6.2 Pressure driven Microsyringe

The deposition system developed at Centro Interdipartimentale di Ricerca "E. Piaggio" at the University of Pisa consists of a stainless steel syringe with a 20 micron glass capillary needle (fig.1) [1, 2]. The needle is connected to the syringe barrel and held in place by a small o-ring. A solution of the polymer in a volatile solvent is placed inside the syringe, which has a capacity of about 10 ml.
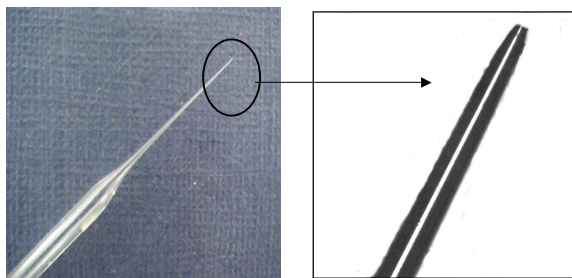

Fig. 1 Profiles of needle tip

The syringe, which has no plunger but is driven by filtered compressed air at a pressure of about 20–200 mmHg, is fitted to the z-axis of a 3-axis stepper-motor micropostioning system with a resolution of 0.1 micron. A planar substrate, generally a glass slide of dimensions 3X3 cm, is fixed on the x and y axes of the micropositioner, and is made to move under the syringe during deposition. In preliminary experiments, it was ascertained that the most reproducible technique for obtaining smooth, even micro-sized patterns was to apply a low and constant pressure (rather than a pulsed pressure) to the syringe and to allow the polymer to be dragged across the surface of the substrate, much like writing with an ink pen. Drops of polymer are "drawn" along the substrate, keeping the syringe tip just high enough to ensure that it does not contact the substrate. A schematic illustration of the pressure controlled deposition system is shown in figure 2.
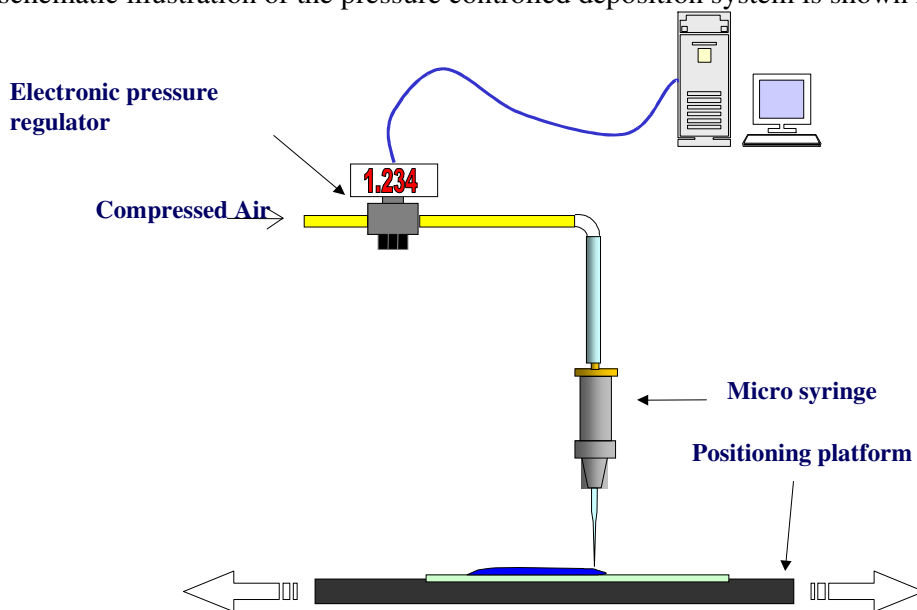

Fig. 2 Schematic illustration of the pressure controlled deposition system

The entire system including valves, pressure sensors and position controllers is interfaced to and controlled by a PC through a GPIB card. Appropriate software to drive the system, written in C, allows simple patterns such as lines, rectangular, hexagonal or triangular grids, and spirals to be deposited. More complex patterns are easy to design and incorporate into the software.

The pressure sensor and associated control system allows the applied pressure to be controlled to within ±5mmHg, and the velocity of the substrate with respect to the syringe to be varied between 0.5 and 2.5 mm/s. Each polymer concentration was deposited in various conditions of applied pressure and x, y motor velocity in order to determine a range of optimum operating conditions.

