are produced in large series. In spite of the diversity of these application areas, the most viable micro fabrication technologies for their high-throughput manufacture are different implementations of micro-injection moulding and roll-to-roll (R2R) processes. A key requirement for successful application of these technologies is the availability of precision production master having a long lifetime that can deliver a very high yield.

Currently, the process of Nickel electroforming proves to be a viable and widely used method for producing replication tools [1] due to the fact that it provides for a good hardness and wear

resistance. The process also allows features of more complex shapes and various sizes to be replicated. Moreover, a very wide range of technologies can be used to fabricate electroforming masters with desired structures, e.g. laser ablation,  $\mu$ EDM, photolithography, EB lithography and focussed ion beam (FIB) machining. However, the use of any particular technology is viable only within a given optimal processing window in regards to achievable resolution, surface quality and removal/structuring rates. Therefore, usually a sequence of processes has to be applied for machining of complex topographies, which incorporate different length scale features. While the increase of the process steps leads to an increase in the overall uncertainty and total error associated with any selected manufacturing route, a well-designed process chain can have a significant positive impact on accuracy and cost-effectiveness of the whole master-making process [2]. Therefore, the process chain design should be paid a special attention in order to achieve the targeted compatibilities of component technologies, and at the same time to minimise the effects of various factors that can influence their overall performance. It is still a challenging task to combine technologies with different cost-effective processing windows, and thus to design viable tool-making process chains for producing components with both micro- and nano-features and/or realising complex 3D shapes.

Some attempts have been made to develop such process chains [3,4] however further research is necessary to address the complex

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issues that arise in achieving a length-scale integration and 3D patterning simultaneously in a given process chain.

In this paper a novel combination of complementary technologies is utilised to fabricate masters for serial replication. To validate such a process chain, the produced masters were used for R2R serial imprinting of organic electronic devices.

## 2. Process chain design

The proposed process chain for fabricating Ni shims that can be used as masters for serial replication is presented in Fig. 1. It employs the Focussed Ion Beam (FIB) technology to machine precisely very complex nano and micro 2.5D and 3D structures [5]. However, this technology can be applied for patterning accurately and cost-effectively only relatively small areas, e.g. up to  $100 \times 100 \mu\text{m}$  without stitching, due to its low material removal rates. Thus, it is not suitable for structuring bigger surfaces. Hence, to make the best use of this process, it should be applied only for patterning of relatively small areas, e.g. the machining of sub-micron and nano-structures on nanoimprint lithography (NIL) templates [6]. Micro-structuring, which usually is required for patterning larger areas of the masters/templates, can be performed more efficiently by employing other technologies such as photolithography or laser ablation. Therefore, by structuring a template in two stages, first by performing micro and then high resolution sub-micron and nano patterning, the fabrication time can be reduced drastically and thus the cost-effectiveness of such master-making process chain will be improved significantly. Then, by employing Step and Flash Imprint Lithography (S-FIL) [7] it is possible to use such templates to pattern 4" or 8" wafers, which can be employed as masters for the subsequent electroforming process. Finally, Ni shims fabricated in this way can be integrated onto the R2R rollers, and thus, used for imprinting of UV and thermally curable polymers.

A more detailed description of the different "component" technologies and steps that are integrated in the proposed process chain is provided in the following sub-sections.

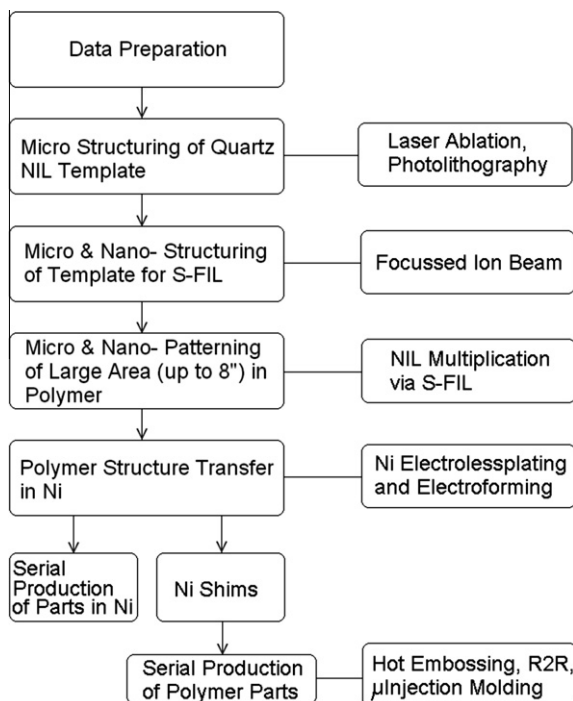


Fig. 1. The stages of the proposed master-making process chain.

