

Formation of Round Channel for Microfluidic Applications

A. Zahra, G. de Cesare, D. Caputo, A. Nascetti

Abstract—PDMS (Polydimethylsiloxane) polymer is a suitable material for biological and MEMS (Microelectromechanical systems) designers, because of its biocompatibility, transparency and high resistance under plasma treatment. PDMS round channel is always been of great interest due to its ability to confine the liquid with membrane type micro valves. In this paper we are presenting a very simple way to form round shape microfluidic channel, which is based on reflow of positive photoresist AZ® 40 XT. With this method, it is possible to obtain channel of different height simply by varying the spin coating parameters of photoresist.

Keywords—Lab-on-Chip, PDMS, Reflow, Round microfluidic channel.

I. INTRODUCTION

MICROFLUIDIC devices have significant applications in biological processing and chemical reactions and also show potential use in “lab on chip” (LoC) technology.

Lab-on-Chip (LoC) device is an example of system yielding a sensor like system requiring minimal quantities of biological samples. A variety of recent technological breakthroughs in the fabrication of thin film physical [1] and optical [2] sensors have made possible the development of lab-on-chip (LoC) systems where several functional modules are integrated onto a single substrate [3], [4]. Each module has a specific function in the sample-treatment chain: of sample injection, reaction, separation and detection.

The high integration level of the LoCs allow to accomplish complex chemical or bio-chemical functions of large analytical devices on a single sensor like system with a fast response time, low sample consumption and on-site operation.

These LoCs are very useful for early diagnosis of various diseases [5], [6] like glioblastoma multiforme (GBM), medulloblastoma (MB), by performing PCR (Polymerase chain reaction) to detect the specific cancer stem cells (CSC).

The use of microfluidic devices help to reduce the laborious work and experimental error with increase in throughput. Sizes of microfluidics devices may range from micrometer to

few square centimeters. These devices are capable to handle fluid volume even smaller than pico liter scale. Glass is a popular substrate but it is not widely used for making microfluidic channel, due to its long process time. Nowadays, polymeric materials such as PDMS, polymethyl methacrylate (PMMA) are often used for microfluidic system fabrication. PDMS and PMMA have many advantages over the glass substrate [7] such as low cost and they are easy to use. Generally a microfluidic device consists of a channel with inlet, outlet and a reaction chamber as shown in Fig. 1.

Many researchers groups [8], [9] have reported the leakage of fluid from the reaction chamber while performing a chemical reaction. This leakage is due to heat generated or consumes during reactions, which increase the pressure inside the chamber and therefore induces the failure of the experiment.

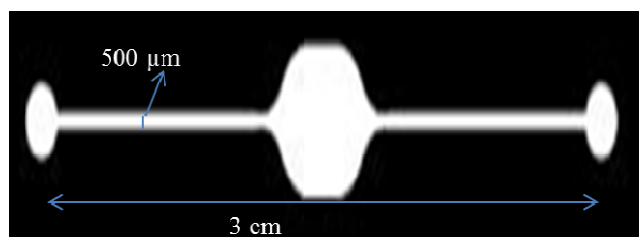


Fig. 1 Microfluidic Channel with inlet and outlet and a reaction chamber

To overcome this problem it is necessary to confine the liquid into the reaction chamber. One way to solve this problem is to develop membrane type microvalves along the flow channel. When the temperature of these valves is increased they start expanding into the round shape and try to close the microfluidic channel. It happens due to the pressure development while heating the valves. However, the shape of microfluidic channel made by conventional approach is rectangular. This rectangular channel is not completely match with the round shape of membrane deformation [8] which also results to the leakage of fluid. The complete procedure to close the channel while heating the microvalves is depicted in Fig. 2. The only way to remove this problem is to make perfect sealing between the microfluidic channel and membrane of the valves. Round shape of PDMS microchannel is highly desirable for perfect matching with membrane type microvalves.

Round shape PDMS flow channels also have significant impact on the maximum pressure head and flow rates, both of which are improved by replacing square flow channels with

A. Zahra is with Center for Life Nano Science at Sapienza, “Istituto Italiano di Tecnologia” Rome 00184 Italy (phone: +39-0644-585475 e-mail: andleeibitd@gmail.com, Zahra@dit.uniroma1.it).

G. De Cesare is with Department of Information Engineering, Electronics and Telecommunications “Sapienza Università di Roma”, Rome 00184, Italy (phone: +39-0644-585431 email: decesare@die.uniroma1.it).

