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Highly doped N type nc-SiGe:H deposited in a low frequency PECVD system at 200ºC

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ABSTRACT : *Highly doped phosphorus doped nc-SiGe:H is deposited by low frequency (LF) PECVD at 200ºC, 1.2 Torr of pressure at RF frequency of 110 kHz. The dilution ratio defined as $R = Q_{H2}/(Q_{SiH4} + Q_{SiH4})$, where the Q_s are the gas flow of the reactive and diluent gases is studied for obtaining the highest conductivity possible in undoped films of nc-SiGe:H, later these conditions are used to dope the material with P in the range 2 – 12%. The best films are obtained at 4%P gas content and resulted in a conductivity value at room temperature of 0.11 $\Omega^{-1}cm^{-1}$, it is also noticed that the doping efficiency and the defect generation remain constant and independent of the doping level after 4%P gas. From FTIR analysis it is found that the presence of P reduces the weak Si-Si bonds and enhances the population of Si-H and GeH bonds.Because its improved characteristics films of this material can be used in TFTs as source and drain regions, in flexible electronics as an inorganic semiconductor and in solar cell as emitter.*

KEYWORDS *Nanocrystallyne, doping, conductivity, defects.*

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I. INTRODUCTION

Hydrogenated amorphous silicon germanium films (a-SiGe:H) have been extensively studied because of the tunable band gap that open their use in a wide range of devices as a meaningful building block in multi-junction thin-film silicon-based solar cells [1] or for improving the performance of thin film a-Si:H solar cells [2], in TFTs which are used as IR image-sensign touch displays [3], as thermosensing material in microbolometers [4], just for mentioning some of the most common applications of a-SiGe:H. Recently, the development of flexible electronics requires the deposition of tin film semiconductor materials with high mobility and low deposition temperature, where issues of cost, large area applications and scalability play a crucial role in material selection [5], a-SiGe:H deposited by PECVD at low temperatures seems to be a good alternative as semiconductor material that may fulfill the aforementioned requirements. Many reports on the deposition of a-SiGe:H have been published and the different methods used result in enhancement in some property of the material but, none of these have demonstrated high dark conductivity (σ_d)at deposition temperatures as low as 200C. For instance, by using a hollow cathode reactive sputtering system [6] it have been deposited films of a-SiGe:H at 285ºC with a conductivity of $4.5x10^{-8}$ S/cm; PECVD films deposited with high plasma power at 210-220ºC substrate temperature resulted in conductivity around $1.5\text{-}2x10^{-8}$ S/cm [7]; in a conventional capacitively coupled RF-PECVD system at 13.56 MHz maintained at a high base vacuum of ~10-7 Torr and at substrate temperature of 220ºC [8], have deposited film with conductivity as high as $2.38x10^{-3}$ S/cm; a-SiGe:H deposited by in a 40 MHz VHF-PECVD reactor [9] resulted in σ_d values in the ranging form $2x10^{-11}$ to $6x10^{-7}$ S/cm for Ge content from 0.4 to 0.8 respectively. Recently [10], nc-SiGe:H has been proposed as an ideal absorber material for multi-junction Si thin film solar cells because of its simultaneous spectral sensitivity in the infrared range and good stability against light exposure. For the deposition of nanocrystalline SiGe (nc-SiGe:H) alloys it is necessary a high dilution rate of the reactive gasses as is also mentioned in reference [10], an the authors also the point out that it is quite challenging to produce good quality SiGe thin films with sustained nanocrystallinity, particularly at low growth temperature. Therefore, it is a challenging task to find the way for depositing films of nc-SiGe:H with high conductivity, high Ge content, and low deposition temperature.

In this work, the deposition conditions for a-SiGe:H with high conductivity and improved characteristics in a low frequency PECVD System are presented, and because of the temperature here used the

resulting nc-SiGe:H it is proposed for its use not only in the aforementioned applications, but it is proposed as an inorganic semiconductor in flexible electronics that use substrates as Polyimide (Kapton), Polytheretherketone (PEEK) or Teflon among others.

