International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 12, December-2016

Micro and Nano Scaled Patternings on SU8 and PVP Polymer Substrates by Direct Laser Writing Lithography

E. Srinivasan [1],
Asst. Professor
Department of Electronics and Communication Engineering
Panimalar Engineering College,
Chennai, Tamil Nadu, India

V. Velmurugan[2]
Professor
Centre of Nanotechnology and Research
VIT University, Vellore, Tamil Nadu, India

***Abstract* - Laser exposed direct writing technique is a methodology of optical lithography effectively established fabrication technology for miniature structuring of surface patterns. In this paper we fabricated micro-scaled and nano-scaled patterns on the substrate implemented by mask-less lithography process, films deposition was carried out by spin coating process, patterns on surface of SU8-epoxy based negative photoresist, and sonicated PVP (polyvinylpyrrolidone) substrates carried out by (532nm, 150mW) diode pumped solid state (DPSS) type continuous wave green laser as main source, laser beam penetrates on SU8 substrate get ablated created a path with good depth and non circled bumps was created in PVP substrate, characterized it by Olympus optical microscope under various magnification levels.**

Index Terms: Surface modifications, direct writing, spin coating, curing, SU8, PVP, lines, dot and bump Patternings, Optical Microscope-(OM)

1. INTRODUCTION:

In past decades there was enormous and sophisticated development in micro and nanotechnology, one of the successive key factors is continuous improvement in lithography techniques are driving a force for innovative patterns formations in present industrial areas. So far lithography focused on micro and nano patterning's has been limited industrial applications [1]. Making micro and nano scale patterns on a polymer substrate is of great importance for both fundamental research and practical purpose, the advantage of polymer based substrate are mechanical flexibility, light weighted, enhanced durability with low cost [2], fine patternings on a polymer based substrate with good resolution can be regularly achieved with some sophisticated state of art facilities. However these facilities are not widely accessible mainly due to their high complexity to implement, these are treated as main problems (high complexity and low accessibility) faced by the conventional micro-nano fabrication techniques may be overcome by using recently developed lithographic approaches such as radiation lithography using optical equipments, patternings are mainly focussed on obtaining a patterns with smaller and lateral dimensions via optical lithography for new fields miniaturized devices emerged [3].

Direct writing technique is already proven to be appropriate for printing patterns of different materials in solid phase metals (aluminium, titanium, and tungsten etc.,) [4]. Also these techniques are implemented by the laser beam focusing through objective lens with and without using mask and make the patterns. Depends on laser power density, material property, Micro and nanometre scaled patterns are realized by focusing laser beam in bulk and semisolid materials [5]. However the main drawback of this technique is a 100 class clean room required for implement the laser direct writing lithography [6].

SU8 and PVP materials are used to fabricate the patterns on it. Polymer based microstructures are popular in industry, SU8 is supplied by (Microchem Inc), chemically amplified, high contrast, epoxy based negative photoresist [7]. For patternings on SU8 is the most commonly used to make the separation system, micro reactors, micro needles, valves, polymeric chain reaction (PCR) chips, cell growth substrate and other MEMS systems [8] it has very high optical transparency above ~400nm, ideally suited for patterning high aspect ratio with near vertical side walls structure in very thick and thin films. Negative tone photo resist is highly and chemically stable and resistant to most acids and other solvents [9-10]. The SU-8 photoresist is most suitable for making the micro and nano patterns fabrications because, having good mechanical properties such as excellent chemical resistance [11], biologically benign material with stability, optical transparency and very high absorption coefficient etc., [12-13]. We can define the micro patterns in SU8 by using different exposure methods [14].

