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Physical-chemical properties of PDMS samples used in tunable lenses

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Abstract*—The transparent elastomer Polydimethilsyloxane (PDMS) Sylgard 184 is increasingly used in optical applications, as in the manufacture of micro lenses with variable focal length, in waveguides (optical fibers), or to produce lenses doublets; this is all due to their excellent physical-chemical properties, its low cost, easy operation and nontoxicity. We are able to elaborate thin membranes with this material and we can use them as refracting surfaces on Variable Focal Length Liquid Lens (VFLLL),also we can build a lensbody and use it on tunable solid elastic lenses (SELs); the material works on the visible region of the electromagnetic spectrum. For this work the membranes and the lens body have been subjected to pressure changes, in particular to tension and compression, and we have calculated some interesting features like its Young´s modulus and compressive modulus. At this time there are no reports in the literature of the compressive properties of the material.In this paper we describe the manufacturing process of elastic samples prepared from PDMS and also a physical-chemical characterization and applications are presented. Finally, we present the experimental results obtained from the physical-chemical parameters of the membranes and the lensbody and conclusions; the obtained results show that the PDMS is feasible to use for this application.

***Index Terms*—Elastic membranes, liquid lenses, PDMS characterization, physical-chemical properties of PDMS, tunable lenses.**

I. INTRODUCTION

The fabrication process of membranes and a mainlens bodymade from polymeric material is relatively easy, and the use of this reduces time and complexity in the making of prototypes; besides of its low cost and non-toxicity this material possesses certain features like high transparency and high efficiency and that is why it is an ideal material to use in the optical technology area [1]-[3].

In recent years the elastic polymeric material polydimetilsiloxane known as PDMS Sylgard 184 has become of great interest due to the near future expectations and also it is the one from which more applications have been reported in the literature [4]. Specifically in the area of optics it has been reported the use of liquid lenses with variable focal length and micro lenses formed by two transparent elastic membranes made of PDMS with a liquid in between them, which can change its focal length by modifying the amount of liquid within it, it has also been reported the use of this material for optical waveguides and optical fiber complements [5]-[8]. These works only present the shape that the membranes acquire and the images formed by them; nevertheless they have left behind the fabrication process and the physical-chemical characterization of the membranes. And the same analyses apply for solid elastic lenses.

The inherent properties that exhibit the membranes or the lensesbodiesdepend of their physical-chemical nature and the fabrication process which was used, hence it is important to know about them in order to produce membranes with certain desirable properties for a specific application. The membranes and the lensesbodiesfabrication process may vary, some of them involve soft lithography, others use centrifuge, and it can also vary the concentration of the components for obtaining the mixture and the curing temperature and time [6]-[11]; therefore it can be produce a membrane or a lens body with specific properties by the combination of such parameters.

We propose in this work an alternate method that is easy and cheap for the elaboration of PDMS membranes and SELs,we present a physical-chemical characterization of some of their most relevant parameters for optical applications from which will allow us to know if such membranes or lensbodyare suitable for their use as refractive surfaces on liquid lenses or SELs. A summary is presented of the relevant measured parameters of the items produced with the described process in the next section followed by the physical-chemical characterization and finally some brief conclusions are presented.

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II. FABRICATION PROCESS

The proposed fabrication procedure of PDMS membranes is straightforward due to the easy curing process of the polymer, easy handled of the material and an excellent sealing property that it possesses. The thickness of the membranes is about 0.1 mm to 1 mmwith diameters from 2 cm to 10 cm;the lens body is about 2.2 cm to 2.2 cm of length and thickness is about 2.2 cm to 2.4 cm. The membranes are used as refractive surfaces for liquid lenses and are deformed by modifying the pressure of the liquid contained within themand thelensbodiesare used in SELswhere its shape changes by modifying the load at the lens border. The process to elaborate the mixture is the same in both cases. We decided to use a simple, fast and inexpensive process due to the dimensions and it does not involve the use of sophisticated equipment. We used PDMS Sylgard 184 provided by Dow Corning [4].