3DP system

The 3DP system is composed of three motorized planes mounted on a robust aluminium support to form three axes normal to each other. The system was projected with CAD software (fig.3). Every plane is actuated by a four phases bipolar stepper motor with an angular resolution of 1.8 °/step, dovetailed to a screw with 0.5 mm step, that transforms
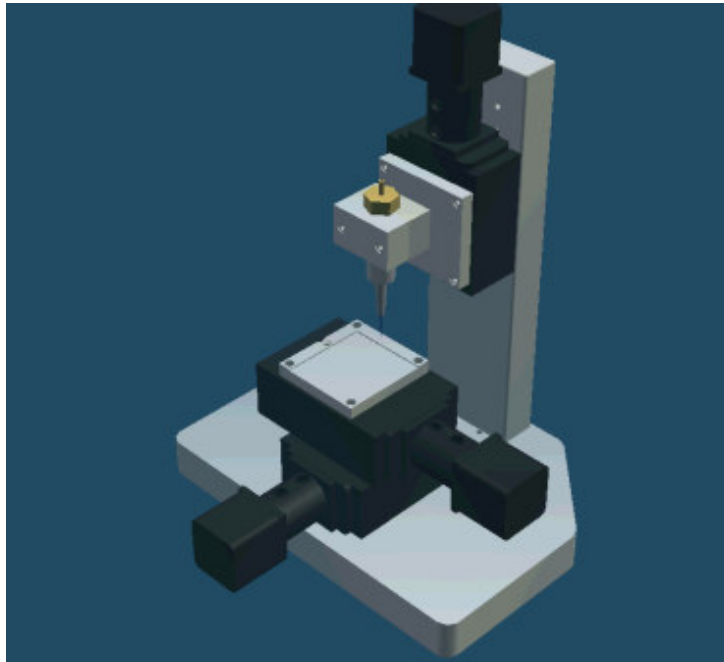
the linear motion into angular motion with a mechanical coefficient equal to 1.388 µm/°. This solution decreases almost completely the mechanical play in the presence of inversion of motion and it is able to ensure a high level of pressure even in presence of high accelerations. Stepper motors were chosen because they do require expensive position sensors. Infact, if driven correctly, it is possible to determine position of the slide at every instant with a high degree of accuracy, if the end position is known when the system is reset.

Fig.3 Mechanical structure projected with CAD software

The driver board

The characterisation required of the stepper motor driver for the system were as follows:
- to be able to give a current of 1.2 Amps

- to give a precision of 0.1 μm
- microstep operation
- to be easily connectable with a control board
- to be mechanically compatible with a rack standard 19" DIN 41494, that contains all electronic devices utilised.

The control board, installed in a PC, and adopted in this system gives major flexibility and allows compete feedback. This board allows simultaneously control of a pair of motors in asynchronous mode or in an interpolated manner. This latter method allows synchronizing the movement of 2 axes in such a way, that for each acceleration the velocity and motion direction selected, the profile of the combined motion is always rectilinear. This board has a microprocessor DSP and is able to autonomously determine the optimal sequence of impulses for perfect control of the motion of each motor, responding contemporarily to the command sent to the PC bus with specific two ports reserved I/O. At any instant the motion parameters and the trajectories to follow can be established and modified. Software with the possibility of providing constant feedback and with which the operator can see and act on the deposition parameters in real time during microfabrication process was developed. For each motor, a sequence of variable frequency impulses are generated by the driver board according to the commands sent through the I/O board of the PC.

Pressure regulator

The micro fabrication technique with the pressure driven micro syringe needs a source of compressed air, whose pressure can be regulated with extreme precision. This characteristic was obtained using an automatic pressure regulator controlled by an integrated microprocessor. With this device it is possible to:
- have a pressure range of 0-0.9 MPa;
- programme it through keyboard commands
- visualize the imposed pressure in MPa on a 3 digit display