Polyvinylpyrrolidone (PVP) is deposits as substrate also used for the micro and nano patterning's, PVP belongs to the conjugated polymers family, its having good stability and capability to dissolve in deionised water [15] , excellent transparency, fine charge storage capacity and optical properties, it is water-soluble and physiologically inert polyamide polymer [16]. The first patterned PVP is obtained by micromolding in the capillaries techniques [17]. PVP improves the film formation and it can thermally be cross-linked yielding a composite with outstanding thermal stability and high mechanical strength and the low light scattering due to the amorphous structure make it an ideal polymer for composite materials in optical applications [18]. Among the many polymers the polyvinylpyrrolidone has good filmforming exhibits good optical quality (high transmission visible range) for various applications [19], also crossover many commercial and technical applications such as Pharmaceuticals, agricultural chemicals adhesive, beverages, cosmetics, detergents, textiles, lithography, photography, paper manufactures and processing, oil-well drilling, fuel cell batteries[20-21] also in ceramics and fibreglass and etc.,[22].

2. RESEARCH METHODOLOGY AND IMPLEMENTATIONS:

2.1. Process Flow:

Setup has been implemented in different ways of laser exposure, without using mask and templates beam exposed directly to the prepared substrate and design the pattern on the top surface of the polymers, process flow is shown below Fig.1.

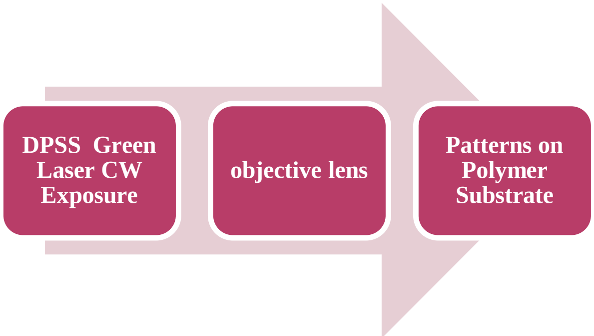


Fig.1 Flow chart for direct patternings

The beam which converged and exposed by focal lens with various objective magnification and Numerical aperture, beam ablates the prepared substrate, creates various patterns in microns.

2.2. Glass cleaning:

Prepared substrate and solutions which are deposited on the surface of the glass slide, to get the fine depositions of the substrate glass cleaning process is mandatory and the required glass slide for the substrate deposition is about 75mm long x 25mm wide, thickness of the glass slide is about 1.45mm. The specified glass slide is sliced into a (1:3) ratio and it implements into a cleaning process into a various solvents cleaning for removing the contaminated particles and oil residues.

2.3. Synthesis of materials:

For cleaning solvents like to concentrate nitric acid (HNO_3) Hydrochloric acid (HCL), acetone, and soapy water and final rinse in the distilled water and dry the slide by blowing the fresh air. After some time use a lint free wipe and cotton swab to gently rub the surface clean of dirt and residues and get the fine and uniformed topographic surface of substrate, 2.5mg of rhodomine dye mixed with 15ml of carbinol to get more absorption which is nearer to wavelength 532nm, from these 4ml of mixed solution mixed with 4gms of PVP, sonicating it up to 15 minutes with temperature about 75ºC to get the slurry as PVP one of the substrate for patternings on the other SU8 supplied MICROCHEM epoxy based negative photo resist, both substrates are deposited on the separate glass slide by spin coating technique, for laser writing, during depositions the well cleaned and without residues glass slide placed on spinning chuck, rotated it up to 4700rpm for 100 seconds, every 10 seconds pour the 3 drops PVP slurry on the glass slide during spinning, and get the uniform coated substrate and similar procedure for depositing the SU8 negative photoresist substrate. DPSS, 532nm continuous wavelength 147mW (~150mW) powered green laser used for substrate ablations and topographic modification.

2.4. Design Implementations:

Laser direct writing technology was implemented by various focusing of beam exposures, horizontally direct exposure and vertically downwards directions. The beams are diverted by using reflecting mirror and prisms (beam diverter)

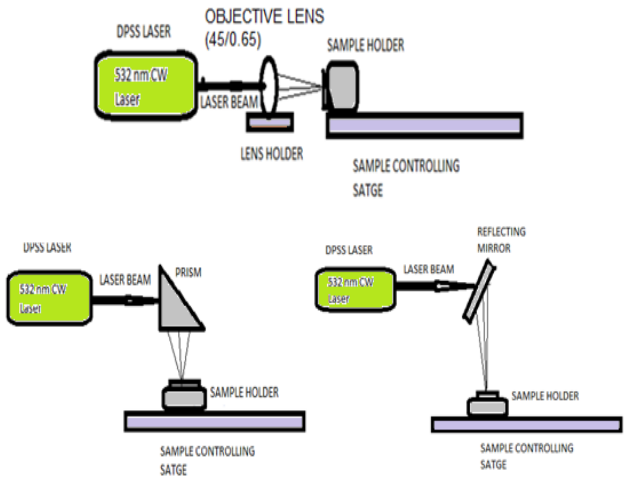


Fig.2. Different phases of experimental setup for laser direct writing techniques