The developed methodology for the fabrication process mainly consists of 4 steps: 1) Weigh of the components in a 10:1 proportion (base: curing agent) for this step we used an Ohaus analytical balance model Voyager [12]. 2) Manual components mixture until homogenous phase is obtained (a laboratory beaker was used for this step). 3) Extraction of air bubbles in the mixture (we used a vacuum chamber and an ultrasonic tub), we applied vacuum and ultrasonic bath alternately for a total of 10 cycles with a duration of 3 minutes each. Finally, 4) the mixture is poured on a flat glass surface previously cleaned, and letting at rest for curing in a horizontal position inside of a laminar flow hood for 7 days. The laminar flow hood (Labconco model 36212-04) was used to avoid the pollution form solid particles. The entire process was done at a temperature of 25 °C (the optimal mechanical resistance was achieved in 7 days like all of its mechanical and electrical properties.) It is important to mention that the curing time may vary when the temperature is modified; curing times of about seconds to hours has been reported previously [10], [11].

III. CHARACTERIZATION OF MEMBRANES AND THE LENSBODY MADE OF PDMS

The membranes and the lens body characterization consists in knowing the highest number of physical and chemical properties in order to predict its behavior but above all to know those which are related with its performance when these are subjected to pressure changes. We present in the next sub-sections the chemical, mechanical and optical properties measured of the PDMS items.

A. Chemical Properties

Some of the properties presented below were provided by the supplier or taken from the literature. The polydimetilsiloxane is a widely used organic polymer due to its high crystallinity, non-toxicity, high viscosity in liquid state; it is non-flammable and inert; it is also known as dimethicone. The chemical formula of PDMS is $(CH_3)_3SiO[Si(CH_3)_2O]_nSi(CH_3)_3$, where *n* is the number of repeating unitsof the monomer $[SiO(CH_3)_2]$. Commercially it is supplied in two components, a lot-matched base (vinyldimethylsiloxane with a platinum catalyzer) and a curing agent (hydridedimethylsiloxane done).The addition of the platinum catalyzer helps to the reaction to the functional vinyl group of the monomer base ($SiCH=CH_2$) and the functional hydride group (SiH) that is present in the catalyzer (curing agent) and it results in the curing of the mixture of the two components. This reaction is known as catalytic hydrosilation [13].

The supplier suggests mixing the components in a 10:1 proportion (base: curing agent) in order to obtain a mixture with mechanical and thermal efficient properties but it can vary according specific needs. Molecular weight of the repeating unit of the PDMS is 207.4 g/mol, with an average numeral molecular weight of27000, a density of 0.982 g/mL and it possesses a viscosity of 500 cSt [4].

We obtained aRamann spectrum of the membrane using a Ramann spectrometer QE6500 from Ocean Optics, with a light source of wavelength of 785 nm. The spectrometer has a probe with a two fiber end, one for collection and the other for illumination of 200µm and 90 µm respectively. Fig. 1 shows the graph of light dispersion from 800 nm to 935 nm, and as seen in the figure, dispersion lines appeared giving us information about the molecular composition of the membrane.

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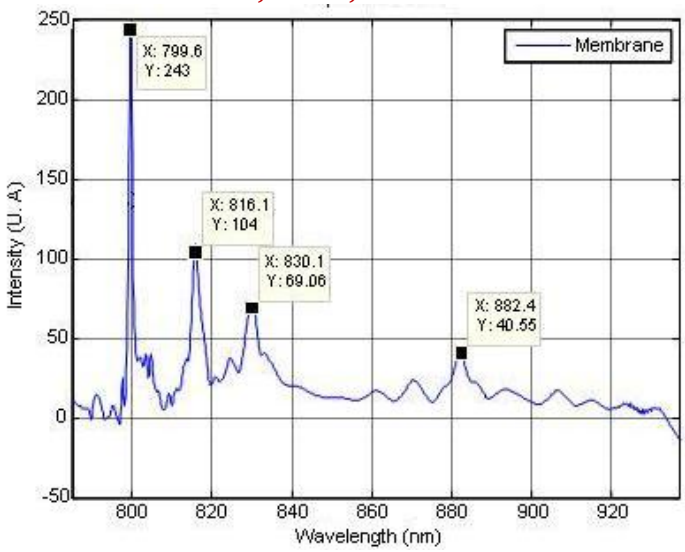

Fig 1- Raman spectrum from the membrane made of PDMS.