II. EXPERIMENTAL

Films of undoped and doped silicon-germanium (a-SixGe_{1-x}:H) were deposited by low frequency (LF) PECVD in a capacitive discharge system (AMP-3300 system, Applied Materials) at RF frequency of 110 kHz. The deposition parameters for depositing the films were: substrate temperature of 200 °C, chamber pressure of 1200 mTorr, and a power density of 90 mW/cm^2, the source gasses used were silane and germane. Then the H_2 dilution rate defined as R = Q_{H2}/(Q_{SiH4} + Q_{SiH4}), was chosen as that for which the undoped nc-SiGe:H had its highest dark conductivity, the ratio resulted to be R = 20 and the following rate flows were set constant for tall the experiment; Q_{SiH4} = 5 sccm, Q_{GeH4} = 45 sccm and Q_{H2} = 1000 sccm. With these flow rates a high dilution rate and high pressure are reached, both requirements for the incorporation of nanocrystals of the material into the growing amorphous net. For the phosphorous doped films the flow rate of PH_3 was varied in the range 1-12% in gas phase in accordance to the relation $\%PH_{3\,gas\,phase} = Q_{PH3}/(Q_{SiH4}+Q_{GeH4})$, given that our gas source of P_{H3} is 1% diluted in H_2, the Q_{PH3} was adjusted for maintaining the dilution rate R = 20. Table 1 shows a summary of the deposition parameters used in this work. The films were deposited on c-Si wafers and on n corning glass substrates 2947.

An Atomic Force Microscope (AFM) was used for measurement of the mean roughness (Ra) of the films, the system used was the Nanosurf Easy Scan 2.3. Besides, a high-resolution Field Emission Scanning Electron Microscope (FE- SEM) FEI Scios was used to see the changes in surface morphology of the films undoped and doped as the P is incorporating into them. The thicknesses of the films were measured with a DekTak XT-E profilometer, and from those the deposition rate of the films was calculated. The hydrogen bonding in the films was studied by FTIR spectroscopy. The IR absorption spectra of the films were measured with a Brucker infrared spectrometer, Model Vector-22 in the range 400–4000 cm^{-1}. On the corning glass substrates 2947 and through a shadow mask, Ti strips of 0.3 μm thick, 16 mm long and 2 mm separation were e-beam deposited. On these glass substrates the a-SiGe:H films were grown, in this way the interface metal-semiconductor is preserved from environment impurities, they were used for electrical characterization. The room-temperature conductivity (σ_{RT}) and activation energy (E_a), were obtained from the measurements of temperature dependence of conductivity σ(T). The temperature range used was from 300 to 470 K in steps of 10 K using a slow ramp 5 K/min in a vacuum chamber held at a pressure of 20 mTorr. The current voltage (I-V) characteristic curves were measured using a Keithley Inst. Electrometer model 6517-A.

Table 1. Deposition parameters for a-SixGe1–x:H obtained from LF-PECVD

Process ID	PH_3 (sccm)	SiH_4 (sccm)	GeH_4 (sccm)	H_2 (sccm)	$[P]_{\%GAS}$ (%)
1562-1	0	5	45	1000	0
1582-1	50	5	45	950	1
1582-2	100	5	45	900	2
1582-3	200	5	45	800	4
1582-4	300	5	45	700	6
1582-5	400	5	45	600	8
1582-6	500	5	45	500	10
1582-7	600	5	45	400	12

III. RESULTS AND DISCUSSION

As the starting point for this work, we want to find the best dilution ratio R = Q_{H2}/(Q_{SiH4} + Q_{SiH4}), for the deposition of films of undoped silicon-germanium with the highest conductivity, therefore we used a constant flow of Q_{SiH4} = 5 sccm, and Q_{GeH4} = 45 sccm and the Q_{H2} was varied from 1000 to 4000 sccm for for R values of 10, 20 and 40, the room temperature conductivity σ_{RT} was measured for every R value, and they are shown in Figure 1. From Figure 1 we can see that the highest conductivity is obtained at R = 20, therefore 20 is the R value used in order to study the effect of P on the films.

