DESIGN AND FABRICATION OF THE MICROCHANNELS FOR MICROFLUIDICS APPLICATIONS

Monica NĂDĂŞAN[1], Adrian MANEA[2]

În acest articol prezentăm o nouă metodă bazată pe tehnici litografice în vederea fabricării microcanalelor pentru aplicaţii în microfluidică. Orginalitatea lucrării constă în proiectarea şi fabricarea unică a microcanalelor recomandată pentru o scară largă de aplicaţii în microfluidică, cum ar fi alinierea cristalelor semiconductoare coloidale. Comportarea fluidelor la scală micrometrică este diferită faţă de compartarea macrometrică din cauza tensiunii la suprafaţă, a energiei disipate, şi datorită rezistenţei care începe să domine sistemul. La scală mică (diametrul canalelor circa 10μm) trebuie să luăm în considerare numărul Reynolds, care este foarte scăzut, adică lipseşte fenomenul de turbulenţă, şi rămâne numai cel laminar. Microcanalele au fost fabricate pe plachete de $SiSiO_2$ prin corodare chimică, litografie optică şi corodare cu ioni reactivi. Am folosit profilometrul pentru a măsura adâncimea microcanalelor, microscopul cu transmisie de electroni pentru captarea imaginilor solutiei coloidale cristaline, apoi eşantioanele au fost caracterizate cu microscopul cu scanare de electroni pentru a observa aliniamentul lateral şi vertical al nanocristalelor.

In this work a new method relying on the lithographic techniques is presented for the fabrication of the microchannels. The originality aspects of the work come from the unique design of the microchannels able to handle a large range of the microfluidics applications, such as the alignment of the colloidal semiconductor nanorods. The behaviour of fluids at the microscale is different from 'macrofluid' behaviour because the surface tension, energy dissipation, and fluidic rezistance start to dominate the system. At small scales (channel diameters of around 10μm) we have to take into account the Reynolds number, which is extremly low, i.e. the turbulent flow lacks, and flow will remain laminar. The microchannels were fabricated on $SiSiO_2$ wafer by wet chemical etching, optical lithography, and reactive ion etching (RIE). The profilometer is used to measure the microchannel's depth, by means of transmission electron microscopy (TEM) we have imaged the nanorods solution, and the samples were characterized by scanning electron microscopy (SEM) in order to observe the lateral and vertical alignment of the nanocrystals.

Keywords: microfluidics, nanotechnology, optical lithography, etching, nanorods

[1] Eng., Dept. of Electronics Technology and Reliabilty, University POLITEHNICA of Bucharest, Romania, nadasan1969@yahoo.com
[2] Prof., Dept. of Electronics Technology and Reliabilty, University POLITEHNICA of Bucharest, Romania

1. Introduction

Microfluidics deals with the behavior, precise control and manipulation of fluids that are geometrically constrained to a small, typically sub-millimeter, scale. Usually, micro means one of the following features: small volumes (nanoliter), small size, low energy consumption, effects of the micro domain. Is is a multidisciplinarity field intersecting engineering, physics, chemistry, microtechnology and biotechnology, with practical applications to the design of systems in which such small volumes of fluids will be used, [1]-[6]. Microfluidics has emerged in the beginning of the 1980s and is used in the development of inkjet printheads, lab-on-a-chip technology, DNA chips, micropropulsion, and microthermal technologies.

In this paper we propose a new method for the fabrication of the microchannels for microfluidics applications relying on the lithographic techniques and imaging the colloidal nanocrystals solution.

Lithography, in the context of microfluidics applications, is a highly specialized printing process used to put detailed patterns onto silicon wafers. An image containing the desired pattern is projected onto the wafer, which is coated by a thin layer of photosensitive material called "resist". The bright parts of the image pattern cause chemical reactions which cause the resist material to become soluble, and thus dissolve away in a developer liquid, whereas the dark portions of the image remain insoluble. After development, the resist forms a stenciled pattern across the wafer surface which accurately matches the desired pattern. Finally, the pattern is permanently transferred into the wafer surface, for example by a chemical etchant which etches everywhere that is not protected by resist.