The pressure regulator is connected to the PC with an AD/DA board

AD/DA board

The AD/DA board has 12 input and 2 output 16 bit channels.
The dynamic range for the analogue parameter in input is ±10 V to ±10 mV. This board was used to control the pressure as well as the laser sensor, described in the following. The output channel has a range between 1 to 10 V.
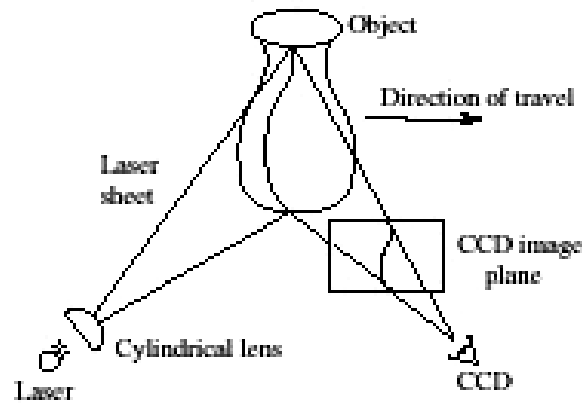
Sensor laser

To resolve the problems connected with the non planarity of the substrate and to reduce problem of tip breakage it is necessary value with high precision and for al the plane of micro fabrication the distance between the needle and the substrate. A laser sensor was used to measure the tip-substrate distance automatically connected to the PC.
The sensor should have these characteristics:
- high resolution
- low sensitivity to the external environment
- high stability
- capacity to operate with different materials
- non-destructive, non-contact
- small dimensions so as to be easily installed near the syringe.

A system with laser triangulation was selected. The physical principle used is shown in figure 4.

Fig.4 Operating principle of the laser sensor.

The collimated beam is incident on the target or surface and the position of the reflected beam is detecte4d by the sensor with great precision. Variations between the light source and the target are transduced into a linear movement of position of the reflected light spot received by the sensor. This information is then analysed by an electronic circuit with an integrated microprocessor in the device and converted into a parameter proportional to the distance. This sensor has a resolution of 1 micron at a reference distance of 30 mm and an output in current proportional to the distance from the target. The sensor element is a linear array CCD at high resolution that transforms the light information that arrives form the surface under examination into an electric value.

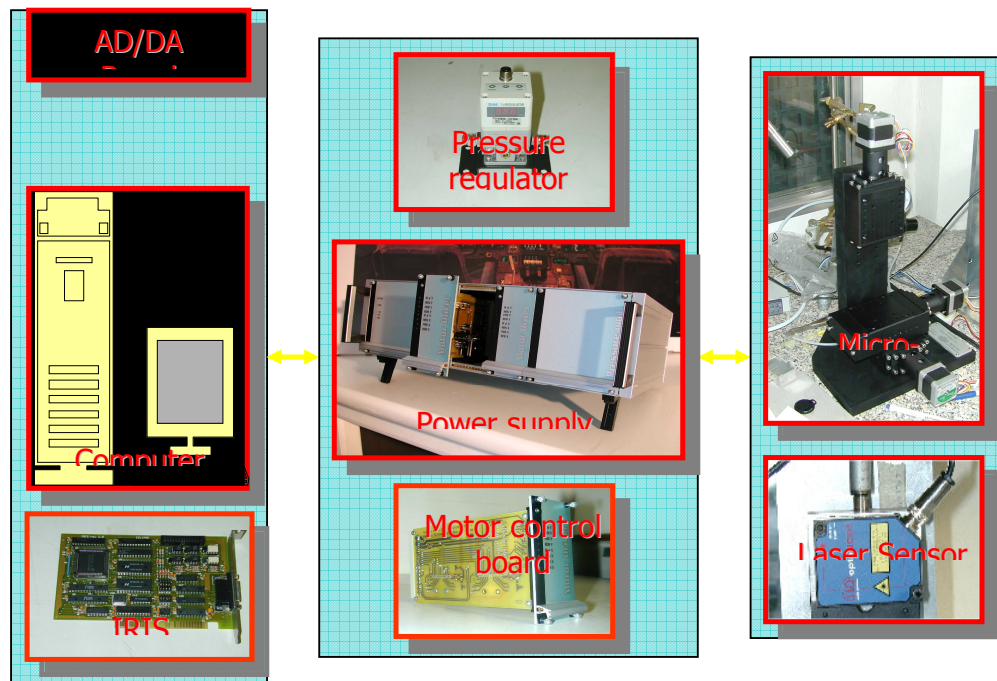A complete scheme of the microfabrication system is shown in figure 5.

