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ORIGINAL ARTICLE

# Structural and optical study of indium and gallium arsenide nanostructures prepared by magnetron sputtering

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## Abstract

Currently, the obtention of nano-structures based on III-V materials is expensive. This calls for novel and inexpensive nanostructure manufacturing approaches. In this work we report on the manufacture of a nanostructures consisting of alternating layers of In and GaAs on a Si substrate by magnetron sputtering. Furthermore, we characterized the produced nanostructures using secondary ion mass spectroscopy (SIMS), X-ray diffraction analysis, and Raman spectroscopy. SIMS revealed variation in the concentration of In atoms across In/GaAs/In interphases, and X-ray diffraction revealed planes corresponding to phases associated with GaAs and InAs due to In interfacial diffusion across GaAs layers. Finally, in order to study the composition and cristal quality of the manufactures nanostaructures, Raman spectra were taken using laser excitation lines of 452 nm, 532 nm, and 562 nm at different points across the nanostructures. This allowed to determine the transverse and longitudinal optical modes of GaAs and InAs, characteristic of a two-mode behavior. An acoustic longitudinal vibrational mode LA(Γ) of GaAs and an acoustic longitudinal mode activated by disorder (DALA) were observed. These resulted from the substitution of Ga atoms for In atoms in high concentrations due to the generation of Ga(VGa) and/or As(VAs) vacancies. This set of analyses show that magnetron sputtering can be a viable and relatively low-cost technique to obtain this type of semiconductors.

**Keywords:** III-V semiconductors; Raman spectroscopy; SIMS; X-ray.

## Introduction

The development of III-V nanostructure layers by epitaxial growth technologies has prompted the production of materials with functional properties suitable to develop optoelectronic devices [1-8] and more efficient solar cells than those traditionally manufactured with semiconductors such as germanium (Ge) and silicon (Si) [9-12]. Currently, the preparation of these nanostructures is costly, so less expensive preparation methods,

such as magnetron sputtering, are being explored. The aim of these nanostructure preparation alternatives is to obtain good structural quality III-V semiconductors compatible with traditional semiconductor technology [13-16]. The success of these efforts will translate in the production of low-cost optical devices, compared with some widely used epitaxial techniques, such as molecular-beam epitaxy (MBE) [17-23].

Among the III-V semiconductors alloys containing elements such as gallium (G_), arsenic (As), indium (In), among others, are of interest because the controlled introduction of impurities in the semiconductor matrix generates stoichiometry-dependent changes that vary its optical and electrical properties. For instance, gallium arsenate (GaAs) doped with indium (leading to InGaAs) has the possibility of obtaining bandgaps from 0.36 eV (corresponding to InAs) up to 1.44 eV (corresponding to GaAs) [24, 25]. InGaAs has been synthesized using physical and chemical methods such as metal-organic chemical vapor deposition (MOCVD) and/or molecular-beam epitaxy [26, 27], in which complex procedures and special experimental conditions are required [28, 29]. Another functional technique for the preparation of this type of alloys is magnetic field-assisted cathodic sputtering, known as or magnetron sputtering. With this technique semiconductor layers can be deposited on monocrystalline, polycrystalline, and/or amorphous substrates. However, reports on the use of this technique for the growth of polycrystalline III-V semiconductors are scarce [30, 31].

In this paper, the results of the manufacture of alternating layers of indium and GaAs on a silicon substrate (100) by magnetron sputtering, and their characterization by secondary ion mass spectroscopy (SIMS), X-ray diffraction, and Raman spectroscopy are reported. SIMS is a surface material characterization technique, capable of detecting surface impurities in concentrations below one part per million and in bulk (of the order of one part per billion), with excellent depth resolution (~10 nm). SIMS also has the capacity to directly provide composition vs. depth profiles in real space, making this approach appropriate to characterize thin films, multilayers, and heterostructures, such as those of the present work [32]. To study InGaAs ternary formation by indium interfacial diffusion in GaAs layers, several composition spectra were obtained at given points across the structure. Finally, the structural characterization was performed by means of X-rays and Raman spectroscopy using three excitation lines.

## Material and methods

High-purity (95.5 %) indium and GaAs targets (100) were used to obtain the samples. The employed silicon substrates (100) were first degreased with acetone and methanol. Next, they were cleaned with 2 % hydrofluoric

acid, rinsed with deionized water, dried with nitrogen, and introduced into the magnetron sputtering chamber. Then, the intended nanostructure was constructed as follows: firstly, a GaAs layer was deposited, operating the GaAs target for 30 minutes. Subsequently, the GaAs-associated shutter was closed, and the substrate's temperature was lowered to 300 °C. Upon reaching this temperature, the indium target source was turned on for a given time (td = 5, 10 and 15 minutes for each of the samples of interest, M1-M3). This process was repeated opening and closing the shutters, one at a time, until three GaAs/In/GaAs/In/GaAs nanostructures were formed. The shutters of the two targets do not completely close the targets, allowing indium atoms and GaAs molecules to escape. The employed experimental conditions are shown in **Table 1**.