The figure 2 describes the different exposure of the laser beam into the substrate in different exposure timings for getting the threshold level, for fine ablating of the substrate, laser various exposures delivers floating power deliver, due to beam penetrates through by the beam splitters and objectives lens. In-spite of above beam bombard will never disturb the surface, due to power delivered level of the laser through objective lens and diverter become reduced.

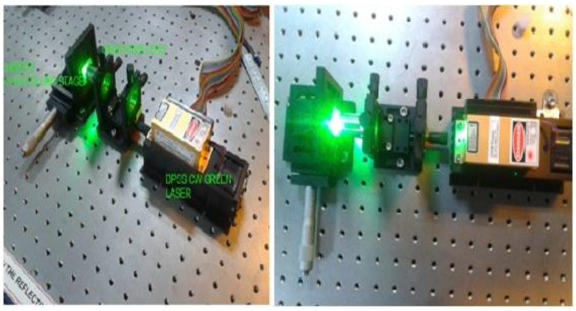


Fig.3. Photocopy of the laser direct writing lithography experimental setup in 100 class clean room

The figure 3 shows the photocopy of the laser direct writing lithography technology, substrate which kept closer to the objective lens, two various lenses are utilized for

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 12, December-2016

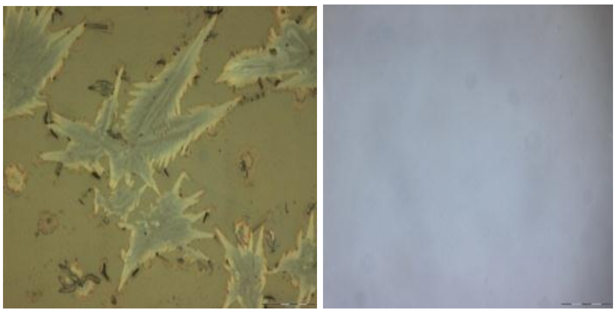
compress and converge the secondary wave propagation of beam, optical objective lenses with various aperture ie.,10/0.25 NA and 45/0.65 NA

The power delivered through by the objective lens 10/0.25 NA is approximately 89.8mW, and through by objective lens 45/065 NA delivers 62.3mW of laser power.

3. RESULTS AND CHARACTERIZATION DISCUSSIONS:

The important aspect of this research we need to find the threshold level i.e. in which dimensions i.e. power, distance, spinning details, and required optical equipments lens, objectives, beam splitters, mirrors and etc

3.1 Patternings on PVP substrate:



(a) (b)

Fig.4. (a)- Glass slide before substrate migrated full of oil residues and rough particles, imaged by optical microscope (100X magnification), (b) after glass cleaning by using various solvents uniform topographic surface of PVP substrate depositions, imaged by OM (50X magnification)

The substrate get expected modification that is said to threshold level for the above the laser power has to kept at the constant level and by continuously varying exposure time periods and find threshold and kept as a reference create a micro-scaled and nano-scaled fine patterns. Fig 4 (a) some of the contaminated dust particles are migrated on the surface of the glass, after glass cleaning, by various solvents and PVP slurry is deposited on the glass slide by using spin coater, rotation of motor 4700rpm and get fine topographic surface of PVP showed in Fig 4.(b).

3.2 UV-VIS absorption spectra

The laser absorption of the substrate not enough for previous PVP substrate so the sensiter rhodomine dye is mixed further with the PVP substrate to get the fine absortion of the laser beam.