Fig. 5 The complete electronic scheme of the microfabrication system

Control software

The microsyringe software was based on a simple concept: the easiest way to design a particular structure is to draw it with a high precision, high resolution pen. For this system easy to use software with the following characteristics has been developed:

- possibility to visualize on the screen the pattern to be microfabricated before start of deposition;
- edit the pattern with a graphic interface acting directly on the drawing of the structure to deposit;
- simulation of deposition to visualize any errors during the design of the structure;
- visualize in one window the state of the system with position control as well as, of the pressure and of the laser sensor, allowing to the operator to act on these parameters at any instant;
- allow the realisation and the visualition of 3D structures in a facile manner and with automatic techniques.

The software was developed in Windows with Visual Studio 6 software.

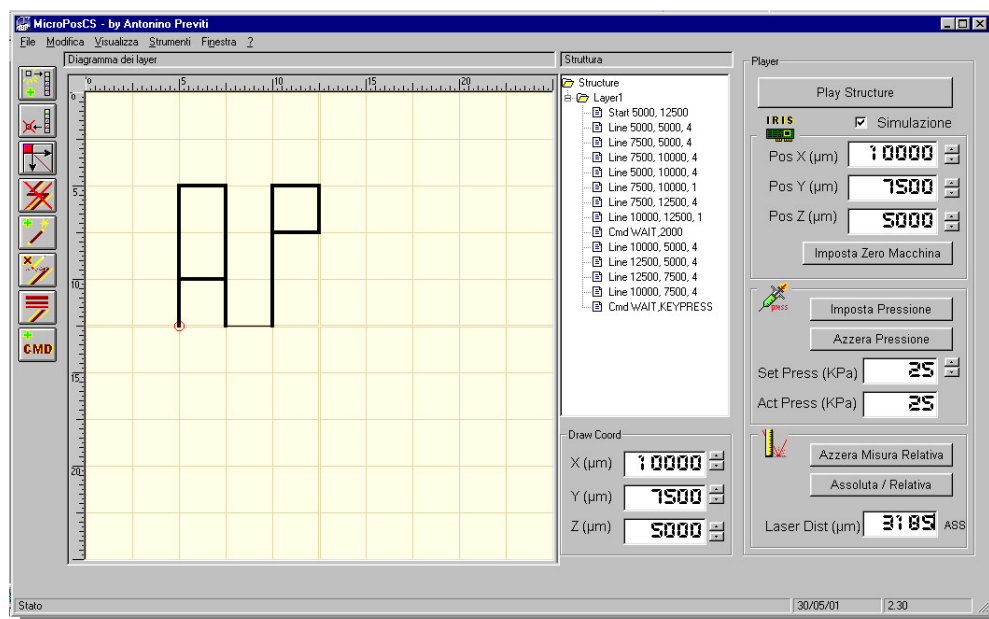The graphic window that appears to the operator is shown in figure 6.

Fig. 6 Graphic window of the software

The deposition plane is a square of side 25 mm and this gives a resolution of 50 μm per pixel. At the right of the deposition plane is the window for control of the elements that constitute the pattern and under this control there is a window that allows modifications and visualisation of the spatial coordinates relative to the deposition plane. At the top right there is the button that start the deposition phase which can be real or simulated. Under this bottom there are the controls of the position of the needle while realises the structure. Under there is the buttons for pressure control. The last button at the bottom right in the window is the control of the position of the needle relative to the substrate, detected with the laser sensor.

An important feature pf this system is that the operator can design a structure with a particular geometry, and this structure can be drawn on the software, or imported from another program, and the scaffold can be realised immediately.

6.3 Fluid dynamic model of Microsyringe deposition

A fluid-dynamic model, which enables the prediction of width and height of the patterns, was developed. The model is focused on the conditions at the tip of the needle, just before the fluid is expelled.