### 2.1. Data preparation

This initial stage of the process chain involves the CAD design of the desired structures and the follow-up data preparation for their machining. For relatively simple features like 2.5D channels bit-map data can be designed externally and uploaded into most of the FIB systems, or can be created directly using their build-in pattern generators. A more sophisticated approach requires the use of a lithography software and hardware like Elphy Quantum (Raith GmbH) or Nanomaker where various 2D shapes can be easily designed, multiplied, and if necessary the respective exposure doses specified. However, the generation of complex 3D shapes like diffractive optical elements, necessitates a different approach. It was proposed to realise such 3D structures by designing their features in any 3D CAD package and then, by following a sequence of translational operations, the 3D geometry to be converted into a stack of layers ordered along the vertical axis of the model [8]. After such a 'slicing' step, the model is exported into a GDSII stream file format and each GDSII layer represents a set of exposure pixels defining a cross-section, a slice, of the model at a given point along its vertical axis.

### 2.2. Template structuring

The next stage of the process chain involves fabrication of the template necessary for UV-NIL. The patterning of the template mesa is performed in two stages: micro-structuring and nano structuring, respectively. The material used for the UV-NIL templates is fused silica as it has to fulfil several very important requirements, in particular, the workpiece must have a good transparency in the UV region, it should be amorphous, since the lack of polycrystalline structure is a pre-requisite for patterning surfaces with very low roughness by dry etching and/or FIB machining, and must have a good wear resistance.

#### 2.2.1. Micro-structuring

This stage involves mainly the usage of two technologies: photolithography and laser machining, which are cost-effective for micro-structuring relatively large areas of the template with a good dimensional accuracy and surface roughness. In another study, it was reported that F2 laser ablation when used for direct structuring or in projection mode is suitable for machining the mesa of fused silica templates without triggering any material crystallisation [9]. The later is an important issue since any phase transformations would have affected the machining results of any subsequent nano-structuring steps employing FIB. In case of photolithography, after exposure, a follow-up step is required to transfer the pattern to the mesa by dry etching. It is worth pointing out that there are certain drawbacks associated with such a micro-structuring step. In particular, if photolithography is employed, the resolution is limited to 1 micrometer [10], a mask is required, the 3D patterning capabilities are limited and a subsequent dry etching is necessary. F2 laser ablation is an attractive alternative solution due to its direct structuring capability, however it should be noted that this machining route is significantly more expensive than the photolithography one.

#### 2.2.2. Sub-micron and nano-structuring

The next stage in the process chain is FIB machining of sub-micron and nano-features over the pre-existing micro-topography.

The FIB milling process offers many advantages, e.g. flexibility, high resolution and high surface quality that are extremely important for master making [5,11]. However, a major disadvantage of this technology is its relatively low removal rates. To address this issue a multi-ion beam concept was proposed that combines the high resolution capabilities of the FIB technology with the higher

throughput advantage of parallel lithography systems. In particular, to satisfy the requirements for high productivity, a projection maskless nano-patterning (PMLP) system is under development [12]. Its working prototype, which incorporates 48,000 beams working in parallel, demonstrated a significant increase of the removal rates and improved resolution compared to conventional single focused ion beam systems.

Another important issue when structuring processes are integrated in process chains is the alignment of new features to any pre-existing features/topography on the wafer or workpiece. In the proposed chain this alignment could be realised either by manually positioning the sample stage while inspecting the sample in SEM or FIB imaging mode, or automatically, by using “feature recognition” option available in some FIB systems to find alignment marks machined in the previous processing steps.

Finally, an ion beam sputtering simulation software can also be employed to reduce some negative effects such as re-deposition of sputtered material and over-etching [13]. Its utilisation as a data pre-processing step before FIB milling makes possible the optimisation of the process parameters and even model modifications in order to counteract the material re-deposition.