Recently, it was reported local alignment of nematic liquid crystal through the fabrication of local micrograting structures by curing an UV material via a two-photon excitation laser-lihography process [7]. Also a new method of fabricating nanogaps using a combination of self-assembled molecular and electron beam lithographic techniques was developed and tested [8]. The method enables us to control the gap size with 2 nm accuracy and designate the positions where the nanogaps should be formed with high-resolution patterning using electron beam lithography.

2. Fabrication of the microchannels by optical lithography

The first step in the fabrication process consists in the design of the desired mask. By means of Raith 150 software we have designed the microchannels mask with following dimensions: a) the reservoirs size 1000x1000μm; channels size 20x4800μm; distance between channels 20μm (fig. 1).

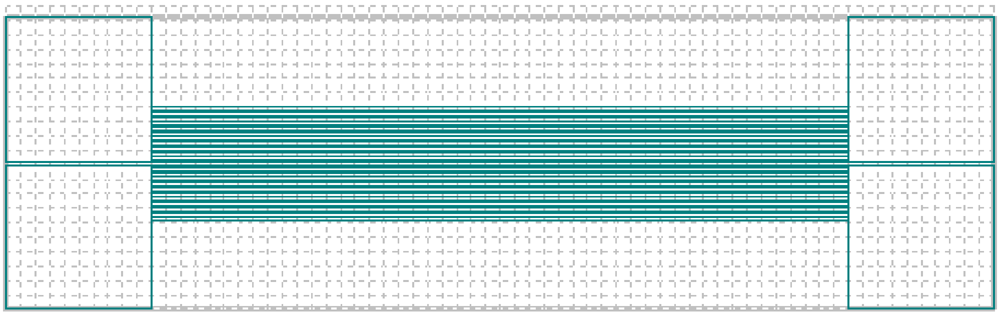

Fig. 1. The microchannels mask

We have fabricated the microchannel by standard procedure of the optical lithography. The $SiSiO_2$ (SiO2 thickness 5µm.) wafer was cleaned primarily by acetone and isopropanol to remove the contaminations. The wafer was then covered with primer and photorezist (AZ 5214E) by spin coating in order to form a thick layer. The spin coating was imposed at 4000rpm, 40sec. and has produced a layer between 1.2 - 1.3 µm thickness. The photorezist-coated wafer was then baked 1min. at 120^0C to evaporate excess solvent. After baking, the photorezist is exposed 3sec. to the designed microchannel mask at 14mW UV light (transfer the pattern from the mask to photorezist). An another bake process (2min. at 112^0C) is performed before flood exposure so that the photorezist becomes harder and the layer is more durable for the wet chemical etching or RIE etching. As developer we have used AZ726 MIF, and stoped the developing process by immersion the wafer in deionized water. The such obtained microchannels depth is around 1.22µm (see fig. 2)