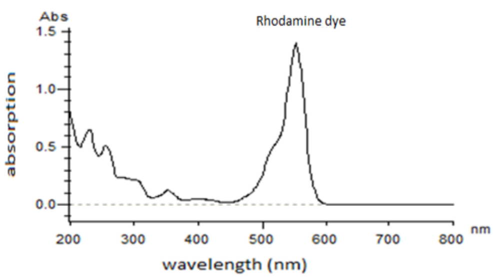
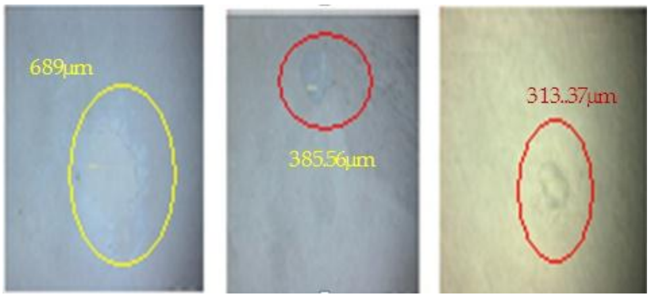


Fig.5 UV-VIS for Rhodomine-B Dye

By mixing of 250ml of DI water and 3mg of rhodamine dye the resulting wavelength is approximately 546nm almost nearer to 532nm DPSS type laser , and absorption range upto1.45, ratio of mixing the carbinol and rhodomine ratio (1:80). The absorption and wavelength is given by the Ultratech UV-Visible Spectroscopy showned in fig 5.



(a) (b) (c)

Fig. 6. PVP substrate with and without sesister rhodomine die,(a) ablated substrate due to laser exposed 10 minutes, (b) mild etching substrate, laser exposed through reflecting mirror for 4 minutes, (c) substrate without dye beam exposed through objective lens (10X/0.25NA) a non uniform circular bump was created. Imaged by optical microscope 5X magnification

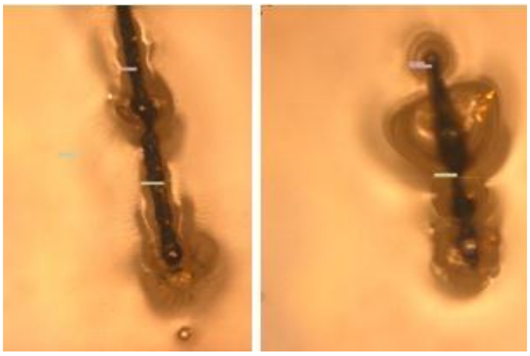
Fig 6.(a) shows that the surface of the PVP substrate get ablated up to the surface of the glass slide by laser direct exposure without using any objective lens and distance between to substrate is 3.4mm. The ablating of substrate is due high power ~147mW beam bombardment take place on the substrate. The size of the ablated region is 689µm time of the laser exposure is 10minutes, next two cases beam exposing distance form source (laser) to lens 7mm, from lens to sample substrate 0.2mm, totally 7.2mm distance beam penetrate through the objective lens to make the surface modifications on the substrate. Fig 6(b) represents the mild etching of the PVP substrate and the exposing method of laser is though the reflecting mirror i.e., the direction of the beam in the downward direction which focused the substrate. The size of the etched surface is about 385.56µm. Fig 6 (c) represents the non uniformed circled bump produced by the laser exposed through the objective lens (10X/0.25NA), and the size is bump is approximately 313.37µm. all substrate modifications are characterized by the optical microscope at 5X magnifications.

3.3 Patternings on SU8

SU8 (negative tone epoxy based photoresist) is a high contrast epoxy based photoresist when it make into faster drying process then more polar solvent systems result in improved coating quality and it increases the process throughput. The power level of the laser is maintained at constant and by varying the time of exposures and find the threshold level. During long time of exposure on top surface of substrate get scattered because the beams which creates effective heat zone at the laser spot on the substrate and by keep on reducing the time of exposures substrate will give the expected ablated level

International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 5 Issue 12, December-2016

3.4 Threshold level on SU8

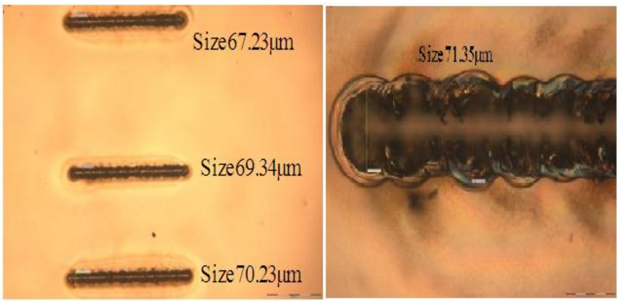


(a) (b)

Fig. 7. Laser ablated SU8 substrate according to threshold level for etching and laser exposing time duration 3 minutes.