Following the deposition conditions summarized in Table 1, phosphorous doped films where the flow rate of PH_3 was varied in the range 1-12% in gas phase were deposited. Figure. 2, shows the AFM 3D surface

images of a-Si_xGe_{1-x}:H films and depicts the evolution of surface topography of the samples as the gas-phase phosphorus content was increased from 0% (fig. 2-a) up to 12% (fig. 2-i). As can be seen in fig. 3 the mean roughness (R_a) for doped films reaches its maximum value of 0.64 nm at 4% phosphorous content ($[P]_{\%gas}$), and decreases if the P content increases or decreases from this point as it is depicted in Figure 3. R. Jiménez [11], reported values for R_a in the order of 1 nm for a high R (R = 20) and a deposition pressure range of 0.6-1.2 Torr at 200 °C. While B. Yan [12] reported a value for R_a of 3.2 nm corresponding to films deposited at 2 Torr and 190 °C. Roca [13] reported that the average roughness for a-Si:H deposited at 250 ºC and under RF power of 22 W increased from 1.7 nm to 7.8 nm in the pressure range 0.3 Torr to 2.5 Torr, in contrast, our results show that the R_a values are always below 0.7 nm regardless of the phosphorus concentration, while the high deposition rate is maintained. All of the aforementioned results were for undoped a-SiGe:H and all of them showed that as the pressure increases the average roughness also increases. In this work it is important to notice that the resulting Ra is always less than 0.7 nm and Roca [13] noticed that Ra increases abruptly at deposition pressures above 1 Torr, pressure which corresponds to the onset of the contribution of nanocrystals and clusters to the deposition. The point here is that at the constant pressure of 1.2 Torr, Ra keeps its value always less than 1 nm and, the Ra values also depended on the P content with a Ra decreasing to 0.42 nm at 12% P content. Wang [13] pointed out that Ra decreases as the deposition pressure increases. He explained this behavior saying that the increasing pressure causes aggravated collision between particles and subsequent loss of kinetic energy when these particles reach the growing surface, which is beneficial to suppression of ion bombardment.

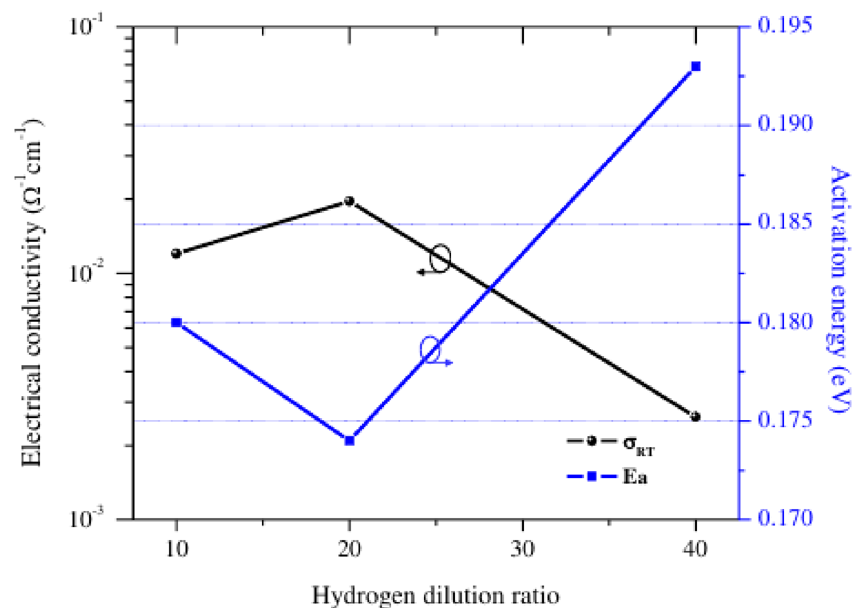

Figure 1. σ_{RT} of the a-Si_xGe_{1-x}:H films as a function of hydrogen dilution ratio R.