Four lines were found at 799.6 nm, 816.1 nm, 830.1 nm and 882.4 nm where the first line corresponds to the laser carrier, and the other three correspond to the CH interaction (curved vibrational mode for being a tridimensional molecule). It has been reported in the literature the Strech vibrational modes in the visible region of SiO interaction of 490 cm^{-1} and for the SiC of 713 cm^{-1} [14]. Experimentally it has been observed that the liquid medium (distilled water – non-ionized) does not react with the membranes nor any change in their physical properties is produced.

We also used a confocal laser scanning microscope model LEXT 3D OLS3100 from Olympus and an optical profilometer model Wyko NT from Veeco in order to obtain the surface texture, the rugosity of the sample, the size of the pore and the membrane topography which are shown in the next figures.

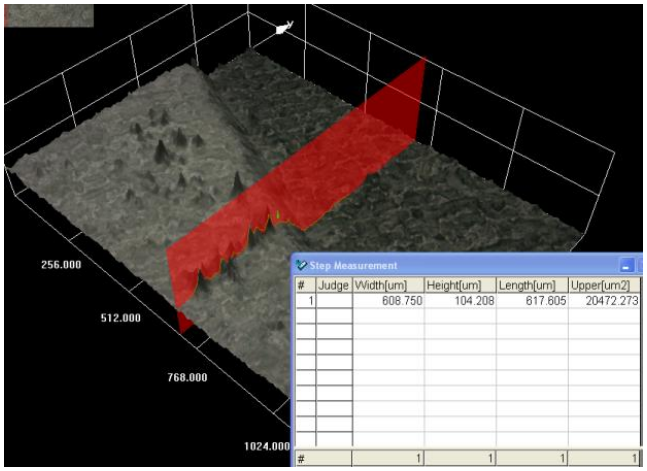

Fig 2.-Texture of the membrane obtained with a confocal laser scanning microscope.

Figs. 2-5 show that there exists presence of dust particles over the membrane surface. We found that the average rugosity was of 550.87 nm and the maximum pore size was of 80 µm by 80 µm. Fig. 2 shows irregularities in the membrane texture and this cause diffuses reflections for short wavelengths. Fig. 3 shows the membrane rugosity, it can also be seen in this figure variation in the topography in the form of peaks and valleys and this could be due to trapped air between the membrane and the microscope slide, a difference of 3 µm peak-to-valley is shown.

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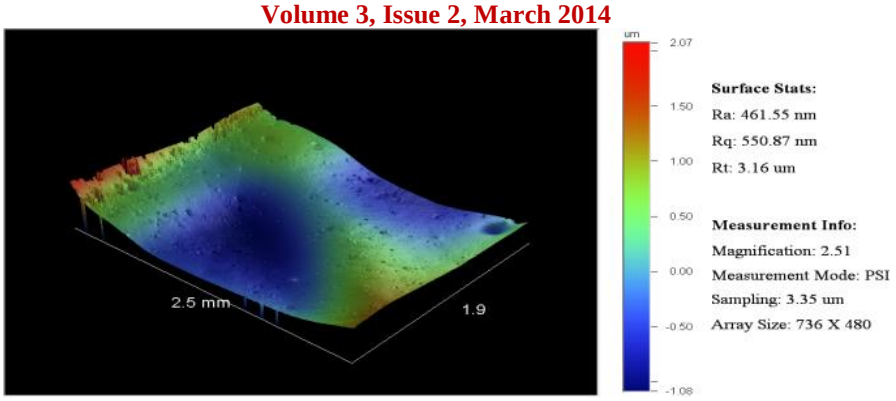

Fig 3. Topography from a cross-section of the membrane.

In Fig. 4 we present an area section of the membrane of 1.9 mm by 2.5 mm and the profile in the x and y axis is depicted.