D. Caputo, is with Department of Information Engineering, Electronics and Telecommunications “Sapienza Università di Roma”, Rome 00184, Italy (phone: +39-0644-585832 email: caputo@die.uniroma1.it).

A. Nascetti, is with Department of Astronautics, Electrical and Energy Engineering University of Rome “La Sapienza”, Rome 00184, Italy (phone: +39-0644-589771) email: augusto.nascetti@uniroma1.it).

round profile [10].

Different Research groups have developed round microfluidic channel by using different approach. Futai et al. [11] used a backside diffused light photolithography strategy combined with small aperture sizes on their photo mask. They produce 200 μm wide semi round channels. Abbas et al. [12] used a positive photoresist (AZ9260, Hoechst) combined to a plasma polymerization technique of tetramethyldisiloxane (TMDS) to create 25 μm wide straight round channels. Song et al. [13] used metal wire moulding technique to form 300 μm wide and perfectly round channels.

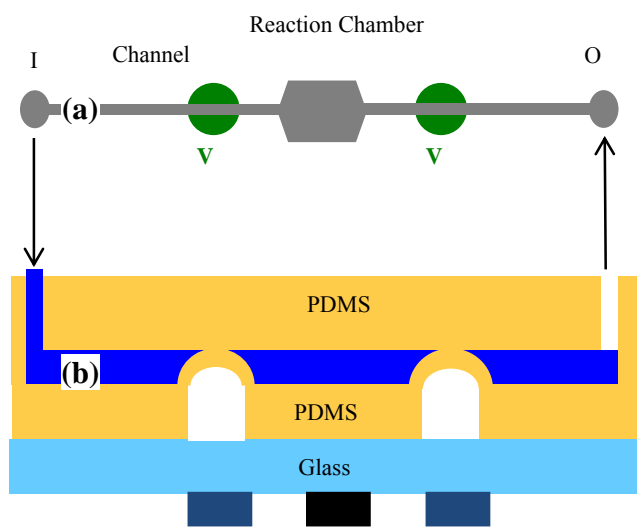


Fig. 2 (a) Top view of microfluidic channel with inlet (I), outlet (O), reaction chamber and membrane type microvalves (V) (b) Cross sectional view of Microfluidic channel with membrane type microvalves

Lee et al. [14] used the viscous property of liquid PDMS before curing to achieve a coating by stacking three layers of PDMS. Keeping the interlayer in the liquid state and the upper layer with a 300 μm wide squared channel, a meniscus was formed and gave rise to a semi rounded channel. Abdelgawad et al. [15] used straight-squared channels of PDMS filled with liquid PDMS and they injected an air stream to produce 100 μm wide round channels with a maximum length of 16 cm. Magalie. De Ville et al. [16] used the method which is based on the optimization of the wetting parameters to produce hydrophobic/hydrophilic steps for the channel pattern definition together with the use of alginate sodium salt aqueous solution. From these results it is obvious that the preparation of PDMS round channels is a real challenge.

The reported methods require sophisticated equipment and several steps. In this paper we are presenting a very simple method to make round shape microfluidic channel. This approach is based on reflow of thick film photoresist. Photoresist reflow is a unique method to generate a hemispheric cross-section using heating effect of photoresist. This approach is very useful for 3-D microstructure fabrication. The channels fabricated by this method have variable height related to spin coating parameters used to

deposit the photoresist, like spin speed and spinning time. In particular in the Fig. 3, the thickness of AZ® 40 XT is reported as function of spin speed and spinning time.

In photoresist reflow the temperature of photoresist is higher than its glass transition temperature. Under reflow condition photoresist become soften and change its shape.

Some photoresists show cross-linking phenomena while heating at temperature, which is higher than the glass transition temperature of photoresist. The resists which show cross link phenomena, they become dense and do not change its shape. However some resists do not show cross-link and they start melting and change its shape. Most negative photoresists cross-link [17] and are not useful for reflow. But all common positive resists do not cross-link and starts soften at approx. 100-130°C [18]. All common positive resists show reflow phenomena and are useful for making round shape profile. The shape of PDMS microchannel depends on the structure of mold made by photoresist. Therefore it is possible to make round channel only by making round shape mold.

II. EXPERIMENTAL PROCEDURE

There are series of various AZ materials which show reflow at certain temperature and time. The choice of resist to make a mold for channel depends on the required channel thickness some values reported in Table I [18].