### 2.3. UV-NIL imprinting

The multiplication of the features, structured on the fused silica templates, onto a wafer is carried out through UV-NIL. Although thermal NIL is widely employed for micro and nano replication of thermosetting polymers, it is difficult if not impossible to replicate micro and nano-features in one processing step due to issues related to the difference in polymer flow in micro and nano patterns, big thermal shrinkage and variations of residual layer thickness [9]. UV-NIL and especially the Step and Flash Imprint Lithography (S-FIL) process is considered to be a better option for the simultaneous imprinting of nano and micro patterns as they offer several major advantages [7] such as: uniform patterning of large surface areas, due to low imprinting forces and low resist viscosity, absence of thermal expansion, uniform thickness of the residual layer, capability to imprint micro and nano-features simultaneously, imprinting of complex 3D topography and the possibility to use fragile substrates. Therefore, the S-FIL technology was selected for the proposed master-making process chain.

However, it should be noted that the big disadvantage of this technology is the limited number of resists suitable for the S-FIL process and the constraints regarding the height of the imprinted features, typically up to 500 nm.

### 2.4. Electroforming

This stage is necessary in order to fabricate exact replicas in Ni of the S-FIL imprinted wafers and then to utilise them as masters for serial replication. The electroforming process allows very precise replicas of wafers with nano- and micro-topography to be produced [14]. Furthermore, it is a relatively fast and inexpensive way for fabricating robust tools, Ni shims, which can be used as hot embossing stamps, skins for R2R rollers and injection moulding inserts for serial production of polymer components [15].

Finally, it should be noted that the proposed master-making process chain can also be implemented for serial production of components in Ni resembling the direct LIGA process [16]. Since the latter is not within the scope of this paper it will not be further discussed.

### 2.5. Serial replication

The proposed master-making process chain is suitable for a number of serial replication technologies for scale-up micro man-

ufacture including micro-injection moulding ( $\mu\text{IM}$ ), R2R imprinting and hot embossing (HE) together with some of their variations such as compression injection moulding and R2R thermoforming [17]. A common challenge in all of them, especially when micro and nano-structures have to be replicated simultaneously, is the reliable and cost-effective fabrication of stamps or inserts. This, together with the necessary optimisation of the respective replication process, determines the overall effectiveness of such manufacturing platforms. The specific characteristics of these high-throughput replication processes are discussed below.

$\mu\text{IM}$  and HE appear to be the two most industrially viable processes for fabrication of micro-components [18]. Due to its specific processing conditions, HE is widely used for replicating structures/features with dimensions in the sub-micron range and high aspect ratios. However, the HE cycles are relatively long, usually 5–10 min, and therefore this technology is more suitable for small to medium series production and prototyping [18,17]. Conversely, the shorter cycle time in  $\mu\text{IM}$  determines its effectiveness for large series production, more than 1000 parts.

Injection compression moulding can be considered as a hybrid process that combines the capabilities of  $\mu\text{IM}$  and HE. Especially, the shrinkage of the polymer during the cooling stage of the process can be minimised during its compression stage, which leads to a better replication accuracy. This technology is widely used for mass production of CDs and DVDs [17].

R2R imprinting has attracted researchers and industry with its potential for a high-throughput manufacture [19,20]. The process is capable of replicating micro and nano topography at relatively high speed. Depending on the set-up, thermo and UV curable resist materials can be used. One of the major difficulties associated with this technology is the structuring of the rollers, particularly at nano scale. A variation of this technology is R2R thermoforming. It is used for forming of foils with complex cross-sections as it allows easy demoulding but high aspect ratios could hardly be achieved.

In this research to validate the proposed process chain, the produced masters were used for R2R serial imprinting.

## 3. Experimental set-up

### 3.1. Test device

To test the proposed master-making process chain a single device organic thin film transistors (OTFT) was selected. With commonly used low-cost industrial patterning and printing technologies for producing such devices, the maximum operation frequencies that can be expected from organic logic gates are in the 10 kHz range due to the limited resolution of these techniques; the minimum achievable channel length is in the range of 10  $\mu\text{m}$ . By applying the new process chain it will be possible to reduce the critical dimensions of OTFT towards the sub-micron channel length regime, and thus to achieve switching frequencies in the MHz region. Apart from achieving this downscaling, the minimisation of the parasitic capacitance is another contributing factor in reducing the switching time. To validate this, together with the capabilities of the proposed process chain, the OTFT design presented in Fig. 2 was selected. Fig. 2a shows the photomask design. The test device comprises of different layouts of pads, connectors-width-to-distance tests, areas for channels to be machined by FIB (Fig. 2c) and various test-structures parallel and perpendicular to the R2R imprint direction.