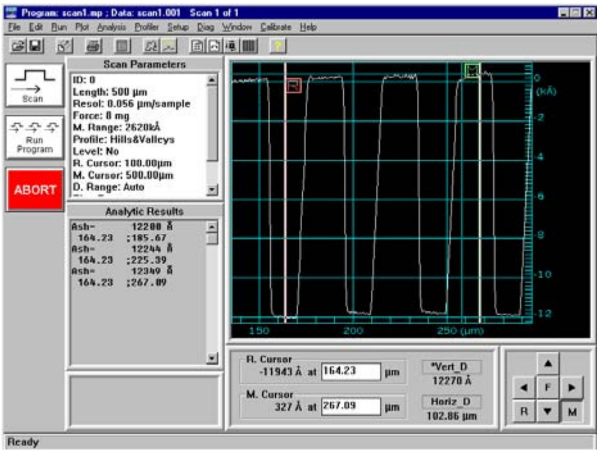

Fig. 2. Sample microchannel 5 after optical lithography

3. Wet chemical etching

For SiO_2 etching we have prepared the solution with ammonium fluoride NH_4F (12g) in water H_2O (17ml.). When the salt was melt we put hydrofluoric acid 38% HF (3ml.). The sample was immersed in this solution different time (5 min., 15min., 60 min. respectively) and then the etching process is stoped by immersing the sample in deionized water. We can see that the longer time is the wet etching process the microchannels profile is changing from the rectangular shape in the sinusoidal one (fig. 3) due to the isotropic process imposed by HF. For microfluidics application this cross-section not exactly rectangular cannot be neglected because it adds extra impedance to the flow due to the small dimensions used. This drawback can be fixed by means of reactive ion etching (RIE), described in the next section.

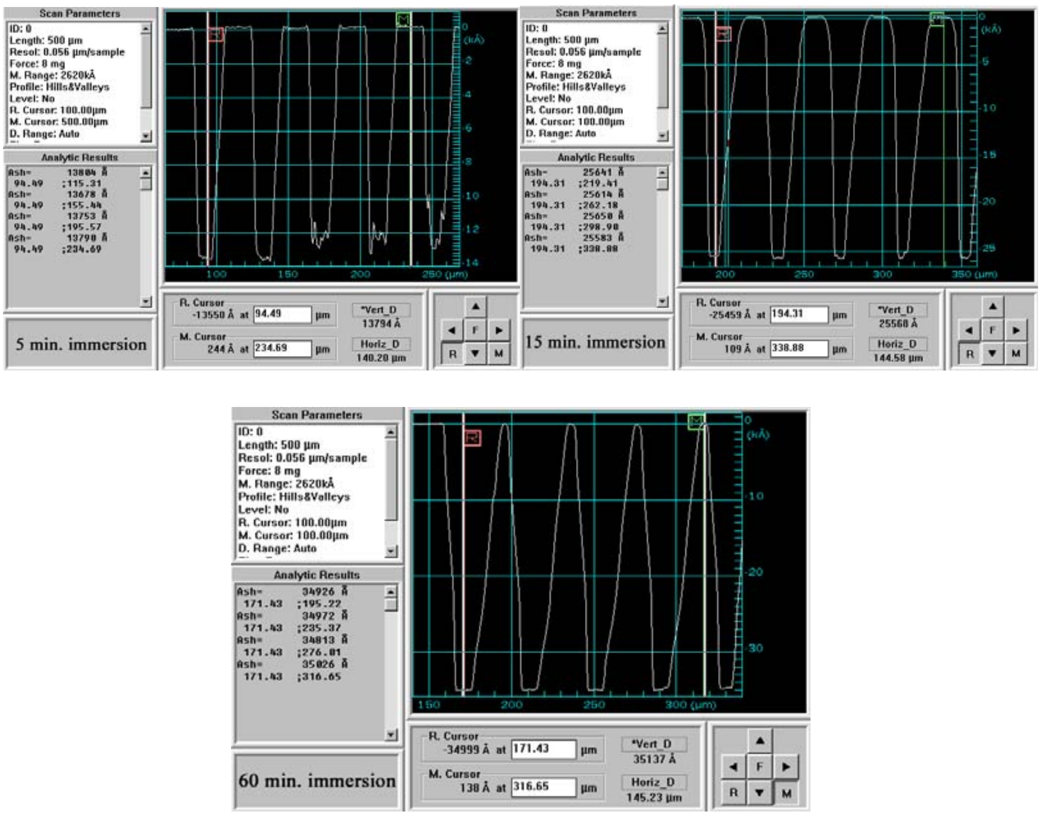

Fig. 3. Sample microchannel 5 after wet etching a) 5min. immersion the microchannel depth is around 1.3µm; b) 15min. immersion the microchannel depth is 2.1µm; c) 60min. immersion the microchannel depth became 3.5µm

4. Reactive ion etching (RIE)

In contrast with the typically isotropic profiles of wet etching, the RIE can produce very anisotropic etch features, due to the mostly vertical delivery of reactive ions. Etch conditions in RIE system (RIE IONVAC) depend strongly on the many process parameters, such as pressure, gas flow and RF (radio frequency) power. We performed the RIE process by mixing tetrafluoromethane CF4 (20sccm) and O2 (2sccm) at 200W, 20mtorr for 10min. In this way the surface quality of the etched microchannels is very good and will not affect the flow behavior, as it still will be laminar flow. In figure 4 is shown the sample after optical lithography process and the obtained profile after RIE process.