At the time of 3 minutes exposure the laser beam which create a tolerable heat at the substrate and it perform the good and fine ablating process. Finally at 3 minutes exposure the laser beam will never damage the substrate. The width size of the ablated region is approximately 201.3µm, in Fig 7 (a), this describing threshold level of the laser direct writing for SU8 substrate ablation for fine patternings. The sample substrate has moved manually in approximated distance to confirm the threshold level again and the width of the pattern approximately 292.9µm, width size of the other pattern is approximately 304.2µm showed in Fig 7 (b).

The drawback of the patternings on SU8 substrate is gluing properties to overcome this, before exposing the laser beam the substrate should kept for the process of curing i.e. the spin coated substrate is kept for UV light exposure to bring the SU-8 from transparent to visible light and removing the gluing properties. The specification of UV light is 500W UV HALLOGEN, which supplied from 250V, 50HZ frequency AC supply. After curing the SU-8 the substrate should placed very nearer to the objective lens (45/0.65NA) for the ablation process. The sample is hold at sample controlling stage to move sample in X-direction and Y-direction to form a line pattern



(a) (b)

Fig 8. Line pattern on SU8 substrate imaged by optical microscope, (a) pattern in 5X magnification, (b) 50X magnification

The laser exposing time period is 3mins and every 3minutes the manual (X-Y) controlling stage is move up to 15mm in the (x) horizontal direction and make a line. After the stage is moved up to 30mm in Y direction again make line pattern follow the same to get three lines showed in Fig 8(a), The size of the first line about 67.23µm, 69.34µm, and 7.023µm in that single line is further zoomed i.e., imaged at 50X magnification, and get high resolution image of the photoresist pattern and width size ~71.35.

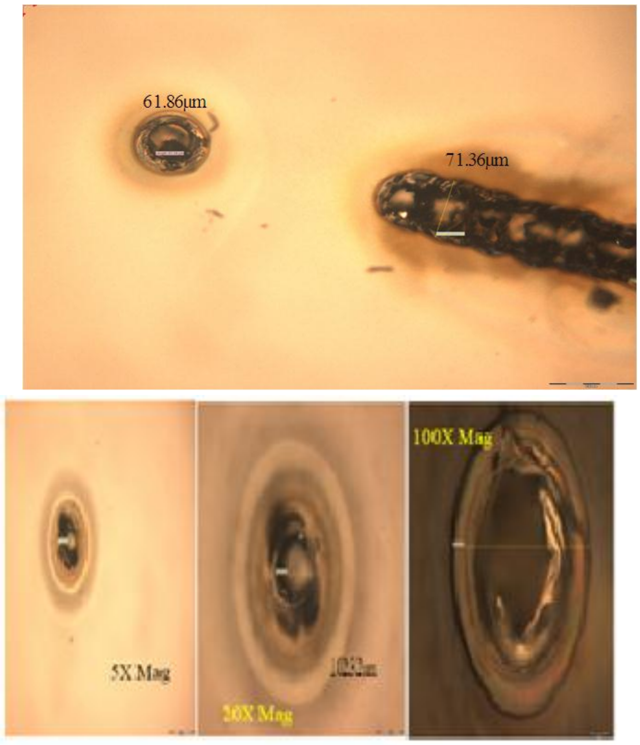


Fig.9 Dot and line pattern on SU8 Substrate, imaged by Olympus optical microscope

The fig.9 Shows the dot and line pattern under the same time of beam exposure and substrate get ablated and the size 61.86µm for dot and 71.36µm for line, characterized by OM under different magnification 5X, 20X and 50X. If we keep on increasing the time of exposure the width size get slantly varied the size variation table is showed below

3.4 Table1: Finding the width size variations from the average size of the SU-8 ablated surface based upon the different time of laser beam exposure.