We assume that there is a very simple geometry at the tip, and that the fluid exits in the form of discrete drops, which are hemispherical in shape. As shown in figure 7, the forces in play at the tip where the drops form are:

- the weight of the drop, mg.
- the driving pressure, P
- the additional pressure due to vapour pressure of the solvent and the head of polymer solution in the capillary needle, P^*.
- the surface tension between drop and glass, γ

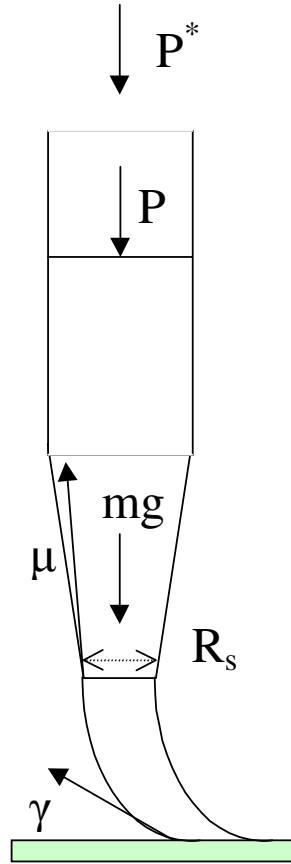- dynamic friction between fluid and glass, which is a function of the viscosity, μ, of the solution.

Fig. 7 Forces in play at needle tip

Considering equilibrium conditions, we can write a simple system of equations balancing forces and energy. Two equations can thus be obtained.

$$\begin{cases} mg + PS - \mu\dfrac{\partial v}{\partial x}2\pi rh - \gamma 2\pi r\cos\theta + P^*S = 0 \\[2mm] mgh + PV - 2\mu hv - 8\gamma\pi r^2 + P^*V - \dfrac{1}{2}mv^2 = 0 \end{cases} \qquad (6.1)$$

where

m = the mass of the drop, of volume V, density, ρ, and radius r
h= height of the drop above the substrate
R_1= the radius of the tip;

$$\gamma = \frac{mg}{2\pi R_1^2}, \, [10] \,(6.2)$$

θ= the angle of contact between solution and glass and v is the velocity of the drop.
By expressing the mass of the drop as $2/3\pi r^3\rho$, and going from Cartesian to cylindrical co-ordinates, $x = r\cos\Phi$. We assume that there is no variation in shape of drop and $\Phi = 2\pi$, so that dx=dr. Thus, we obtain:

$$\begin{cases} \dfrac{2}{3}\rho g r^3 + PR_1^2 - 2\mu h r\dfrac{\partial v}{\partial r} - \dfrac{2\rho g \cos\vartheta}{3R_1} r^4 + P^* R_1^2 = 0 \\[4mm] \dfrac{2}{3}\rho g h \pi r^3 + PR_1^2 h - 2\mu h v - \dfrac{8\pi\rho g}{3R_1} r^5 + P^* R_1^2 h = 0 \end{cases} \qquad (6.3)$$

which can be expressed as a function of the radius of the drop

$$\begin{cases} a_1 r^3 + a_2 - a_3 r\dfrac{\partial v}{\partial r} - a_4 r^4 + a_5 = 0 & (6.4a) \\[4mm] b_1 r^3 + b_2 - b_3 v - b_4 r^5 + b_5 - b_6 r^3 v^2 = 0 & (6.4b) \end{cases}$$

where

$$a_1 = \frac{2}{3}\rho g$$

$$a_2 = PR_1^2$$

$$a_3 = 2\mu h$$

$$a_4 = \frac{2\rho g \cos\theta}{3R_1}$$

$$a_5 = P^* R_1^2$$

$$b_1 = \frac{2}{3}\rho g h \pi$$

$$b_2 = PR_1^2 h \pi$$

$$b_3 = 2\mu h$$

$$b_4 = \frac{8\pi\rho g}{3R_1}$$

$$b_5 = P^* R_1^2 h \pi$$

$$b_6 = \frac{\pi\rho}{3}$$

Equation 5.4a can be integrated with respect to r to obtain the velocity of the drop as a function of r, the diameter of the tip, giving:

$$v = \frac{a_1}{3a_3} r^3 - \frac{(a_2 + a_5)}{a_3}\ln r - \frac{a_4}{4a_3} r^4 + K \qquad (6.5)$$

which can be substituted into 5.3b, giving an equation that depends only on r. In order to simplify the equation, we can assume $ln\ r \approx r$, since r is small.
An 11th degree equation is obtained, which can be written as:

$$c_{11} r^{11} + c_{10} r^{10} + c_9 r^9 + c_8 r^8 + c_7 r^7 + c_6 r^6 + c_5 r^5 + c_4 r^4 + c_3 r^3 + c_1 r + c_0 = 0 \qquad (5.6)$$

where:

$$c_6 = \left(\frac{2Ka_1b_6}{3a_3} \right)$$

$$c_5 = \left[-b_4 - \frac{b_6\left(a_2^2 + a_5^2 + 2a_2a_5\right)}{a_3^2} \right]$$

$$c_4 = \left(\frac{b_3a_4}{4a_3} \right)$$

$$c_3 = \left(b_1 - \frac{b_3a_1}{3a_3} - b_6K^2 \right)$$

$$c_1 = \left[\frac{a_2b_3}{a_3} + \frac{a_5b_3}{a_3} - \frac{2Kb_6\left(a_2 + a_5\right)}{a_3} \right]$$

$$c_{11} = \left(\frac{-b_6a_4^2}{16a_3^2} \right)$$

$$c_{10} = \left(\frac{b_6a_1a_4}{6a_3^2} \right)$$

$$c_9 = \left(\frac{-b_6a_1^2}{9a_3^2} \right)$$

$$c_8 = \left[\frac{-a_4(a_2 + a_5)b_6}{2a_3^2} \right]$$

$$c_7 = \left[\frac{2b_6a_1(a_2 + a_5)}{3a_3^2} + \frac{a_4b_6K}{2a_3} \right]$$

$$c_0 = \left(b_2 - b_3K + b_5 \right)$$

A MATLAB routine was written to solve equations 4 and 5. The value of the constant K, for each pressure, was obtained by resolving equation 4 for $r=R_1$, where the velocity of the fluid is zero (condition of no-slip of fluid) at the walls of the tip of the needle. Known values of viscosity, contact angle, density of polymer solution, and diameter of tip of needle were input for a range of pressures (P+P*) between 0 and 200 mmHg and a corresponding vector of K values was obtained. For all pressures greater then 10 mmHg, K is approximately constant for a given polymer solution (between 4.10^{-4} m/s and 1.10^{-4} m/s for PCL and between $2 \cdot 10^{-4}$ m/s and 7.10^{-6} m/s for PLLA). K represents the limiting value of velocity of the drops for very small tip diameters, and as expected decreases with decreasing solution viscosity.

The roots of equation 5 were found by inputting a range of pressures, P+P*, between 0 and 200 mmHg. Of the 11 solutions obtained, we selected the real solution that was closest to the experimentally measured value of half width of the patterns. In general only one of the 11 roots was suitable.

The value of the additional pressure P*, was then obtained by comparing the nominal pressure applied to obtain a given line half-width (corresponding to r). The difference between experimental applied pressure and P+P* was taken to be P*. Using this method, P* was constant with a value of about 1000 Pa or 7 mmHg for all pressures above 10 mmHg. Using this technique, the curve obtained from the experimental data