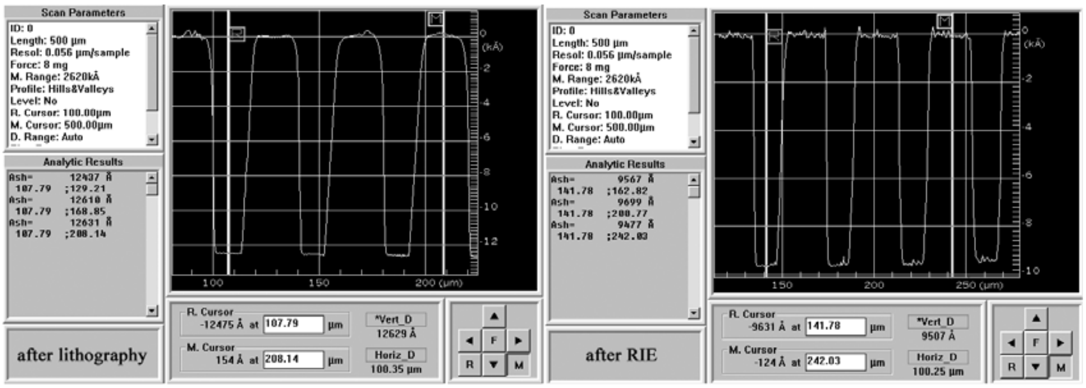

Fig. 4. Sample microchannel 3 fabricated by RIE

For our applications neither this method is too convenient due to the channels' depth not very high (about 1.3 microm. before photorezist removal), therefore we tried a **deep reactive ion etching** (DRIE) with STS Multiplex ICP AOE system. This machine consisting of multiplex ICP AOE (Advanced Oxide Etch) can be setup to process wafers up to 200 mm, but currently setup is 100mm wafers size. Against the above described RIE process, in DRIE we added a gas flux of octafluorocyclobutane C4F8 (20sccm), set the pressure at 4mtorr and reduced the etch time at 6min. A drawback of this method is that it should be used with a metal mask layer. The metal layer (Cr 100nm) was evaporated before optical lithography process. By means of this method the microchannels' depth is around 4µm (fig.5). After lithographic process we performed an evaporation of the 30nm Au layer and functionalization it with 1,6 hexane dithiol in order to facilitate the covalent binding of the semiconductor nanocrystals to the solid surface using self-assembled monolayers.

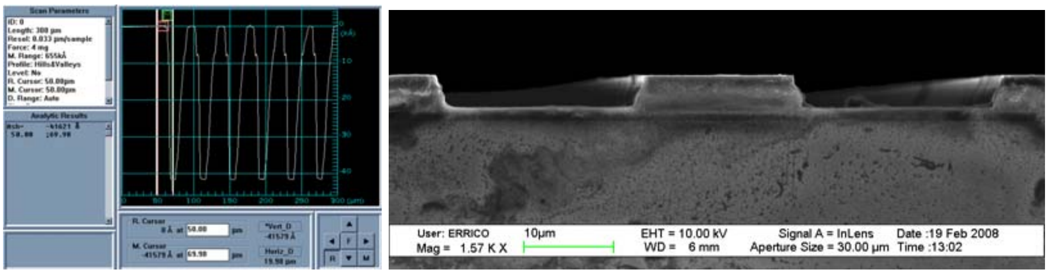

Fig. 5. Sample microchannel 48 fabricated by DRIE: a) profilometer; b) SEM image

5. Applications of the nanorods on the microchannels

Finally, we have tried to image the nanorods on the microchannels. First we used an old dumbbells nanorods solution (see fig.6), synthesized as is reported in [9]. The nanorods are on the top of the channels, because after lithographic process we performed an evaporation of the 30nm Au layer and functionalization it with 1.6 hexane dithiol in order to facilitate the covalent binding of the semiconductor nanocrystals to the solid surface using self-assembled monolayers.