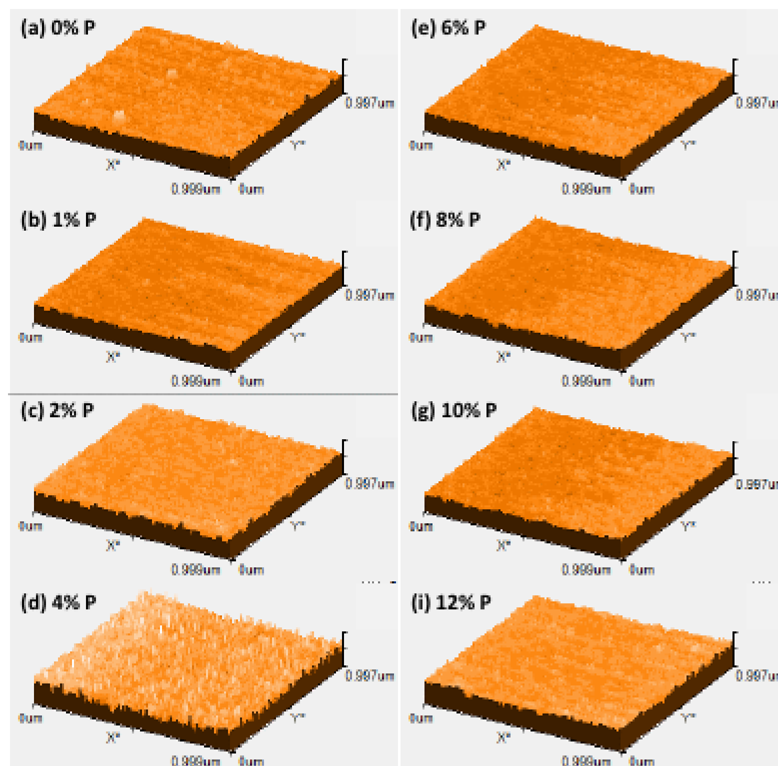

Figure 2. AFM surface 3D images for a-Si$_x$Ge$_{1-x}$:H films as a function of gas-phase phosphorus doping.

There are no current discussions on the effect of P on a-SiGe:H or a-Si:H, the most complete discussion up today is the one in Chapter 5 of the book of R. A. Street [14]. In the book the first thing to be underlined is the dopants catalytic influence on the deposition rate, where gas phase concentration of 1 % Phosphorus doping suppresses the growth rate. The same pattern is observed in the deposition of a-SiGe:H at 1.2 Torr of pressure, but after the initial reduction of the growth rate because of the P, it reaches a minimum at 2% P content in gas phase, there is a slight increase in the deposition rate reaching a maximum at 10% P and again going to the minimum value of 7.5 nm/min at 12% P content in gas phase, all this can be seen in Fig. 4. The conductivity as a function of temperature and P concentration is shown in figure 5 for the range 300 to 463K. From these data, the dark conductivity at room temperature (σ_{RT}) was obtained. The activation energy Ea is derived from a plot of log of conductivity against 1/T that for n-type doping can be interpreted as Ea = $(Ec – Ef)_0$ which denotes the value of (Ec – Ef) extrapolated to T = 0 [15]. Figure 6 shows the σ_{RT} and the Ea as a function of P content in gas phase. In figure 6 we can see the increase in conductivity due to the P incorporation, It reaches a maximum at $[P]_{\%GAS}$ = 4% P and results in σ_{RT} = $1.1x10^{-1}$ Ω-1 cm^{-1}. As the $[P]_{\%GAS}$ increases the conductivity decreases but in a more o less constant value in the range $6.4x10^{-2}$ to $4.3x10^{-2}$ for $[P]_{\%GAS}$ of 6% to 12%.

The doping model developed for Street is based only in results from amorphous semiconductors (Si and Ge), and it states that the defect density increases with the square root of the phosphorus concentration in the gas phase, which will means that the conductivity of the doped films will be reduced in the same ratio as the P content increases. But for our depositing conditions it is not observed in this tendency, instead of it there is, as expected, an increase in conductivity with the incorporation of P at 1% and 2 % in the reactive gases, then jump at 4% at above 5 times the value of the conductivity of the undoped sample and again decreases and remains more or less constant for P content up to 12%. At this point it is pertinent to say that the depositing conditions of high pressure and high dilution rate promote the growth of nanocrystalline material (nc-SiGe:H). The resulting material contains nanocrystals of around 10 nm in diameter as depicted in figure 7, in which the surface of the films undodep (7a) and with 4% p in gas phase (7b) are shown and in 7c, the size of the nanocrystals is demonstrated. In figure 5 we can see two distinct slopes in plots of the data of conductivity and temperature, identifying the two phase structure of the material, the amorphous and crystalline phases which make a dominant contribution to the electrical conductivity of the material in lower and higher temperature respectively [16].