SIZE VARIATIONS				
TIME OF EXPOSURE	WIDTH IN µm			AVERAGE VALUE
3MIN	7.16	7.29	7.73	7.39333
4MIN	7.85	7.45	7.85	7.71667
5MIN	8.43	8.27	8.27	8.32333
6MIN	8.27	8.43	8.41	8.37
7MIN	8.83	8.83	8.45	8.70333
8MIN	8.83	9.38	9.39	9.2

The Fig 10 shows that the image of direct exposure along with the image dimensions which is uploaded in Gwyddion software version 2.26 and get the three dimensional image with the measurement of height, length and width of the ablated region on the SU-8 substrate. The height of the ablated region is ~310nm and width of the deposited SU-8 substrate is 2.2mm and length is 2.9mm.

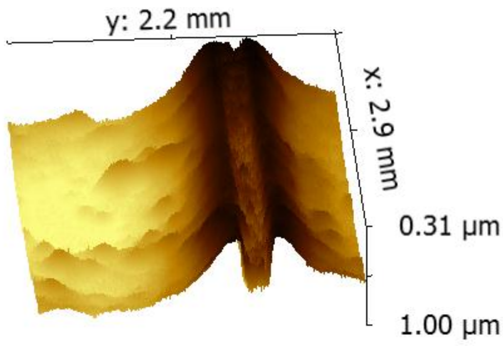


Fig.10 Three dimensional view of line pattern on SU8 substrate imaged by Gwyddion version 2.26

The 3 dimensional image of SU8 substrate after performing the curing process which describes that the substrate get uniform ablation of substrate by the laser beam and make a constant and flexible line path.

4. CONCLUSION

The setup is relatively simple and by using the reflecting mirrors and other optical equipments configurations the patterns and directions can be easily changed. It is advisable to use continuous wave lasers due to high intensities a special care must be taken when selecting the pinholes for optical filtering, during the patternings even substrate surface uniformity is obtained with Rhodomine dye the PVP doesn't restructure at low power density. And at high power density, the PVP starts bulging non-uniformly with minimum size of 313.37µm. Optimization of parameters involved may improve the pattern formation on PVP substrate with uniformed and high resolution patterns. In SU8 Uniform coating is achieved, minimum power needed to ablate 87.6mW powered beam exposed for 3 minutes time duration, optimization of parameters resulted in uniform structure and its size is nearer to the expected level ie., ablated region 61.86µm minimum width is 7.1µm, and height is minimum 310nm is size. Future developing process that the ablated parts of SU-8 sticks on the surface and suitable methods for removing those ablated parts has to be determined. By using mask with high power beam exposure we may get fine patternings.