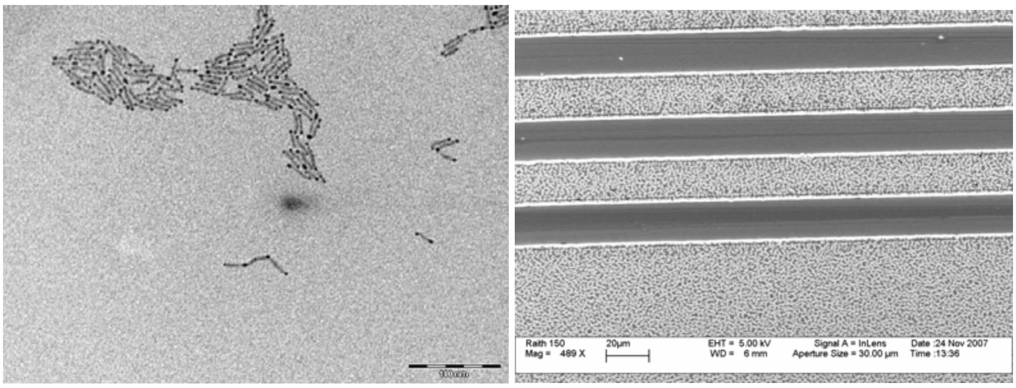

Fig. 6. a) TEM image of the dumbbells nanorods on coper-covered TEM grid; b) SEM image of the dumbbells on the microchannels

When we drop casted the nanorods solution on the microchannels and applied a external electric field we note that not only the temperature has an influence but also the polarization of the electric field, so for positive electric field it was hard to take well-focused TEM images. (fig.7)

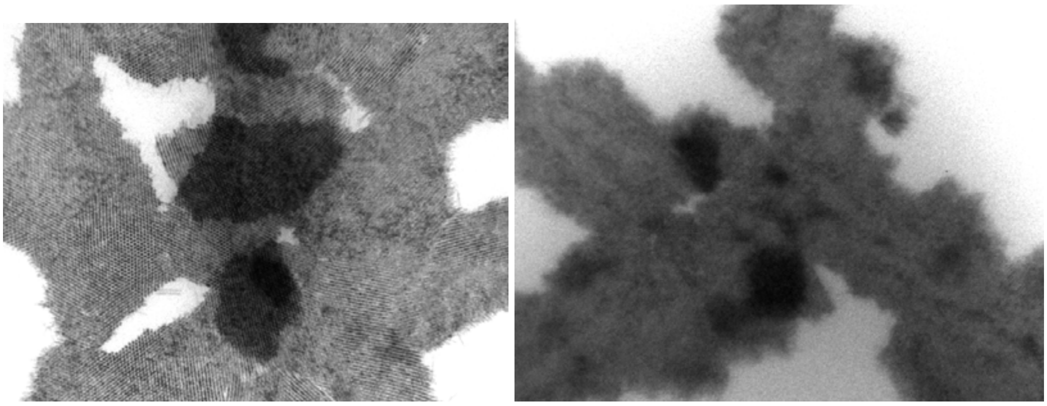

Fig. 7. TEM images of the nanorods in the microchannels. a) a voltage of -32V was applied between two adjacent channels and heated at 40^0C; b) a voltage of +32V was applied between two adjacent channels and heated at 40^0C

We supposed that this is due to the very long and complex lithography process, therefore we simplified the pattern and fabricated the microelectrodes (microstripes) only of Au/SiO2, 50x200 μm., by means of bromograph. (see fig.8)