TABLE I
 LIST OF AZ PHOTORESIST WITH THICKNESS ACHIEVED

Positive photoresist	Thickness achieved
AZ® 1500 series	1-4 μm
AZ® 4533	3-5 μm
AZ® 4562 or 9260	5-30 μm
AZ® 40 XT	> 30 μm

To make channel for microfluidic application generally we need thick layer so we have chosen AZ® 40 XT for making the mold for channel. AZ® 40 XT is a thick positive chemically amplified photoresist with superior performance for wet and dry processes. It is capable to give thickness of photoresist around 60 μm in a single coat and thickness up to 100 μm is possible via multiple coating. Experiment was start using glass substrate coated with chromium (Cr) for mold making. Chromium coated substrate shows a good adhesion between the substrate and device [19], [20].

AZ® 40 XT has been spread on the glass substrate coated with chromium. By varying the spin speed and spinning time thickness of the photoresist can be changed. Values of thickness achieved have been checked by profile meter, some typical values have been reported in Fig. 3.

To achieve thickness of photoresist around 50 μm spinning is done at 1500 rpm (round per minute) for 20 seconds. Softback was at 127°C for 7 minutes. Lithography was performed using hard mask made in Cr. Mask has channel shape of dimension around 500 μm wide and 3cm long, as shown in Fig. 1 (printed in opposite way channel shape painted black other part is transparent for positive photoresist).

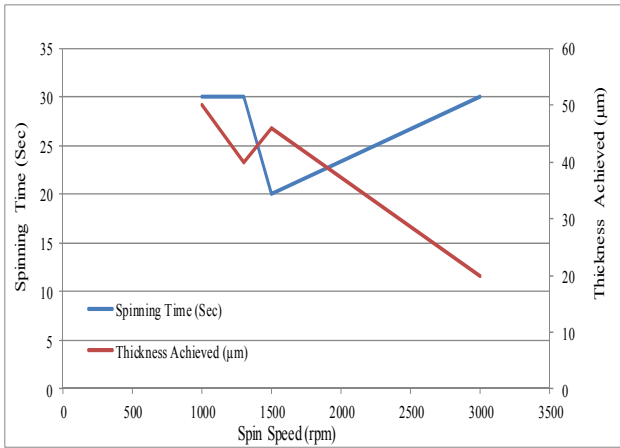


Fig. 3 Variation of thickness of AZ® 40 XT photoresist with spin speed and spinning time

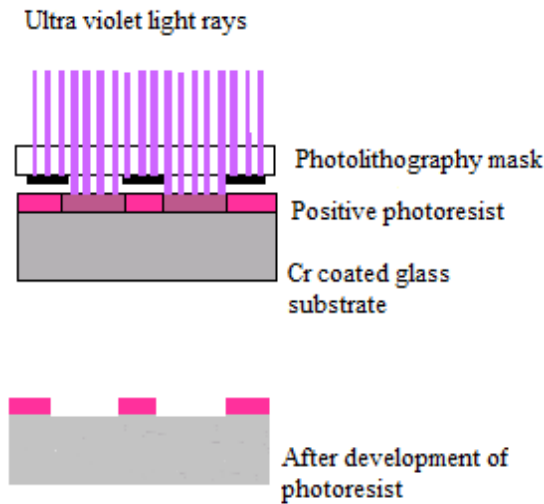


Fig. 4 Photolithography process for positive photoresist

Energy of exposure was 800 mJ/cm^2 . Post exposure bake was at 105°C for 90 seconds. Finally we put the sample into 726 MIF developer solutions for 4 minutes at room temperature. Lithography process for positive photoresist is shown in Fig. 4.

For making the reflow of photoresist, it is necessary to heat the photoresist for certain period of time at a fixed temperature higher than the glass transition temperature of photoresist. The whole process of reflow is shown in Fig. 5.

Initially, when the sample is at room temperature the shape of mold was rectangular, Fig. 5 (a). When the sample is heated at 130°C for 1 minute, reflow began and photoresist become soften at this temperature. The shape of mold starts to change as shown in Fig. 5 (b). Corners of mold start to attend round profile but still it is not completely round. When the temperature is reached at 130°C for 2 minutes mold changed its shape completely. The rectangular shape of mold changed completely into round shape as shown in Fig. 5 (c). In this way we achieved round shape of mold.