fits the curve obtained from the model almost perfectly. The data are presented in figures 8 a, b, c and d, in which line widths for the polymers at different concentrations are plotted as a function of applied pressure.
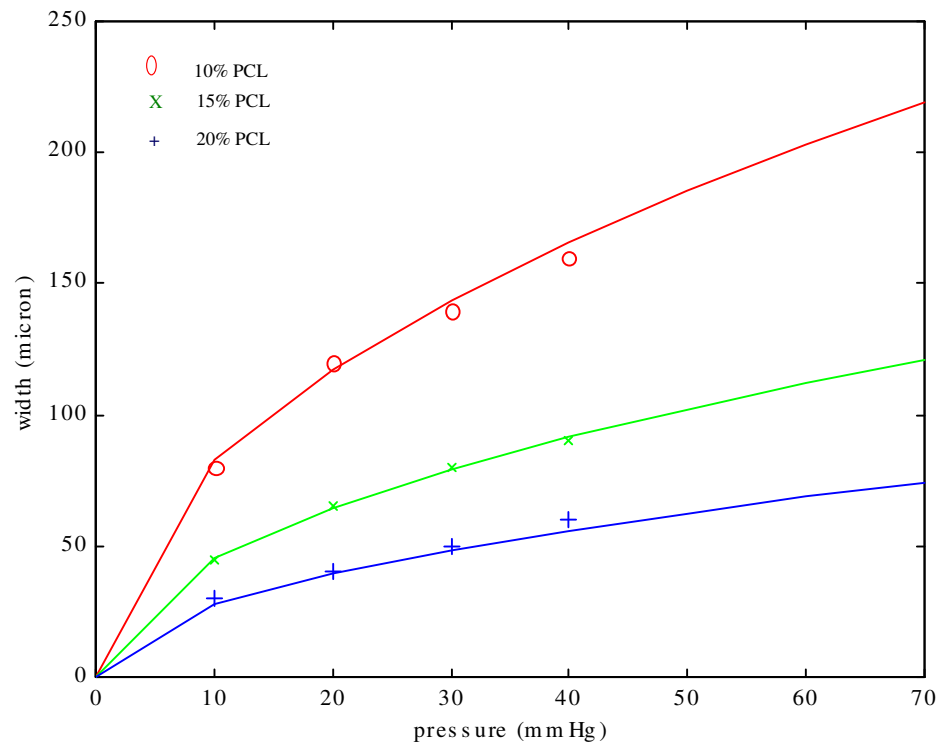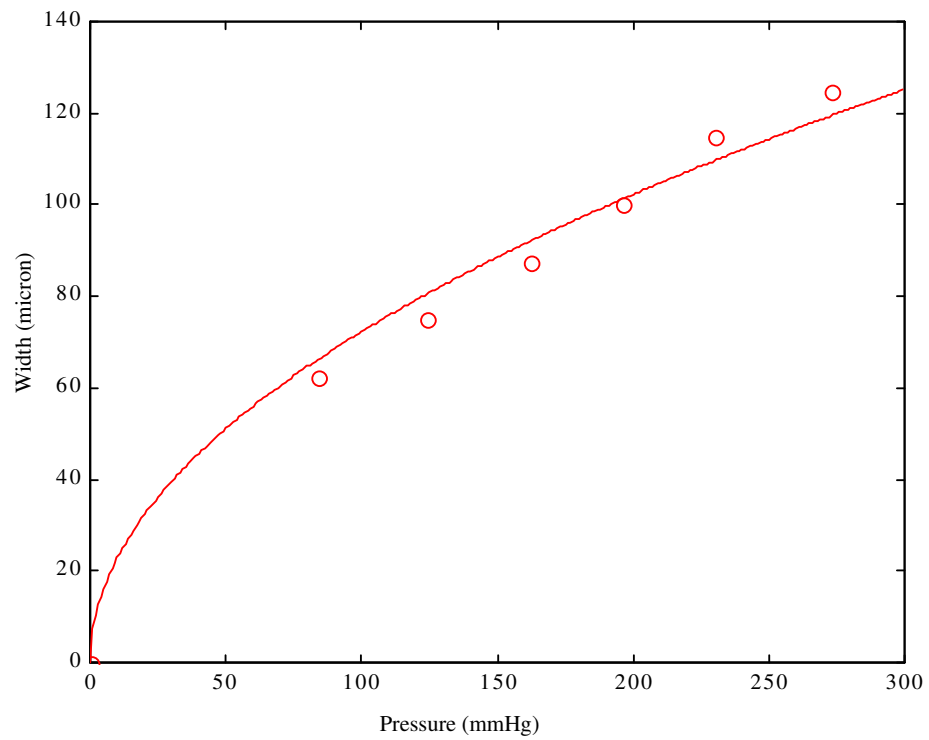