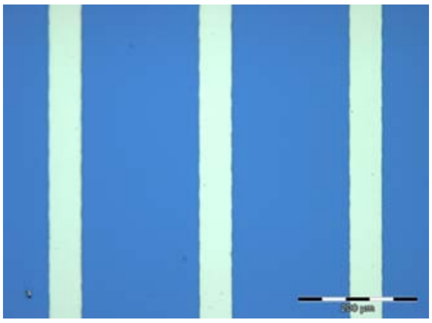

Fig. 8. Microelectrodes of Au/SiO2 (50x200μm) obtained with the bromograph by lithography

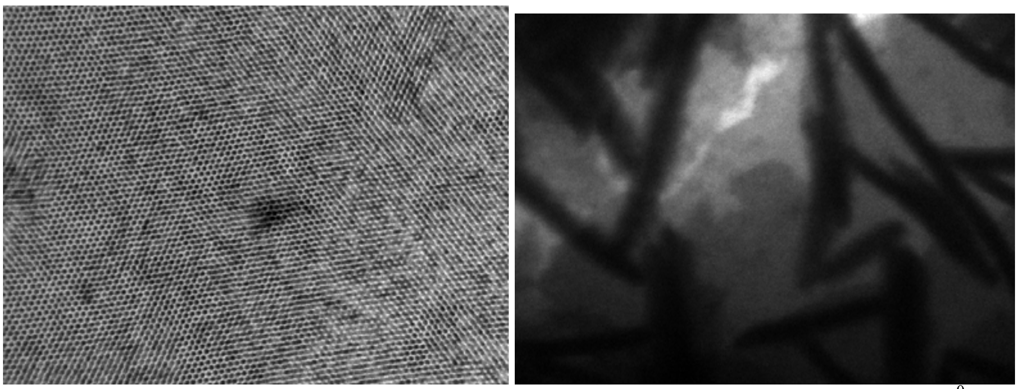

Fig. 9. TEM images of the nanorods on the microstripes. a) the sample was heated at 40^0C and a voltage of -32V was applied between two adjacent electrodes; b) the sample was heated at 40^0C and a voltage of +32V was applied between two adjacent stripes

Also in this case we have observed that applying a positiv voltage makes difficult to take the TEM images, but we can forsee the large area with plaques containing vertical aligned nanorods, like on the microchannels. Therefore we decided to continue the experiments with negative voltage only. We prove the effect of the liquid crystal as well as of the external electric field (fig.10). Applying -32V on the microelectrodes and putting the solution, we noticed the transition from the isotropic phase to the smectic one, and adding a drop of the twisted nematic liquid crystal we observed the vertical alignment on a few hundreds nm.