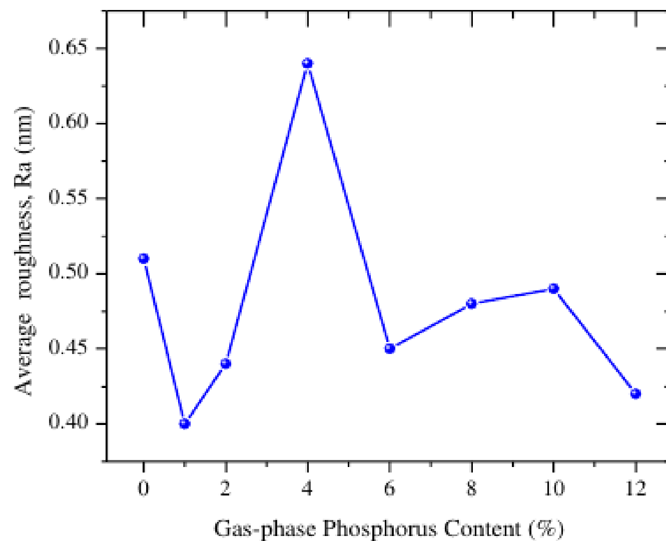

Figure 3. Average surface roughness (Ra) for the a-SixGe$_{1-x}$:H films as a function of gas-phase phosphorus content

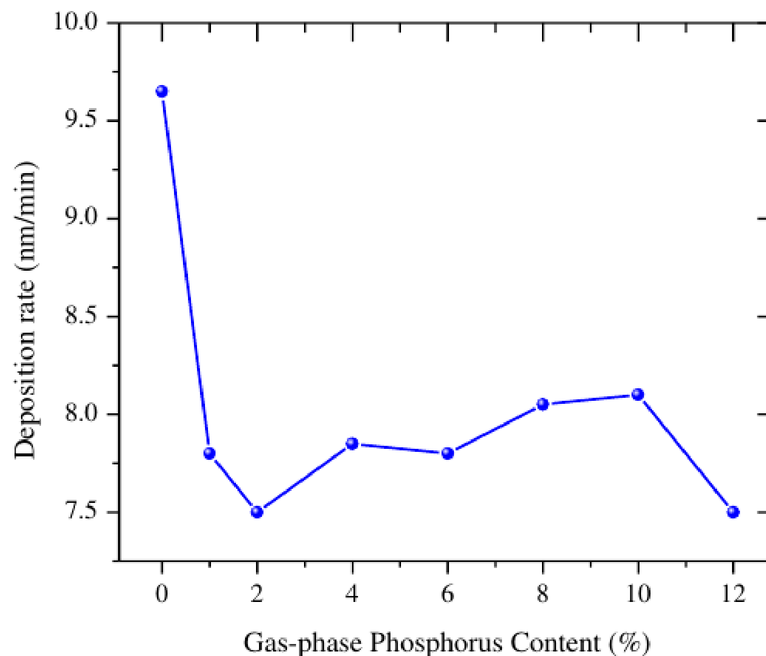

Figure 4. Deposition rates of the a-Si$_x$Ge$_{1-x}$:H films vs. the variation of [P]$_{GAS}$%.

There is no observation of a continuous dropping in the doping efficiency for P content above 1% in gas phase as explained by Street, instead of these, the activation energy and as a consequence, the Fermi level remain almost constant for P content above 4% in gas phase as can be seen in Fig. 4. This fact has been already noticed by Stutzmann [17] which noticed that arsenic in a-Si:H and phosphorus in a-Ge:H show a constant doping efficiency. Overhof [18], based on the work of Hauschildt [19] observed that the spin density for the sates at midgap is remarkably higher than in the case of a-Si:H and that these spin densities levels can be interpreted in terms of a rather broad density of states peak that does not change with doping. This high "intrinsic" density of dangling bonds leads to a high defect density of states in the gap and explains the result that for a major shift of the Fermi level a relatively high doping level is needed. In our case and under the depositing conditions already mentioned, we needed more than 1% of P to obtain a substantial shift on E_F. In other words, the doping model of a-Ge:H explains the behavior of P doping nc-SiGe:H, but the very high doping

level needed to see this behavior, suggest that depositing conditions have much more influence in doping mechanisms and in the generation of dangling bonds and that all of this affect the structure of the material.