The main functional structure of the device consists of multiple micro-channels as shown in Fig. 2b that are interconnected through micro- and sub-micron trenches. Also, this design is a good example of a proper implementation of the function and length-scale integration concept [21] as it incorporates both micro-

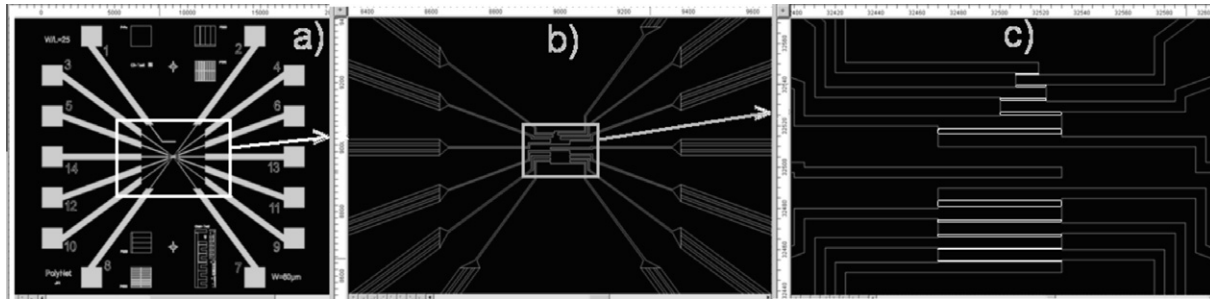


Fig. 2. The OTFT design (a) overall view, (b) magnified central area, and (c) the interconnecting trenches to be structured by FIB.

and sub-micron dimensional features on a millimeter-scale area. Magnified image of the center of the OTFT design is shown in Fig. 2c. As it can be seen on the sketch, the interconnecting channels vary in size. The minimum trench width is 450 nm, followed by 600 nm, 900 nm, 1.2  $\mu\text{m}$ , 2.4  $\mu\text{m}$  and 5  $\mu\text{m}$ . Their length is determined by the distance between the big micro-channels and is in the range from 11.25 to 60  $\mu\text{m}$ . The targeted depth of the whole structure is 450 nm.

### 3.2. NIL template fabrication

A fused silica template with a mesa, with dimensions  $25 \times 25$  mm and height of 30  $\mu\text{m}$  was manufactured by grinding, prior to the micro-structuring stage. Then, the micro channel structures, including the 5  $\mu\text{m}$  interconnecting trenches in the template, were produced by photolithography. Microposit S1813 photoresist was spun on the mesa at 4000 rpm giving a film thickness of  $\sim 1.8$   $\mu\text{m}$  after baking at 97  $^{\circ}\text{C}$  for 2 min. The template was then placed in the Karl Suss mask aligner for exposure. The resist was exposed using a UV light (365 nm wavelength) for 6 s. A developer solution with a ratio of 6:1 DI water to Microposit developer 2401 was used. The developing time was 45 s. The template was then rinsed in DI water and then dried with N<sub>2</sub> gas.

Following the photolithography step, dry etching was used to transfer the pattern into the fused silica. To carry out this operation an Oxford Instrument PlasmaPlus 80 reactive ion etching (RIE) system was used. The resist acted as a dry etching mask with a selectivity of 3:1 to the fused silica. A mixture of 80% carbon tetrafluoride (CF<sub>4</sub>) and 20% oxygen (O<sub>2</sub>) was used as an etching gas. The template was etched for 20 min at 40 mTorr pressure applying 100 W RF power.

The sub-micron and some of the micro-channels on the active area of a UV-NIL template were machined by FIB milling on a Carl-Zeiss XB 1540 FIB/SEM cross-beam system. The build-in pattern generator was used to design the channels. To avoid charging, the template was coated with 20 nm Cr film. The sub-micron and micron trenches up to 2.4  $\mu\text{m}$  were machined on the template by applying probe currents ranging from 50 to 200 pA. Their alignment to pre-existing structures on the template mesa was performed manually by utilising the imaging capabilities of the FIB system.