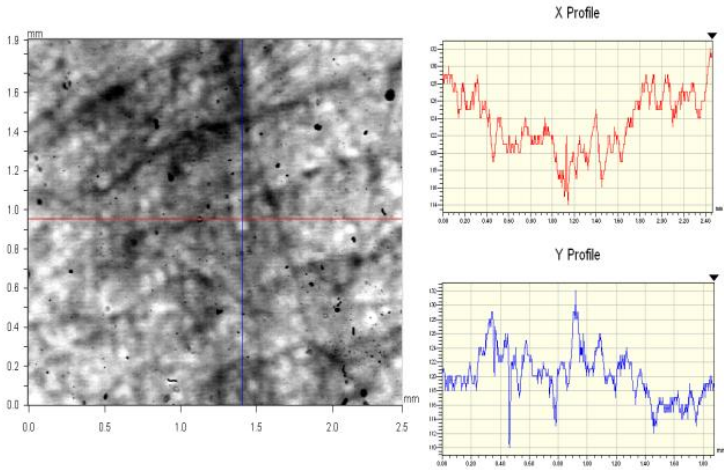

Fig 4. Membrane profile across the x and y axes.

Finally, in Fig. 5 spores and impurities are shown in the surface of the membrane, which may be due to the presence of the air pollution.

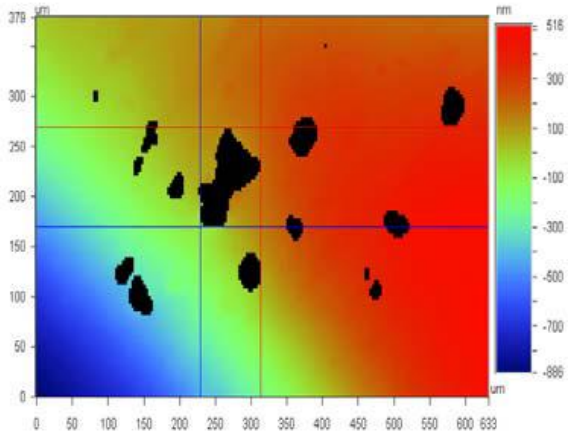

Fig 5.- Membrane cross-scetion with dust particles and pores.

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B. Mechanical Properties

For the mechanical characterization of the elastic material a tensile and compressive test analyses were performed in order to obtain its Young´s modulus, Poisson ratio, shear modulus, yield strength, density, thickness, elastic range, traction limit, fracture strength, compressive modulus and; a commercial device that is equivalent to a universal testing machine was used for this purpose [15].For the tensile test the original length of the probe was 110 mm and the width of 13.7 mm.

The ASTM D695 standards were adopted to perform the compressive test [16]. The probes were made according to the process described in section II with some variations in the temperature and in the curing time; they were introduced into abenchtop muffle furnace for 20 min at a 200 °C temperature. A small percentage of air bubbles were detected in the probes.The probe dimensions were 12.4 mm of length, 12.53 mm of width and 22.08 mm of height. We performed 290 measurements to obtain stress, unitary deformations, elastic modulus and compressive modulus with which a unitary stress-strain curve was built. In Fig. 6 we show a linear regression for of the measurements.

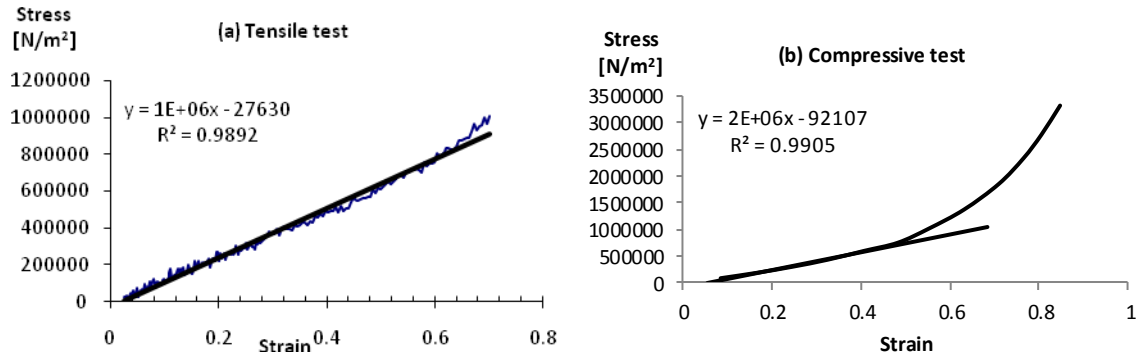

Fig 6.- (a) Linear regression of the measurements of the tensile test. (b) Linear regression of the measurements of the compressive test.