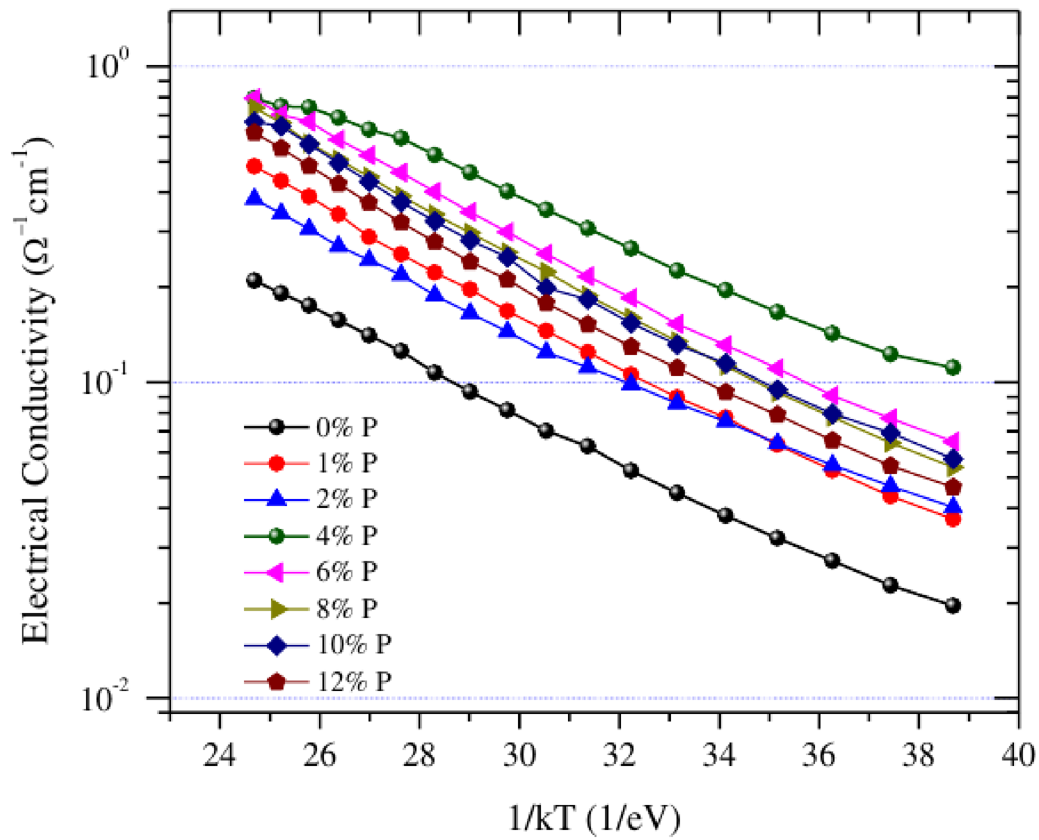

Figure 5 Temperature dependence of conductivity of the a-Six Ge1–x:H films vs. varying [P]GAS%.