5. REFERENCES

[1] Michael Theil, Martin Hermatschweiler, "Three-Dimensional Laser Lithography", Optik & Photonik, December 2011, Vol 4, Page 36-39.
[2] Inkyee Park, Seung H, Ko.Heng Pan, "Nanoscale Patterning and Electronics on Flexible Substrate by Direct Nanoimprinting of Metalic Nanoparticles", Advanced Materials, Wiley Inter Science, April 2008, Vol 20, Page 489-496.
[3] Aranzazu Del Campo, Eduard Arzt, "Fabrication Approaches for Generating Complex Micro and Nanoparticles on Polymeric Surfaces", American Chemical Society, February 2008, Vol 108, Page 911-945.
[4] Alexandra Palla-Papavlu, Valentina Dinca, "Laser Induced Forward Transfer for Material Patterning", Romanian Reports in Physics, August 2011, Vol 63, Page 1285-1301.
[5] Z, L.Li, T.Liu, C.Khin, "Direct Patternings in Sub Surface of Stainless Steel using Laser Pulse", Optic Express, July 2010, Vol 18, Page 15990-15997.
[6] Johan E, Ten Elshof, Sajid U.Khan, "Micrometer and Nanometer Scale Parallel Patterning of Ceramic and Organic-Inorganic Hybrid Materials", Journal of European Ceramic Society, Elsevier-Science Direct, January 2010, Vol 30, Page 1555-1577.
[7] P.K.Dey, B. Pramanick, "Microstructuring of SU8-Resist for MEMS and Bio-Applications", International Journal of Smart Sensing and Intelligent Systems, March 2010, Vol 3, Page 118-129.
[8] Akanksha Singh, Gianmario Scotti, "Laser Direct Writing of Thick Hybrid Polymers for Microfluidic Chips", Journal of Micromachines, July 2014, Vol 5, Page 472-485.
[9] Vania.C Pinto, Paulo J.Sousa, Vanessa F. Cardoso, "Optimized SU-8 Processing for Low-Cost Microstructures, Fabrication Without Cleanroom Facilities", Micromachines, September 2014, Vol 5, Page 738-755.
[10] J.Onishi, K.Makable, "Fabrication of Micro Sloping Structures of SU8 by Substrate Penetration Lithography", Microsyst Technol, Springer-Verlag, February 2008, Vol 14, Page 1305-1310.
[11] M.Gersborg.Harsen, L.H.Thandrup", Combined Electron Beam and UV Lithography in SU8", Microelectronic Engineering, Elsevier-Scinece Direct, February 2007, Vol 84, Page 1058-1061.
[12] G.Hong, A.S. Holmes, "SU8 Resist Plasma Etching and its Operation", Microsystem Technologies, Sringer-Verlag 2004, Novemeber 2003, Vol 10, Page 357-359.
[13] Lin Pang, Nataru Nakagwa, "Fabrication of Optical Structures using SU8 Photoresist and Chemically Assisted Ion Beam Etching", Optical Engineering, Vol 42, Page 2912-2917.
[14] James M, Dykes, David K. Poon, "Creation of Embedded Structures in SU8", SPIE for Optics and Photonics, 2007, Vol 6465, Page 64650N-1 to 64650N-10.
[15] Seung yeon Kang, Michael Gerhard Moebius, "Femtosecond Laser Direct Writing of Nanocrystalline Hexagonal Silver Prisms", Applied Physics Letters, Vol 105, October 2014, Page 141114.
[16] Mohammad Ahmed-Fouad Basha, "Magnetic and Optical Studies on Polyvinyl Pyrrolidone Thin Films Doped with Rare Earth Metal Salts", Polymer Journal, July 2010, Vol 42, Page 728-734.
[17] Rubo Xing, YU Xuan, "Undercut Structures Fabricated by Complementary-Structure Micropatternings Techniques for the Passive-Matrix Display of Organic Light-Emitting Diode", American Vaccum Society, January 2008, Vol 26, Page 1-5.
[18] Hussein Hahim, Abdulameer Khalaf, "Optical Properties of Polyvinylpyrrolidone (PVP) Films Doped with Nickel Nitrate ($NiNo_3$)", International Journal of Science and Research (IJSR), October 2014, Vol 3, Issue 10, Page 1869-1870.
[19] Kothapalle Sivaiah, Koramala Naveen Kumar, "Structural and Optical Properties of Li:PVP & Ag:PVP Polymers Films", Materials Science and Applications, November 2011, Vol 2, Page 1688-1696.
[20] Sukanya Bhattacharya, Dharmendar Kumar Sharma, "Plasticization of Poly(Vinylpyrrolidone) Thin Films Under Ambient Humidity: Insight from Single-Molecule Tracer Diffusion Dynamics", Journal of Physical Chemistry, American Chemical Society, June 2013, Vol 117, Page 7771-7782.
[21] Rachel A. Segalman, "Patternings with Copolymer Thin Films", Material Science and Engineering, January 2005, Vol 48, Page 191-226.
[22] Priyanka H Kamble, Barnali S Banerjee, "Ultrasound-Based Treatment Approached for Intrinsic Viscosity Reduction of Polyvinyl Pyrrolidone (PVP)", Journal of Ultrasonics Sonochemistry, 2014, Vol 21, Page 1108-1116.