The tensile test is shown in Fig. 6(a), the linear regression was performed to the stress-strain curve and we can see that the line starts to separate approximately at 700 KPa, which corresponds to the yield strength since at this point the linear zone of the curve is ended (we decided to round this quantity only for convenience.)As it is not clear the transition zone in the curve, the yield strength should be calculated as the strain at which the material exhibits a plastic deformation of 0.2 %, however the material never enters the plastic zone and it rather seems that it becomes more resistant when the load is increased, opposite to the metals. This is the reason why the yield strength is just on the transition zone (when the linear zone ends in the regression).

In Fig. 6(b) the linear regression of the measurements of the compressive test is shown. We can see that approximately at 1 MPa the linear zone of the curve is ended, therefore this value corresponds to the compressive strength,the stress measured at the point of permanent yield.

A Young's modulus of 1.2 MPa with a standard deviation of ±24.1 KPa was found and a correlation coefficient of 0.9945 at a temperature of 24 °C. A Poisson ratio of 0.46 was obtained and a thickness of 330 µm was measured (we used an OCT 930 system). With these values and from the unitary stress-strain curve we found that the shear modulus of the material is 411 KPa, the yield strength and the traction limit is 700 KPa and 1.9 MPa respectively. A compressive modulus of 2 MPa and compressive yield strength of 1.01 MPa were found.

C. Optical properties of the membranes

We want to know if the membranes are suitable to be used as refractive surfaces of a liquid lens of variable focal length and hence it is important to know the optical properties of the membranes in the visible region of the spectrum to predict its performance. Some relevant parameters are the index of refraction, spectral transmittance and absorption, thickness, anisotropy factor, transmission and reflection coefficients and absorption and dispersion coefficient.

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The index of refraction was experimentally measured with an Abbe projection refract meter model WY1A from Xintian Fine Optical Instrument Corporation, and the value was n = 1.4325±0.0001, which it is similar to those reported in literature [8], [9].

The absorption and transmission graphs were experimentally obtained with a 300-visible UV Spectrometer from Unicam. As we can see from Fig. 7 and Fig. 8 the membranes absorb less than 0.05% and transmit over 95% of the visible radiation, hence we can say that they have an efficient optical performance.

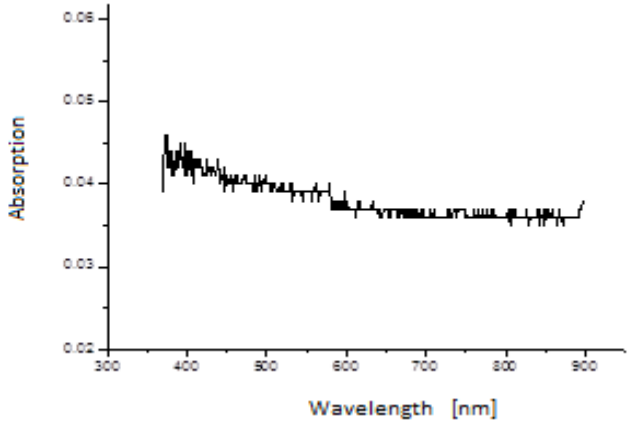

Fig 7. Absorption spectral range of the PDMS membrane of 300 µm width.

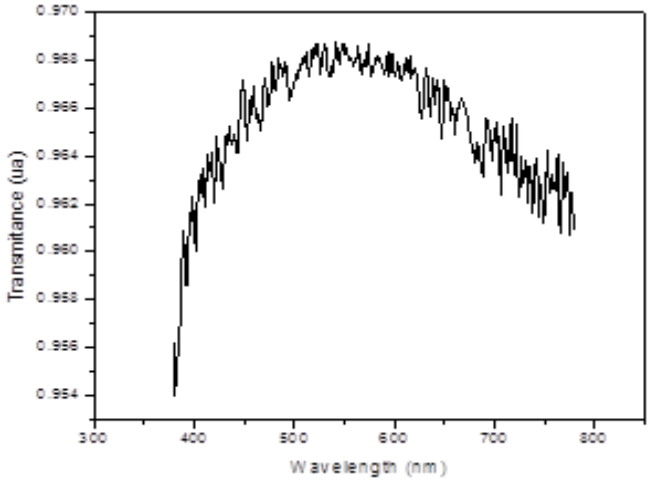

Fig 8. Transmittance spectral range of the PDMS membrane of 300 µm width.