Secondary ion mass spectroscopy measurements were performed in a TOF-SIMS-5 reflection anlyzer (ION-TOF, Germany) consisting of a 5 keV primary energy beam with an approximate angle of 45 °with respect to the sample and a double-focusing mass spectrometer equipped with a photomultiplier. X-ray diffraction measurements were made in a Rigaku miniflex equipment (Rigaku, Japan) in a range of $20° \leq 2\theta \leq 60°$. Raman spectra were taken with a N8 NEOS SENTERRA Bruker equipment (Bruker, USA) that combines a SENTERRA Raman spectrometer with a Nano's N8 NEOS atomic force microscope.

## Results and Discussion

### Secondary ion mass spectroscopy

The constructed Indium-GaAs interfaces (samples M1-M3) were analyzed by determining the distribution of indium and GaAs in the layers by concentration-depth spectra with secondary ion mass spectroscopy (SIMS). This made it possible to accurately infer the shape of indium/GaAs structures [33]. Component concentrations, as functions of the thickness in the M1 sample, are shown in **Fig. 1a**. The signal's amplitude was normalized to the unit to identify the relative contrast of the ions throughout the sample. The curves corresponding to gallium and arsenic showed periodic modulation and revealed layer formation with not very well-defined interfaces in the M1 sample. In this spectrum, a less significant change in the intensity of indium was observed; this was due to indium diffusion in the GaAs layers. In the case of the M2 and M3 samples, a similar behavior is observed in which the amplitude of the oscillations specific to each structure changed. This was due to the variation in the preparation times (and/or thickness) of the samples.

Indium diffusion in the SIMS depth profiles is in accordance with X-ray diffraction (addressed in the following section). Different phases (indium, GaAs, InAs, and InGaAs) were identified in the structures. For instance,

**Table 1.** Experimental conditions.

| Parameter | Value |
|---|---|
| Base pressure (Torr) | $1.0 \times 10^{-6}$ |
| Work pressure (Torr) | $1.0 \times 10^{-3}$ |
| Distance target-substrate (cm) | 5.0 |
| Deposit temperature of the GaAs layer (°C) | 580 |
| Deposit temperature of the indium layer (°C) | 300 |

indium diffused in the GaAs lattice by substituting gallium at different concentrations across the structure, as schematized in Fig. 1b. This is likely due to a greater mobility of indium atoms at the time of sample preparation. In the first cycle of sample preparation, less distributed indium atoms, led to the formation of an InGaAs ternary semiconductor. However, near the surface, where the concentration of indium was higher, the formation of InAs clusters was more likely.