### 3.3. NIL and electroforming

An UV-NIL system, Imprio 55, was employed to multiply the template topography on a 4" wafer employing the S-FIL process. The imprinting was performed on a double-sided polished silicon wafer spin-coated with a 60 nm thick layer of DUV30J, which served as a planarization layer and a bottom anti-reflective coating (BARC). After stripping the Cr coating the wafer and the template were mounted into the NIL system. Utilising a drop-on-demand

build-in dispensing system, an array of pico-litre sized drops of a low viscosity monomer, Monomat™, was spread over the imprinting field before the template was lowered onto the drops. When the surface tension of the liquid is broken, capillary action draws the fluid into the template features and thus spreading the resist across the template active area. Then, the fused silica template was exposed to 355 nm UV light in order to cross link the monomer and solidify it. Finally, the template is withdrawn, leaving an exact replica of the structured template on the wafer surface. To multiply the template topography the process is repeated to imprint an array of fields.

For the 4" (round) Ni shim fabrication a commercial electroforming system, Digital Matrix SA/1m, was utilised. The parameters used to carry out the electro chemical deposition were as follows: electrolyte: Nickel Sulfamate, bath temperature: 50  $^{\circ}\text{C}$ , head rotation speed: 30 rpm, pH 3.96, current density: 0.1/0.5 A/dm<sup>2</sup>, waveform: Spiked down/Ramp down, cycle time: 10 ms. The obtained shim had a thickness of 100  $\mu\text{m}$ .

### 3.4. R2R hot embossing

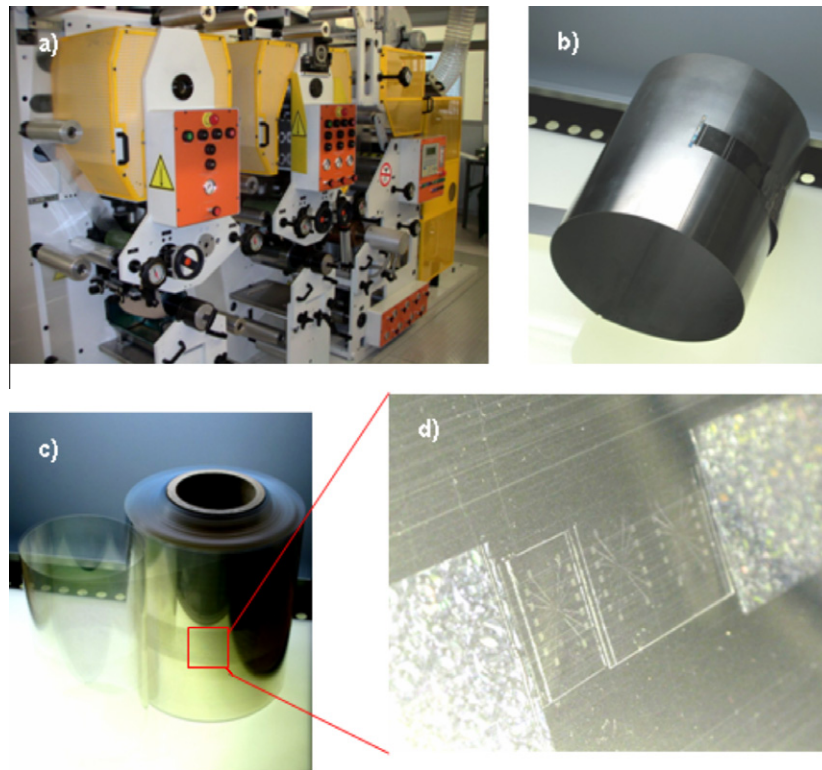
The VTT's Pico pilot production facility, shown in Fig. 3a, was used to test the shim produced applying the proposed process chain. The machine consists of two gravure printing units for thin film deposition and a hot embossing unit. The prefabricated shim was laser welded to a sleeve which was then mounted on the R2R hot embossing machine. The sleeve with the shim is shown in the Fig. 3b.