An OCT was used in order to measure the membrane thickness and so we found a value of 330 µm. Also, a horizontal sweep was made with the OCT and in Fig. 9 we show the membrane profile and its homogeneity in a qualitative way.

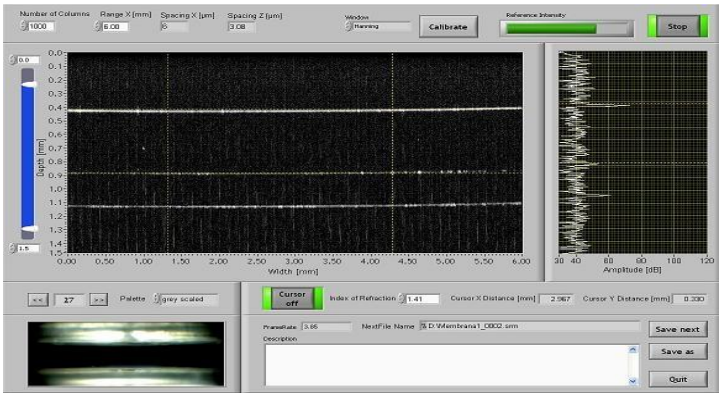

Fig 9. Output of the OCT system

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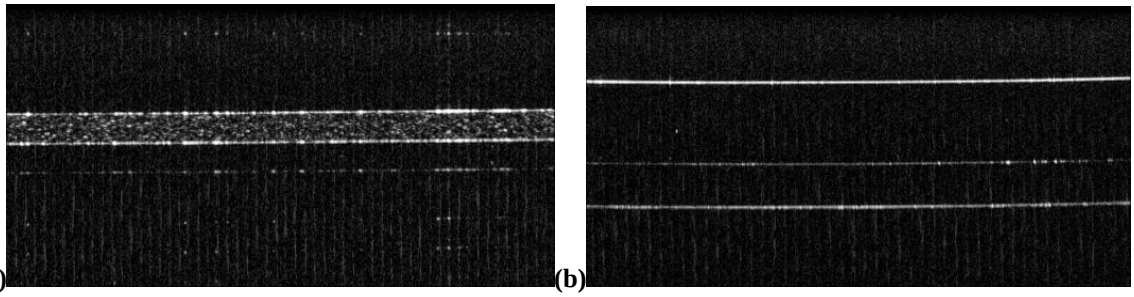
In Fig. 10 we can see a qualitative comparison between the membrane profile and its homogeneity against microscope slide acetate. It can be seen in the upper section of Fig. 10(a) a great amount of diffusive elements that are located in the acetate (bright spots), and in the lower section only the presence of a single diffusive element in the study region of the membrane. In Fig. 10(b) it is shown the membrane along together with the acetate and it was found that the acetate does not show presence of any diffusive elements, however only a single one appeared in the membrane. The OCT device that we used is an OCT 930 Spectral Radar with an operational wavelength of 930 ± 5 nm, an spectral bandwidth of 100 ± 5 nm, an optical power of 2 mW and a depth of image of 1.6 mm.



(a) (b)

Fig 10.- (a)Transversal view from diffusive elements in the microscope slide acetate. (b) Transversal view of the membrane homogeneity

Finally the transmission and reflection coefficients were obtained by placing the membrane among two integrating spheres; we used a He-Ne laser with an operational wavelength of 632.8 nm and an output power of 15 mW [16]. The measured values where T=0.90 and R=0.082. As we take these values together with the membrane thickness and the index of refraction we could calculate the absorption coefficient equals to 0.025, the dispersion coefficient equals to 252.57 and the anisotropy factor equals to 0.959. For the calculation of these data we implemented the Monte Carlo method, along together a developed genetic algorithm and we considered a 10000 photons population.

IV. CHARACTERISTICS OF THE MATERIAL

In this section we present a summary of the properties that the elastic material exhibit, most of them were experimentally obtained and some others were provided by the supplier or taken from the literature [4], [18]-[19], see Table 1.