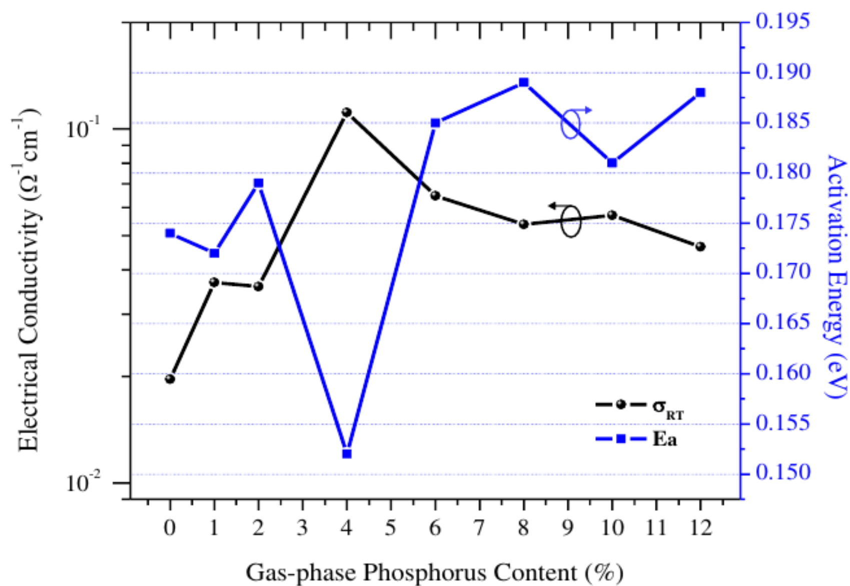

Figure 6. The room-temperature conductivity (σ_{RT}) of the a-Si_xGe_{1-x}:H films as a function of $[P]_{GAS\%}$.Fig. 7

FTIR analysis was performed on all the samples and the result is summarized in figure 8. Let us concentrate on the region 480 to 700 cm^{-1}, 480 cm^{-1} corresponds to the weak Si-Si bond, 560 and 640 cm^{-1} are for the bending mode GeH and SiH bonds. We can see the evolution of this region as the P content increases. If we take as reference the area of this region at 0%P, we can see that for 1%P the area is more or less the same, at 2%P this area seems to reduce a bit with respect to 0%P, but at 4%P the area increases and this is very noticeable, also is pertinent to note the peak belonging to the weak Si-Si bond at 4%, it disappears. The area at 4%P is the largest of all, but all the other from 6 to 12%P are larger than that at 0%P. For 1, 2, 8, 10 and 12%P, the peak at 480 cm^{-1} is barely noticed. For 4%P the 480 peak disappears, but for 6%P this peak is the largest of all the series. The absorption peak at 1880 corresponds to the GeH bond in stretching mode, if again we compare the area under this peak, we can see how it clearly reduces as the P content increases. For all of the aforementioned we can say that the P incorporation reduces the concentration of weak Si-Si bonds and enhances the presence of Ge-H and Si-H bonds. Let us remind that the high conductivity obtained for 0%P was produced for the depositing conditions and the nanocrystal formation. It is very well known that the nanocrystals help to reduce the weak Si-Si bonds, but the addition of P seems also to behave as a catalyst for this action. In other words, the P doping enhances the change in the density of the weak Si-Si bonds and, depending on its concentration it may decrease (as for 1,4, 8, 10 and 12%P) or increase (like in 2, 6 and 12%P). We may say that the P content increases the GeH and SiH bonds in their bending mode and reduces the density of the GeH bonds in stretching mode.

Now we return to figure 6 and in that we can see that from 6 to 12%P E_F remains in the range 0.18 to 0.19 eV, which result in agree with the observation of Stutzmann [17] that for the doping of P in a-Ge:H the doping efficiency remains constant and, that as the E_F moves into either band tails weak bonds loose their bonding electrons, but dangling bond formation manifest as only a secondary effect of the doping process. All this behavior is in accordance with that showed for a-Ge:H [19], and the conductivity of nc-SiGe:H at 4% P is at least 1 order of magnitude larger than that of p doped a-Ge:H regardless of the Si incorporation. The presence of nanocrystlas and the high Phosphorous doping improve the electrical and optical characteristics of the material.

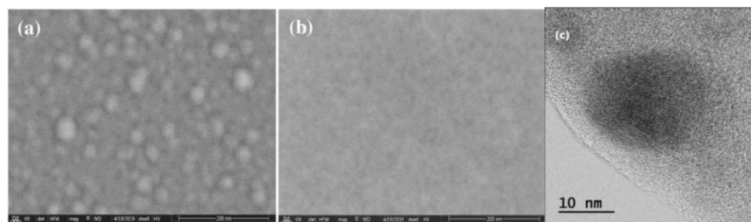

Figure 7. SEM micrographs for top view of the a-Si_xGe_{1-x}:H films for different P-content: (a) 0% P, and (b) 4% P, (c) nanocrystal in the undoped a-Si_xGe_{1-x}:H films.