For R2R tests, mr-I 7030E resist was deposited using forward gravure printing on top of PET Melinex ST504 substrate. The printing cylinder parameters were: mesh 64 lines/cm, depth of cell 44  $\mu\text{m}$  and transfer volume 13 ml/m<sup>2</sup>. Printing speeds of 2, 4, 6 and 8 m/min were used during the resist deposition and then dried with hot air, which temperature was 120  $^{\circ}\text{C}$ . Based on a carried out visual inspection the best printing speed for consistent deposition of high quality films was determined to be 4 m/min. The layer thickness of the resist was measured using a Wyco optical profilometer and it was 300 nm.

After the resist deposition, R2R hot embossing was performed. The embossing speed was 10 m/min, and the applied pressure was 4 bar at a temperature of 120  $^{\circ}\text{C}$ . Fig. 3c and d show the hot embossing roll used in the carried out tests and a zoom-in image of the replicated structure, respectively.

### 3.5. Inspection

SEM images of the features were taken using Carl-Zeiss XB 1540 at each stage of the process chain and then analysed with the build-in SmartSEM software. FIB (50 pA) probe current was used for making cross-sectional cuts of the inspected structures (Fig. 4). To minimise the measurement error all images were taken



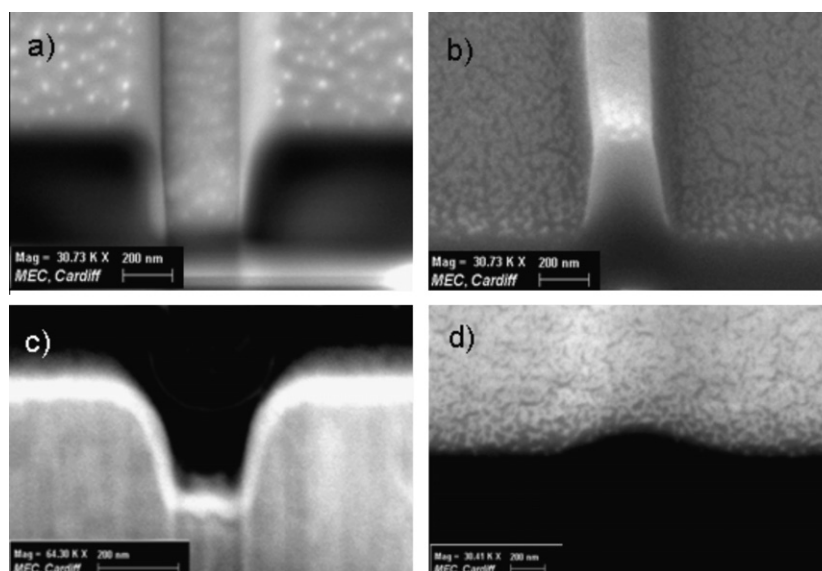
**Fig. 3.** R2R replication process (a) VTT's Pico R2R hot embossing installation, (b) a roller with an integrated Ni shim, (c) an imprinted PET roll and (d) a zoom-in of the imprinted PET roll.

at the same magnification, 3 kV EHT, and the same SEM aperture. After a calibration with a reference sample, the measurement error of the instrument in XY direction was assessed to be in the range of 1–3%. All the samples were measured at 36° tilt in order to be able to image the vertical walls of the structures. In addition, the “tilt compensation” option of the SmartSEM software was utilised in order to measure accurately the features’ height. Cross-sectional SEM images of the 450 nm channel on the quartz template, S-FIL im-

printed wafers, the Ni shim and the R2R replica are presented in Fig. 4.

#### 4. Discussion

The experimental results obtained after each step of the proposed process chain are discussed in this section. As the OTFT



**Fig. 4.** SEM images showing a cross-sectional cut of a 450 nm channel on (a) the fused silica template, (b) the NIL imprinted replica of the template, (c) the Ni shim fabricated from the NIL imprinted master and (d) the R2R replica of the shim.

channels had tapered sided walls, the measurements of their width were taken at the bottom, top and in the middle of each structure. The full width at half maximum (FWHM) value of the channels was used to compare the results after each step of the proposed process chain.

The difference between the width of the channels of the template and the NIL imprint, in percentage, is shown in Fig. 5. It is not difficult to see that there is a tendency the difference to decrease with the increase of the channel width. This can be explained with the fact that with the increase of the width the aspect ratio of the features decreases due to the constant depth of the channels. In particular, it is more difficult to machine high aspect ratio channels and any deviations during the template structuring affect the follow-up replication processes. In addition, it should be taken into account that the relative measurement error, in percentage of the nominal width, increases with the decrease of the nominal dimensions of the channels. The average difference for all channels is around 5.6%, which is more than have been expected. This result could be explained with the resist shrinkage after the UV curing, which was expected to be approximately up to 3% for Monomat™ [7], the measurement errors and the used exposure parameters, in particular curing time, that were not optimised.