Fig.8a Width line of PCL as a function of pressure for different concentration
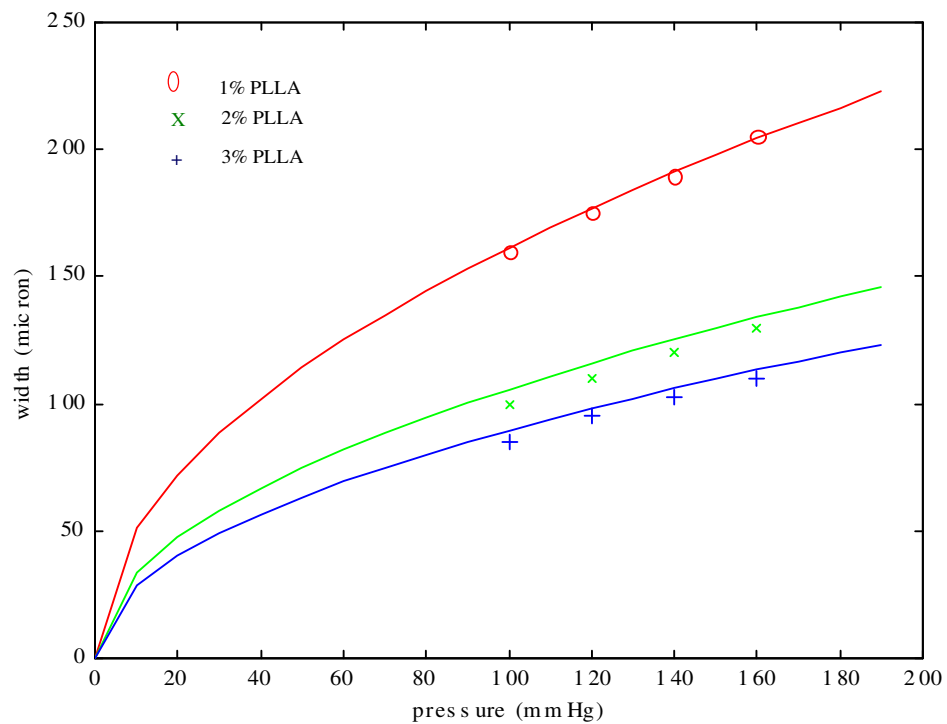
Fig. 8b Width line of PLLA as a function of pressure for different concentration
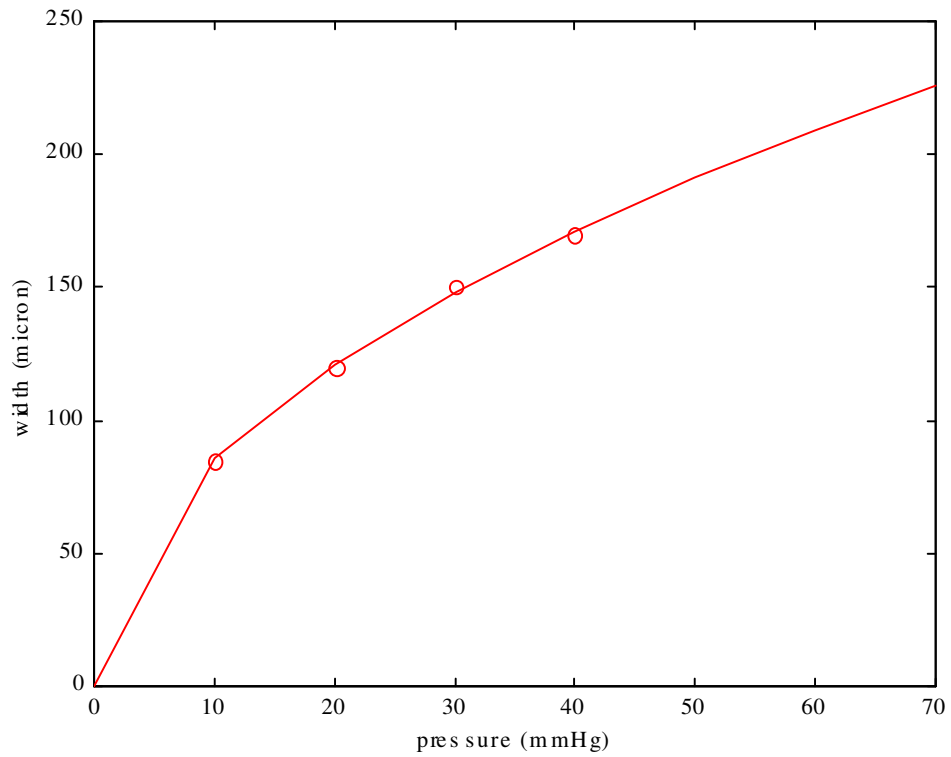
Fig.8c Width line of blend of PCL and PLLA as a function of pressure

Fig. 8d Width line of blend of PLGA as a function of pressure