To make the round shape PDMS microchannel, silicone and curing agent were mixed in the ratio of 10:1. This mixture was put under vacuum for 30 minutes to remove air bubbles. Thereafter the whole mixture was poured onto the mold, which was fixed in steel holder and baked for 1 hour at 85°C , as shown in Fig. 5 (d).

Finally, the device is cut from the mold with the help of knife and the cross-sectional view has been seen under optical microscope Fig 5 (e). We have pour the mixture of PDMS onto the mold made in AZ® 40 XT without reflow and with reflow to see the change in the shape of microchannel in all conditions.

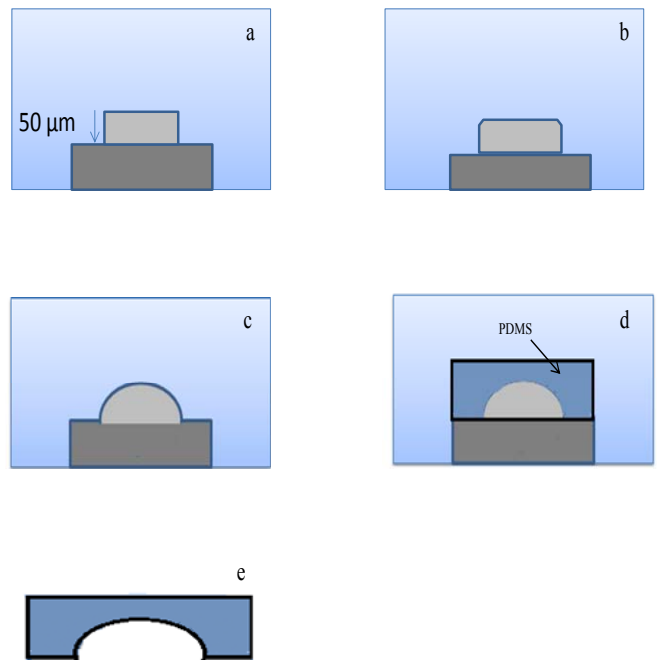


Fig. 5 (a) Structure of mold without heating (b) Structure of mold when photoresist start to change the shape because of reflow at 130°C for 1minute (c) Round shape of mold because of reflow at 130°C for 2 minutes (d) PDMS pouring onto the mold (e) Shape of microfluidic channel after pilling

III. RESULTS AND DISCUSSION

The cross sectional views of channel have been seen under microscope, which clearly show how photoresist change its shape while reflow at certain temperature and time. Fig. 6 (a) is the result of top view of channel. Fig. 6 (b) is the cross sectional view of channel without reflow shows a perfectly rectangular shape. When the mold is heated at 130°C for 1 minute, the cross sectional view of PDMS channel shows that reflow start and the channel start bending from corner but it is not perfectly round Fig. 6 (c). When the mold was at 130°C for 2 minutes PDMS microchannel shows completely round and sharp profile as shown in Fig. 6 (d). This round profile was desirable which shows the successful experiment.

By this approach, we made a round channel of height around $50 \mu\text{m}$. With this method it is possible to obtain the channel of different height simply by varying spin speed and spinning time of photoresist.

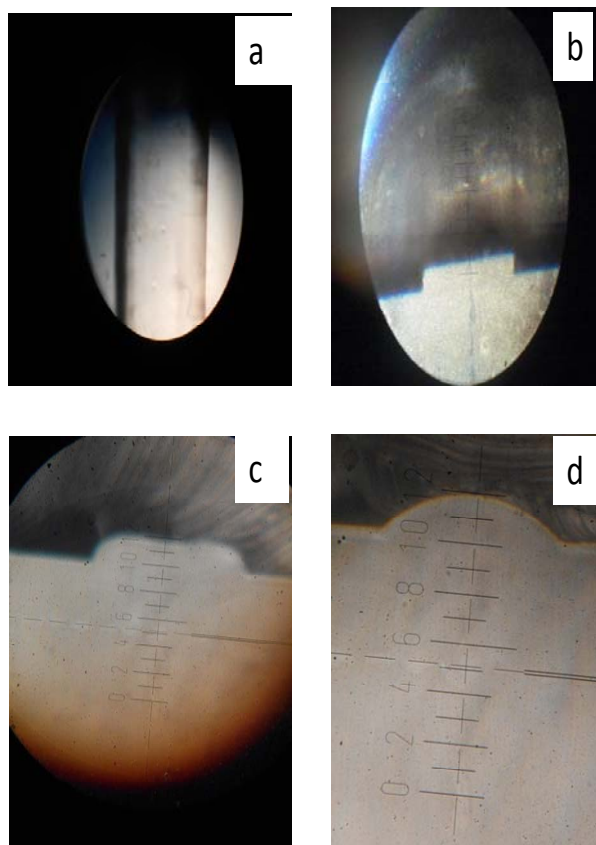


Fig. 6 (a) Top view of PDMS microfluidic channel (b) Rectangular shape of PDMS microfluidic channel without reflow (c) Rounded corner profile of PDMS microfluidic channel, reflow at 130 °C for 1 minute (d) Completely rounded profile of PDMS microfluidic channel, reflow at 130 °C for 2 minutes