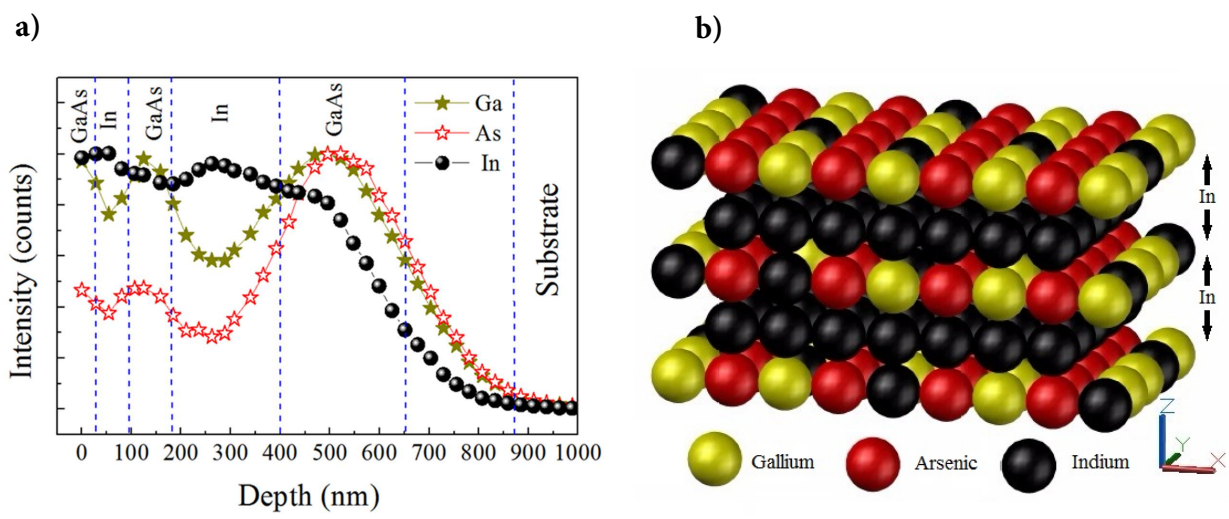


**Figure 1. a)** SIMS spectrum as a function of depth for the M1 interphase. **b)** Schematic of the diffusion of elements in the M3 interphase.

The nature of the Ga-As and In-As bond formation and the composition of the InGaAs ternary structure is the result of bonding events in the interface at the time of sample preparation. In order to corroborate this combination of phases throughout the structure, different Raman experiments were carried out and their outcomes described later.

## X-ray

The X-ray diffraction spectra for samples M1-3, shown in **Fig. 2**, correspond to deposition of indium layers at 5, 10, and 15 minutes at the end of each structure, respectively. For the analysis, the spectra were normalized with respect to the intensity of the substrate plane located at $2\theta \approx 32.2\,°$. In each of the spectra, the crystallographic planes corresponding to the indium tetragonal phase, coming from the indium intermediate layers were identified on the background. Similarly, in positions $2\theta \approx 27.5\,°$, $46.6\,°$, $53.8\,°$, and $55.5\,°$planes with crystallographic directions 111, 202, 311, and 222 were observed. In addition, the following InAs crystallographic planes were identified: 111, 202, 311, and 222. These crystallographic planes were located at $2\theta \approx 25\,°$, $41.6\,°$, $49.3\,°$, and $50.6\,°$, respectively. Finally, a signal of low intensity was identified between the peaks associated with InAs and GaAs binary semiconductors, possibly due to an InGaAs ternary formation by interfacial diffusion of indium into the GaAs layer.

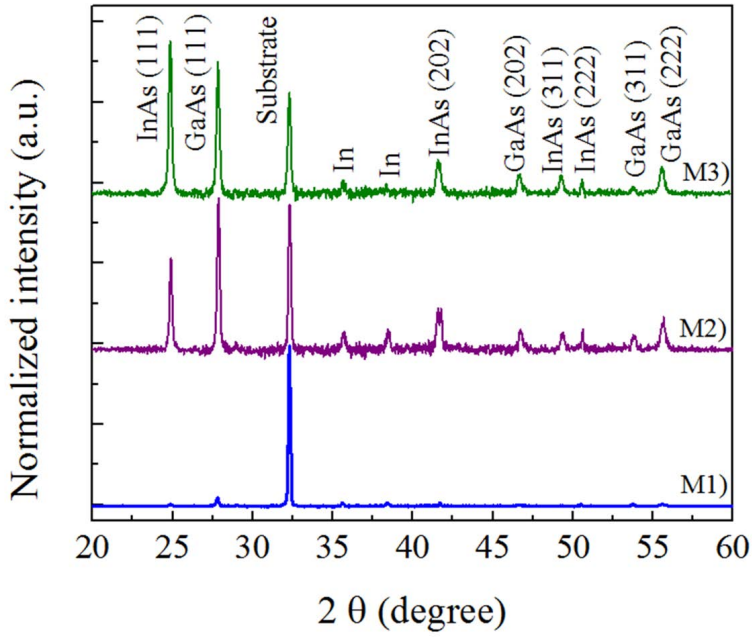


**Figure 2.** X-ray diffractograms for M1-M3 structures.

As observed in the diffractograms of Fig. 2, the integrated intensities of the $I_{InAs}/I_{GaAs}$ ratios increased. These ratios were calculated on the InAs and GaAs plane 111, obtaining values of 0.83, 1.72, and 3.37, across M1 to M3, respectively. These observed ratios revealed an increase of the InAs phase with respect to the GaAs phase due to a surge in the deposition times and/or thickness of the indium intermediate layer. The plane at $2\theta = 32.2\,°$corresponds to the Si substrate (100).