Also, Fig. 5 shows the difference in percentage between the channels' widths of the imprints and the Ni shim produced with them. As expected the tendency of the features with bigger lateral dimensions and low aspect ratios to replicate better was observed again. With an average deviation of 1.5%, the Ni shim appears to be an excellent replica of the NIL imprints.

The changes of the FWHM values for all channels after each processing step are shown in Fig. 6a and b. In spite of the fact that there is a relatively big difference between the size of the channels on the R2R replica and the template it is clear that the plotted lines are relatively parallel, since the difference of the respective FWHM values for all channels is almost constant, approximately 300 and 500 nm for the sub-micron and micron channels, respectively. This shows a high level of consistency in replicating the fused silica and NIL masters, and also indicates very good process compatibility between the component technologies in the process chain, especially for the targeted length scale range.

The big deviation in the FWHM values of the R2R replica channels and those of the Ni shim can be explained with the use of embossing parameters that were not optimised, especially the embossing temperature. In addition, the phenomenon known as polymer relaxation is expected to contribute to this [22,23]. However, it is important to state that this deviation can be minimised

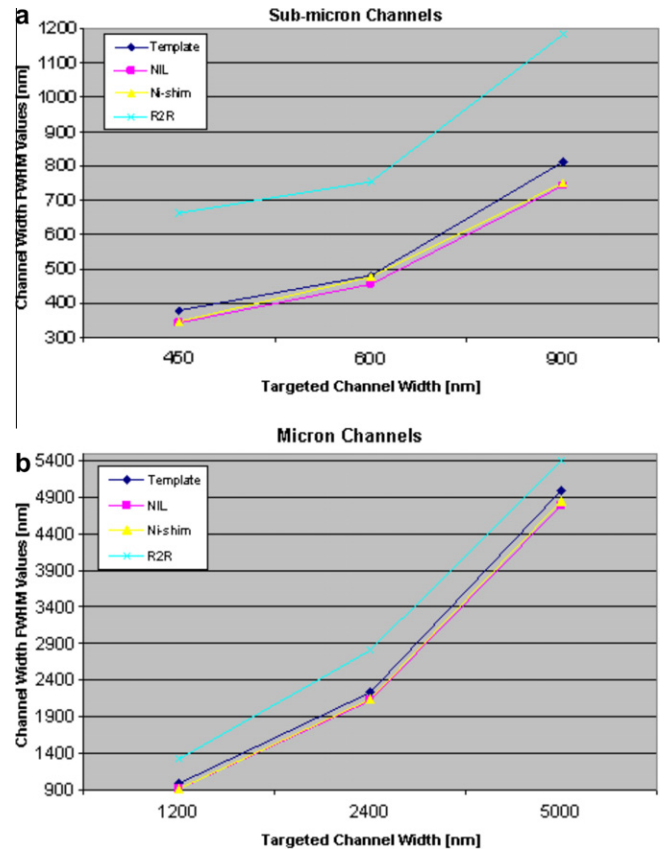


Fig. 6. FWHM of the channels' width, (a) the sub-micron channels (b) the micron channels.

by modifying the device models or by introducing measures to “freeze” the polymer immediately after the thermal imprinting step. Since the main objective of this research is to design and validate a process chain for a reliable fabrication of serial replication tools, the optimisation of the R2R process parameters is not discussed in this study.