IV. CONCLUSION

In this paper, we have presented the procedure to optimize the time and temperature parameters for reflow of AZ® 40 XT positive photoresist. We have developed microfluidic channel of height around 50 μm . We have also optimized the spin coating parameters in order to get channel of different heights. We believe that the technique reported in this work is very useful for many microfluidic applications.

ACKNOWLEDGMENT

Authors are highly thankful to Center for Life Nano Science, "Istituto Italiano di Tecnologia" for providing the financial support for this work.

REFERENCES

- [1] G. De Cesare, M. Gavesi, F. Palma, B. Riccò. A novel a-Si: H mechanical stress sensor. *Thin solid films* (2003) 427(1), 191-195.
- [2] D. Caputo, G. de Cesare, A. Nascetti, M. Tucci; "Detailed study of amorphous silicon ultra-violet sensor with chromium silicide window layer", *IEEE Trans of Electron Device*; (2008). vol. 55 n. 1, 452-456,
- [3] T. Kamei, N.M. Toriello, E.T. Lagally, R.G. Blazej, J.R. Scherer, R.A. Street, and R.A. Mathies. "Microfluidic Genetic Analysis with an Integrated a-Si:H Detector", *Journal of Biomedical Microdevices* 7(2), (2005), 147-152
- [4] J. H. Schaefer, K. Seibel, M. Walder, L. Schoeler, T. Pletzer, M. Waidelich, H. Ihmels, D. Ehrhardt, M. Boehm, "Monolithic integrated

- optical detection for microfluidic systems using thin film photodiodes based on amorphous silicon", *Proc. 18th IEEE Int. Conf. MEMS Miami, FL*, 2005, p. 758
- [5] Costantini F., Nascetti A., Scipinotti R., Domenici F., Sennato S., Gazza L., Bordi F., Pogna N., Manetti C., Caputo D., de Cesare G; "On-chip detection of multiple serum antibodies against epitopes of celiac disease by an array of amorphous silicon sensors", *RSC Advances*, (2014) Vol. 4, n. 4, pp. 2073-2080
- [6] D. Caputo, G. de Cesare, A. Nascetti, R. Negri; "Spectral tuned amorphous silicon p-i-n for DNA detection", *Journal of Non Crystalline Solids*; (2006) vol 352, p.2004
- [7] Zhi Qiang Niu, Wen Yuan Chen, Shi Yi Shao, Xiao Yu Jia and Wei Ping Zhang "DNA amplification on a PDMS-glass hybrid microchip", *J. Micromech. Microeng.* (2006)16:425-433.
- [8] D. Caputo, G. De Cesare, A. De Pastina, A. Nascetti, P. Romano, "Thermally actuated microfluidic system for polymerase chain reaction Applications", *Sensors and Microsystems, Lecture Notes in Electrical Engineering* (2014) 268: 23-27, October 25, 2013.
- [9] Satsanarukkit, P.; van Zyl, J.J.; Hoffmann, M.R., "Novel Parylene fabrication technologies for microfluidic systems with reduced mass, volume, and power consumption," *Solid-State Sensors, Actuators and Microsystems (Transducers & Eurosensors XXVII)*, 2013 *Transducers & Eurosensors XXVII*, pp.155-158, 16-20 June 2013.
- [10] Ryan J. Lemmens, Dennis Desheng Meng "A comparative study on bubble-driven micropumping in microchannels with square and circular cross sections", *Sensors and Actuators A* 169 (2011) 164- 170.
- [11] Futai N, Gu W, Takayama S "Rapid prototyping of microstructures with bell-shaped cross-sections and its application to deformation-based microfluidic valves", *Adv Funct Mater* (2004) 16:1320-1323.
- [12] Abbas A, Supiot P, Mille V, Guillochon D, Bocquet B "Capillary microchannel fabrication using plasma polymerized TMDS for fluidic MEMS technology", *J Micromech Microeng* (2009) 19:045022.
- [13] Song SH, Lee CK, Kim TJ, Shin IC, Jun SC, Jung HI " A rapid and simple fabrication method for 3-dimensional circular microfluidic channel using metal wire removal process" , *Microfluid Nanofluid* (2010) 9:533-540
- [14] Lee K, Kim C, Shin KS, Lee J, Ju BK, Kim TS, Lee SK, Kang JY "Fabrication of round channels using the surface tension of PDMS and its application to a 3D serpentine mixer", *J Micromech Microeng* (2007) 17:1533-1541
- [15] Abdelgawad M, Wu C, Chien WY, Geddie WR, Jewett MAS, Sun Y "A fast and simple method to fabricate circular microchannels in polydimethylsiloxane (PDMS)", *Lab Chip*(2010) 11:545-551.
- [16] Magalie De Ville, Philippe Coquet, Philippe Brunet and Rabah Boukherrou "Simple and low-cost fabrication of PDMS microfluidic round channels by surface-wetting parameters optimization", *Microfluidic Nanofluidic* (2011) pp. 1-9.
- [17] H.-H. Liu, W.-T. Chen, F.-T. Wu, Characterization of Negative tone photoresist based on acid catalyzed dehydration cross linking of novolak having pendant carboxyl group, *J. Polym. Res.* 9 (2002) 251-256.
- [18] http://www.microchemicals.com/technical_information/reflow_photoreist.pdf.
- [19] P. H Holloway and C.C Nelson. In situ formation of diffusion barriers in thin film metallization system. *Thin Solid Films*, 35(1): L 13-6, 1976.
- [20] Milton Ohring. *The Materials Science of Thin Films*, volume 1. Academic Press Limited, San Diego, United Kingdom edition 1992.