IV. CONCLUSION

The depositing conditions for nc-SiGe:H in a low frequency plasma reactor and 1.2 Torr deposition pressure have been studied and optimized for the dilution ratio $R = Q_{H2}/(Q_{SiH4} + Q_{SiH4})$, and R = 20 resulted the best ratio for depositing undoped nc-SiGe:H. These conditions were used to dope the material with P and very high concentrations ranging from 1 to 12% P gas were studied. The highest conductivity was obtained a 4% P gas which at room temperature has a value of 0.11 $\Omega^{-1}cm^{-1}$, this value is one order of magnitude higher that of a-Ge:H, material that has smaller band gap. The P incorporation reduces the concentration of weak Si-Si bonds and enhances the presence of Ge-H and Si-H bonds. At 4% P gas the nano crystals have an average size of 17 nm. The nc-SiGe:H obtained under the depositing conditions here described resulted in a material with superior conductivity and endurance to high energy illumination which makes it ideal for applications in flexible substrates for TFT source and drain contacts and for thin film solar cell emitter.

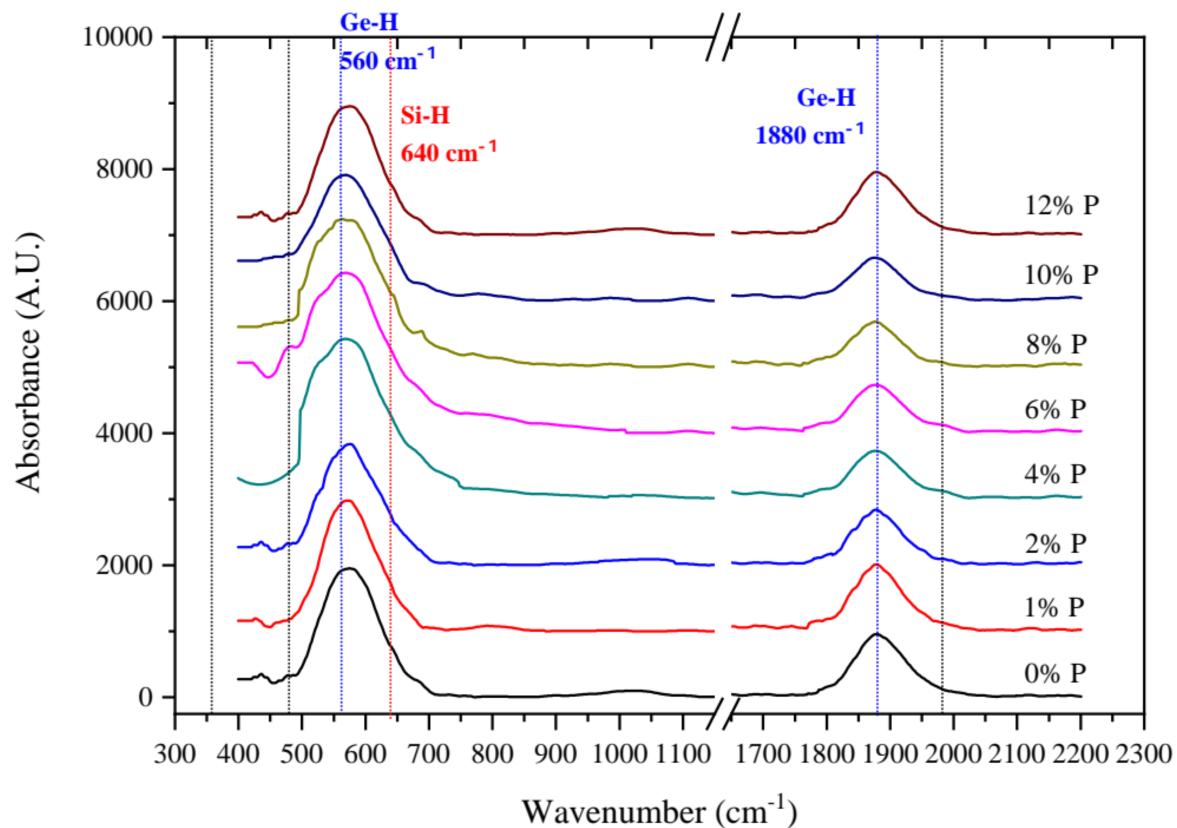

Figure 8.FTIR spectra of the a-SixGe 1–x:H

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