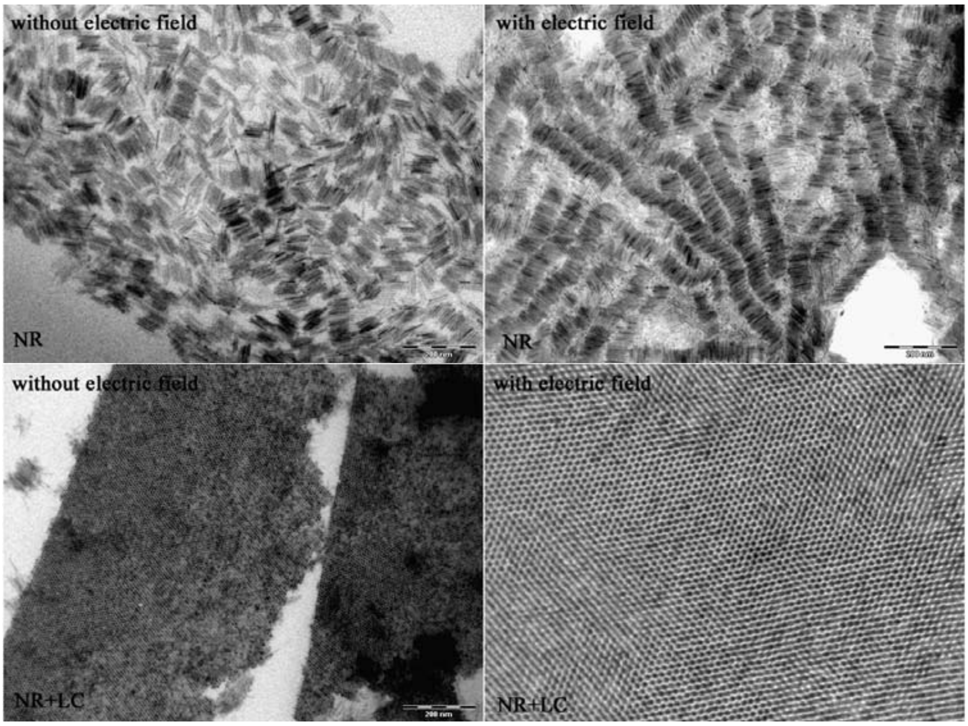

Fig. 10. The effect of the electric field on the nanorods solution with liquid crystals droped on the microelectrodes

6. Polarized light microscopy on nanorods solution evaporated on microelectrodes

In order to search the nanorods solution evaporated on the solid substrate we examine the samples by polarized light microscope, first without analyzer (that means the light is completely nonpolarized), then inserting the the analyzer, and at last rotating it with 15 degrees only. We can see the big difference, because the propagation direction of the polarizer is at right angles to the propagation direction of the analyzer, therefore the field becomes black if no anisotropic sample is on the stage or when viewing an anisotropic substance in an extinction position. The extinction of the light is the darkness that results from rotation of a thin section to a small angle at which plane-polarized light is absorbed by the polarizer. This experiment was done by reflection.

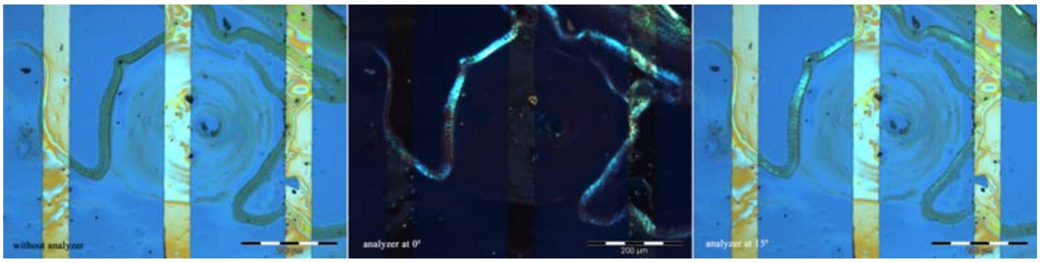

Fig. 11 Polarized light microscopy on the nanorods solution evaporated on the solid surface; imaged by reflection

In order to develop this experiment by transmision and to view the optical luminescence we have fabricated a cell separated by a double sticky tape, and injected 20microl. solution. Again, rotating the polarizer with 15degrees only, we notice the difference between the two images (fig.12).