Andleeb Zahra received Master of Technology (M. Tech) degree in Solid State Materials from Indian Institute of Technology (IIT) Delhi, in 2010 India. Currently she is pursuing PhD from Center for Life Nano Science at Sapienza, Istituto Italiano di Tecnologia, Italy. Her present field of research includes microfluidic devices for lab on chip application, amorphous silicon technology and its application in thin film devices. Other fields of interest are Thermoelectric devices, nanowires.

Giampiero de Cesare received the degree in electronic engineering from the University of Rome "La Sapienza", in 1982. In 1985, he joined the Department of Electronic Engineering, University of Rome "La Sapienza" Italy, where currently he is an Associate Professor. He teaches "Electronics" and "Technologies and Processes for the Micro-Electronic".

His first research activity was on non-destructive testing of semiconductors by using Surface Acoustic Waves on piezoelectric crystals. Since 1988 his main interests are on amorphous silicon technology and its application in thin film devices for imaging detection and photovoltaic systems. He is author of

more than 150 international papers on journals and congress proceeding. He also holds six patents on thin film detectors, and is currently involved in several projects for the development of innovative electronic systems for molecular analysis application.

Domenico Caputo graduated in Electronic Engineering from the University of Rome "La Sapienza" in 1988. He is now Associate Professor with tenure at the Department of Information Engineering, Electronics and Telecommunications at the University of Rome "La Sapienza". He is coauthor of more than 120 papers on international journals and proceedings of international conferences.

His research activities involved characterization and modeling of amorphous silicon devices, development of characterization techniques for determination of defects in amorphous silicon materials and devices, investigation of the reliability of silicon dioxide for application in nonvolatile memories, characterization of high-k dielectric films for ULSI applications. The present research interest is mainly focused on the development of thin film photodetector and systems for biomolecular detection.

Augusto Nascetti graduated in electronic engineering at the University of Rome "La Sapienza", Italy, where he received the Ph.D. degree in electronic engineering in 2000. During his doctorate, he worked on Thin Film Transistors at the Interface Physics Department of the Utrecht University, Netherlands. In 2000, he joined the Imaging Systems group at Philips Research Laboratories in Aachen, Germany. There he worked on X-ray detectors for medical imaging. In June 2004 he joined the department of Electronic Engineering at the University of Rome "La Sapienza", Rome, Italy as Adjunct Professor with the support of the Researchers Mobility Fellowship ("Brain Drain").

Currently he is an Associate professor at the University of Rome "La Sapienza". His present research is focused on electronic systems for radiation detectors and lab-on-chip-systems with integrated amorphous silicon sensors.