Property	Feature	Consequence
Chemical	Repeating unit molecular weight of 207.4 g/mol. Number average molecular weight of 27000. Density of 0.982 g/ml. Viscosity of 500 cSt.	Maximum pore size of 80 µm2. Rrugosity of 550.87 nm. Ramann spectral lines at 816.1, 830.1 and 882.4 nm. Membrane thickness of 0.330 µm.
Optical	Transparent. UV cut-off frequency at 240 nm. Refraction index of 1.4235. Absorption of 0.04% and transmission above 95% in the visible region of the spectrum.	Optical detection from 240 to 1100 nm. Transparent in the visible region of the spectrum. Homogeneous, linear and isotropic.
Electrical	Isolator. Breakdown voltage of 2x107 V/m.	Allows inlays in circuits. No failures when opening connections.
Mechanical	Young's modulus of 1.2 MPz. Poisson's ratio of 0.46. Shear modulus of 411 KPa. Traction limit of 1.9 MPa. Yield strength 700 KPa. Density of 0.982 g/ml. Compressive modulus of 2 MPa. Compressive yield	Adopts the shape of the container. Allows formability and it is reversible when it acts on the deforming.

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	strength of 1.01 MPa.	
Thermal	Isolator. Thermal conductivity 0.2 W/mK. Thermal expansion coefficient 310 µm/m°C.	It may be used as thermal isolator. Does not allow for resistive heat dissipation of electrophoretic separation
Interfacial	Low superficial free-energy ≈ 20 erg/cm2. Elongation at 100 %.	Replicas are easily removed from the mold. Reversible seal in materials which contains aqueous solutions in channels.
Permeability	Water impermeable. Permeable to gasses and non-polar organic solvents.	It allow to transport gas in great amounts of the material. Non-compatible with many organic solvents.
Reactivity	Inert. It can be oxidize by exposition to a plasma Bu4N+F-[(TBAA)F].	Non-reactive with many chemical substances. Surface can be etched. It can be modified to be hydrophilic and also reactive with hydrogen and silicon. Etched with (TBA)F can alter the surface topography.
Toxicity	Non-toxic.	Can be implanted in vivo. It supports growth of mammal cells.

This summary presented in one single document will allow knowing the feasibility of the use of elastic membranesor a lens bodyfor a variety of applications in several scientific and technological areas. In the case of the above mentioned application the fabricated membranes and theelastic lensbody exhibit excellent properties, such as their high crystallinity and their elongation which surpasses the required values and fulfill the required expectations. Finally, because of the properties of the membranesand elastic lensbody depend on the fabrication process and their physical-chemical nature, the fabricated elements under the proposed process in this work exhibit similar properties to those reported before in the literature and variations should be due to the differences in the used process.

V. CONCLUSIONS

In this work we presented a methodology for the fabrication of elastic membranes and lens body of PDMS which are used in liquid lenses and in SELs; also a characterization of their chemical, mechanical and optical properties is reported, which are of great interest for such application. These membranes are used as refractive surfaces and at the same time they serve as containers of the liquid medium between them, besides they change their curvature radius by modifying the pressure in the liquid within them. The lenses bodies are used in tunable optical systems were pressure changes are applied to the main lens body and a shift of its focal distance is presented due to the deformation. The physical parameters and properties of these items can help us to predict and to simulate a desired behavior for the proposed application for instance the range of the applied pressure won't affect their mechanical and optical performance. The proposed fabrication method is simple and inexpensive since it does not involve the use of sophisticated equipment and even still, we could replace those employed for home-made material. PDMS is a material with desirable features and properties for its use in liquid lenses and SELswhich has allowed their use in several fields of technology and knowledge, offering fast, simple and inexpensive solutions mainly in the micro-engineering area. The perspectives of the use of this material are highly promising and every day new applications are reported.

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Agustin Santiago Alvarado. Has been a research professor since 1998 at the Mixteca Technological University. He received his Physics degree in 1993 from the Puebla Autonomous University (BUAP), México. He received his MS degree and PhD degree in optics in 1995 and 2001 respectively both from the National Institute of Astrophysics, Optics and Electronics (INAOE). He is a national researcher for the Mexican National Research System. His research interests include optical testing, optical design and adaptive optics.