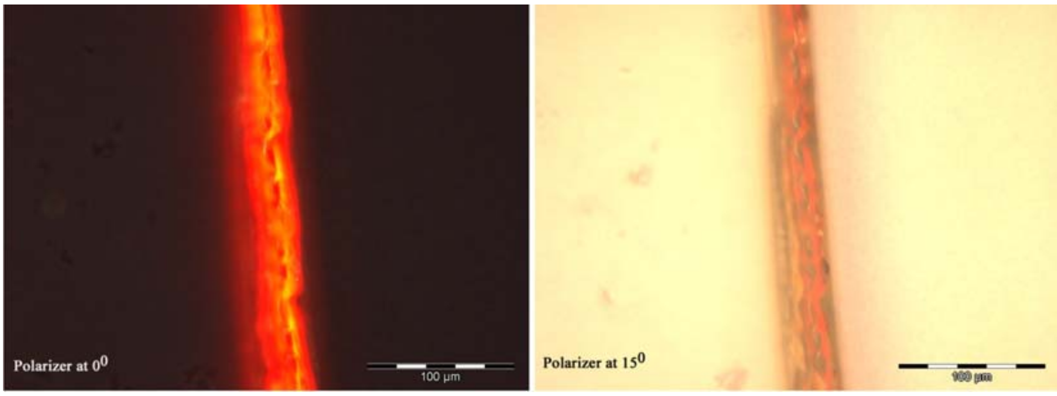

Fig. 12. Polarized light microscopy on the nanorods solution evaporated on the solid surface; imaged by transmission

Table 1

Applications of the nanocrystals

Today	**Tomorrow**
■ Microfluidics–fabrication of the microchannels to perform the separation of different cells, to measure the fluid velocity, pressures, etc. ■ Optoelectronics–to create a LED composed of vertically aligned nanorod grown on GaN substrate, LED monitor, faster memories, etc. ■ Cosmetics industry–tooth paste with nanocrystal, anti-aged implants, solar cream with	■ Energy-new types of batteries, fotovoltaic solar cells ■ Imaging and diagnosis-the quantum dots are metallic nanocrystals which attached by cells and macromolecules are working as fluorescents labels to display anatomic structures or to identify sickness signs. Cell

ZnO or TiO ■ Sport –high performance equipments (stiffness, or, on the contrary, softness rezistence) offered by metallic oxids, bounding with metallic nanoparticles or carbon nanotubes: ski, tennis racket, etc.; ■ Textiles-cloths with antistatic, antispot and antifolding properties	surgery: the nanorobots are able to enter in the cells for repairing or replacing the eventually damaged structures (DNA, mitocondri)

7. Conclusions

We have presented the lithographic techniques for the fabrication of the microchannels, relying on the wet chemical etching and dry etching (optical lithography, RIE, deep RIE). The nanocrystal solution was spread on the microstructures and imaged by means of TEM, SEM and optical microscopy in order to check the lateral and vertical alignment of the nanorods.

The applications of the colloidal nanocrystals are summarized in the table 1.

R E F E R E N C E S

[1] *B. Dindoruk, A. Firoozabadi*, Liquid film flow in a fracture between two porous blocks, Phys. Fluids 6, 3861, 1994

[2] *E. Saatdjian, N. Midoux,* On the solution of Stokes' equations between confocal ellipses, Phys. Fluids 6, 3833, 1994

[3] *S. Reddy, P.R. Schunk. R.T. Bonnecaze*, Dynamics of low capillary number interfaces moving through sharp features, Phys. Fluids 17, 122104, 2005

[4] *M. Fichman, G. Hetsrani,* Viscosity and slip velocity in gas flow in microchannels, Phys.. Fluids 17, 123102, 2005

[5] *A. Goullet, W. Choi*, Large amplitude internal solitary waves in a two-layer system of piecewise linear stratification, Phys. Fluids 20, 096601, 2008

[6] *S. Klongboonjit, C.S. Campbell,* Convection in deep vertically shaken particle beds, Phys. Fluids 20, 103301, 2008

[7] *Chee Heng, Y. Hiroyuki, M. Yusuke, F. Akihiko, O. Masanori,* Local liquid crystal alignment on patterned micrograting structures photofabricated by two-photon excitation direct laser writing, Appl. Phys. Lett 93, 2008

[8] *R. Negishi, T. Hasegawa, K. Terabe, M. Aono, T. Ebihara, H. Tanaka, T. Ogawa,* Fabrication of nanoscale gaps using a combination of self-assembled molecular and electron beam lithographic techniques, Appl. Phys. Lett 88, 223111, 2006

[9] *L. Carbone et al*, 'Synthesis and micrometer-scale assembly of colloidal CdSe/CdS nanorods prepared by a seed growth approach', *Nanoletters*, p. 2942-2950, 2007.