## Raman

To analyze the influence of the indium intermediate layer on vibrational modes, Raman spectra were taken on the layer's surface and in a cross section along the breadth of the structure. The Raman spectra taken on the layer's surface with laser excitation lines of 452 nm, 532 nm, and 652 nm are indicated in **Fig. 3** and **Fig. 4**. Raman spectra revealed the following: a) A change in the Raman line shape due to an increase in the penetration depth of each laser line; b) A two-mode behavior, typical of III-V alloys, i.e. independent TO and LO phononic modes associated with GaAs and InAs [34-36]. c) All the spectra show a shift of approximately 5 $cm^{-1}$ towards low frequencies with respect to the vibrational modes of GaAs and InAs in bulk, due to the stresses generated in the interfaces during the preparation of the layers. d) A change in integrated intensities and in the width at half of its maximum value (FWHM: full width at half maximum) in InAs and GaAs vibrational modes. These are attributed to a change in the percentage of indium incorporated in GaAs, for interfacial diffusion purposes. Additionally, an LA(L) mode is evidenced in approximately 195 $cm^{-1}$, possibly due to a structural disorder. Vibrational modes below 200 $cm^{-1}$ correspond to the activation of the longitudinal acoustic mode (disorder activated longitudinal acoustic-DALA), this being the most evident for longer indium deposition times, indicating a greater structural disorder.

In order to make a more detailed analysis of the Raman spectra taken with different excitation lengths, the penetration depth ($\delta$) of each laser line was calculated using the formula $\delta = \lambda/4\pi k$, where $k$ (extinction coefficient) corresponds to the imaginary part of the complex refractive index $n = n + ik$ [37]. The values obtained, considering indium diffusion in the GaAs layer, are shown in **Table 2**.

When the M1, M2, and M3 samples were excited with the 452 nm laser (**Fig. 3a**), the Raman signal comes from a region that is very close to the surface of the sample ($\delta = 81$ nm). This is the reason why the spectra are noisy and not very well-defined. However, the vibrational modes of GaAs ($\omega_{TO} = 263$ $cm^{-1}$) and InAs ($\omega_{TO} = 218.9$ $cm^{-1}$, $\omega_{LO} = 219.2$ $cm^{-1}$) were identified. The observed vibrational modes for InAs are caused by the presence of indium in the last

**Table 2.** Wavelength, excitation energy and penetration length.

| λ (nm) | E (eV) | δ (nm) |
|---|---|---|
| 452 | 2.54 | 81 |
| 532 | 2.33 | 132 |
| 652 | 1.9 | 251 |

indium-and-GaAs layers of the nanostructure, resulting in InGaAs formation. The vibrational mode about 400 $cm^{-1}$, labeled as IF, comes from electronic transitions.