The significant difference between the channels' aspect ratios on the Ni shim and on the R2R replica – Fig. 7, can be explained again with the effects of the polymer relaxation after the replication step and the insufficient embossing temperature. It can also

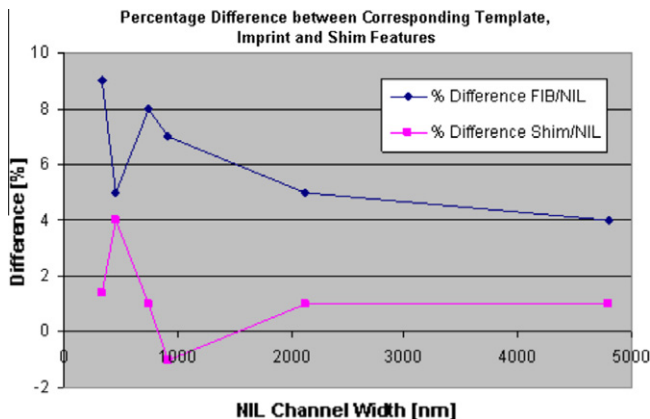


Fig. 5. The percentage difference in imprint features' dimensions with regard to the template and the Ni shim.

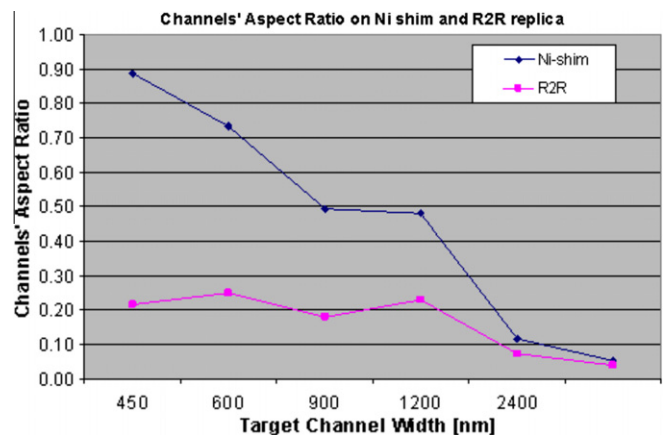


Fig. 7. Channels aspect ratio for the Ni shim and the R2R replica.

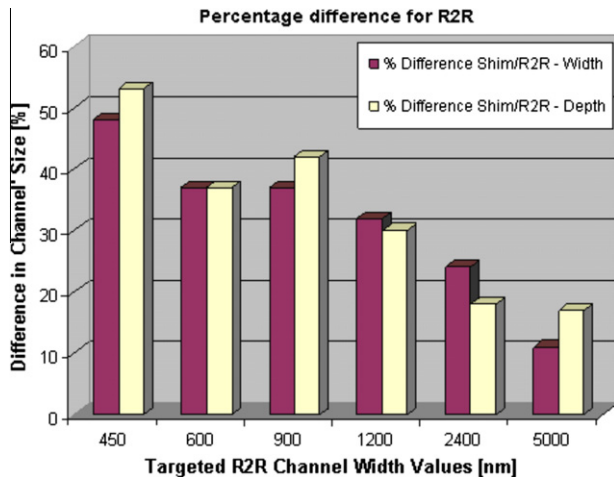


Fig. 8. The percentage difference between R2R and Ni shim features in vertical and horizontal directions.

be seen from Fig. 7 that these effects on the replica are strongly pronounced for the features with higher aspect ratios. In addition, Fig. 8 shows that the difference in percentage between the widths of the channels on the shim and on the R2R replica, and their respective heights are almost the same. This can be attributed to the fact that after de-moulding, the molecular chains tend to regain their original shape, and thus to keep constant the volume of the structures. Thus, the channel height decreases in order to compensate for any expansions in the XY plane.

Even though it is difficult to estimate the influence of different factors at the various steps of the proposed process chain on its overall performance, the following ones are considered to have the highest impact: the measurement uncertainty including the system calibration and the human factor, the optimisation of the processing conditions of different component technologies, and material properties, especially the materials' response to UV and thermal curing.

## 5. Conclusions

A cost-effective process chain for fabrication of Ni shims incorporating different length scale features was proposed and validated for R2R imprinting of organic electronic devices. In this study, the capabilities of photolithography as a micro-structuring technology are combined with those of FIB milling to fabricate templates incorporating sub-micron and nano-features on top of pre-existing micro structures. S-FIL process was employed for consistent multiplication of such templates on 4" wafers in order to replicate reliably different length scale features in one step. The transfer of such polymer replicas into Ni shims was also successfully implemented resulting in relatively negligible deviations from the targeted dimensions. The proposed fabrication route for fabrication of Ni shims incorporating a wide range of micro and nano-features can be used for high-throughput fabrication of organic electronic devices on flexible substrates employing serial replication technologies like R2R imprinting.

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