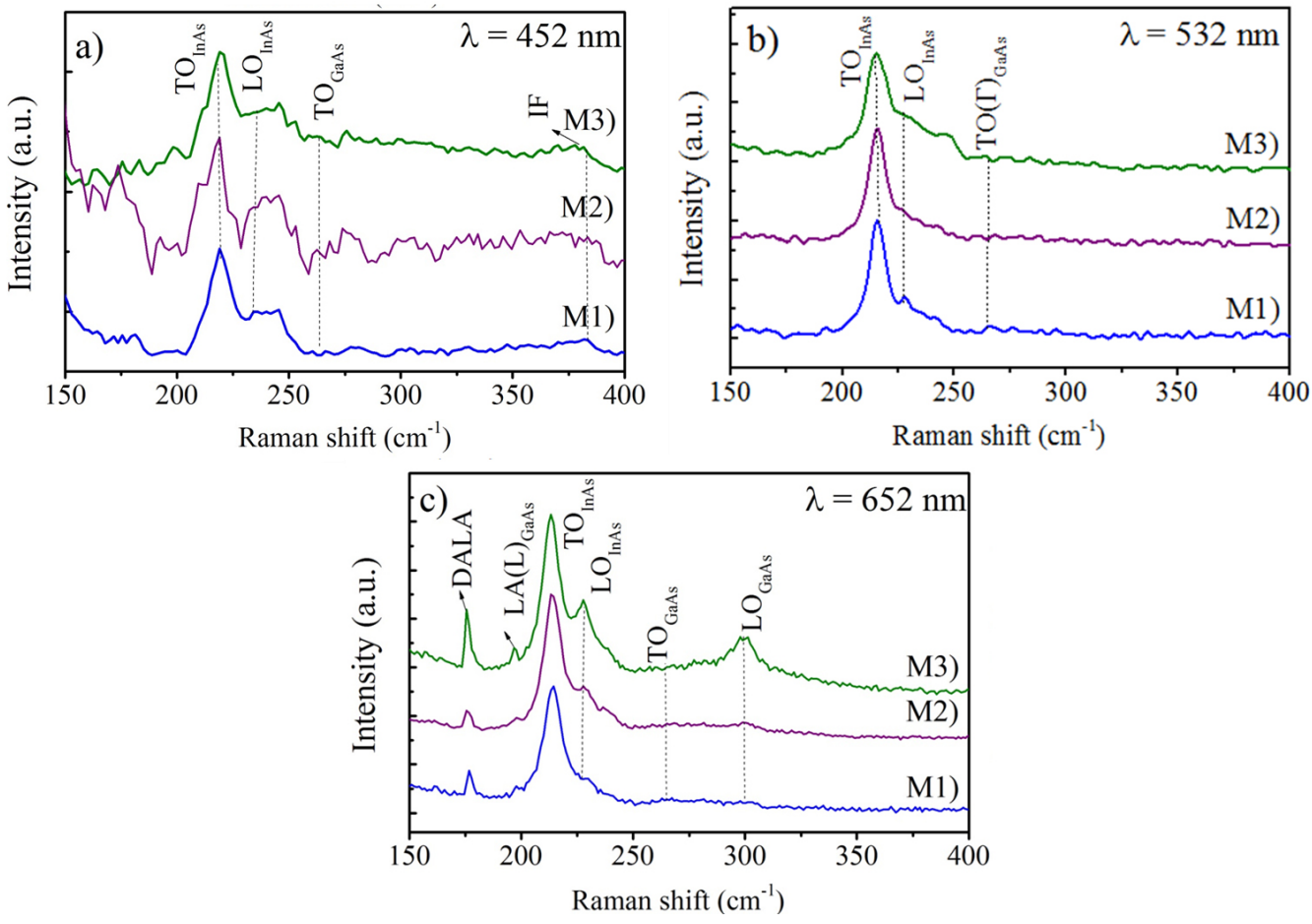


**Figure 3.** Raman spectra of the GaAs/In layers under excitation of **a)** 452 nm, **b)** 532 nm, and **c)** 652 nm.

When the samples are excited with the 532 nm line (Fig. 3b), the Raman signal is more defined because it comes from a depth of $\delta = 132$ nm, and/or a higher volume of Raman excitation ($I_{Raman} \propto Vol$). In this case, the TO and LO modes of GaAs and InAs are of greater intensity and have a width at half the maximum, which is lower than in the previous case of the excited sample at 452 nm.

When the samples were excited with lines of a longer wavelength ($\lambda = 652$ nm) and/or penetration $\delta = 252$ nm (Fig. 3c), the Raman signal comes from the proximity of the interface between the second indium and GaAs layer, according to the SIMS concentration profile spectrum. In addition, from the characteristic GaAs phononic modes ($\omega_{LO} = 300$ cm$^{-1}$), a vibrational longitudinal acoustic LA mode ($\Gamma$) of GaAs ($\omega_{LA}$ ($\Gamma$) $= 198$ cm$^{-1}$) and an acoustic longitudinal mode appeared activated by disorder (DALA) ($\omega_{DALA} = 175$ cm$^{-1}$). This is due to the generation of Ga ($V_{Ga}$) and/or As ($V_{As}$) vacancies, whose intensity is greater when the deposition time of the In intermediate layer is 15 min.

To analyze the compositional and morphological homogeneity of the indium and GaAs layers, Raman spectroscopy measurements were carried out in a cross section, in areas close to the In and GaAs interface and with the highest probability of GaAs content. The Raman spectra taken with a laser line of $\lambda = 652$ nm at three different depths are shown in **Fig. 4**. The three depths were: near the surface (Fig. 4a), at 250 nm (Fig. 4b), and 500 nm from the surface (Fig. 4c). These three spectra had similar shapes, but the relative intensities among their phononic modes differed, possibly due to variation of indium concentration within the structure, favoring the formation of Ga-In and/or the InGaAs ternary bonds.

Then, the set of Raman results allows to corroborate indium atoms diffusion in the GaAs layers, as revealed by SIMS and X-ray analyses. Additionally, although the technique for obtaining material used in this work is different from those usually reported, the diffusion kinetics of indium atoms in GaAs can be related to substrate temperature at the time of sample obtention or to the heat exerted on this type of semiconductors [38]. At a characteristic activation temperature or energy, the atoms can have a sufficient mobility favoring surface and volume diffusion. Another factor strongly related to this diffusion mechanism is the minimization of the elastic energy due to atoms reaching the surface and diffusing; implying a variation in the composition [39], which is a consequence of the employed sample obtention method.

Consequently, our results suggest the possibility of obtaining InGaAs/GaAs-type structures through the magnetron sputtering technique using the method previously exposed under controlled experimental conditions. This permits to have control on lattice defects and concentration

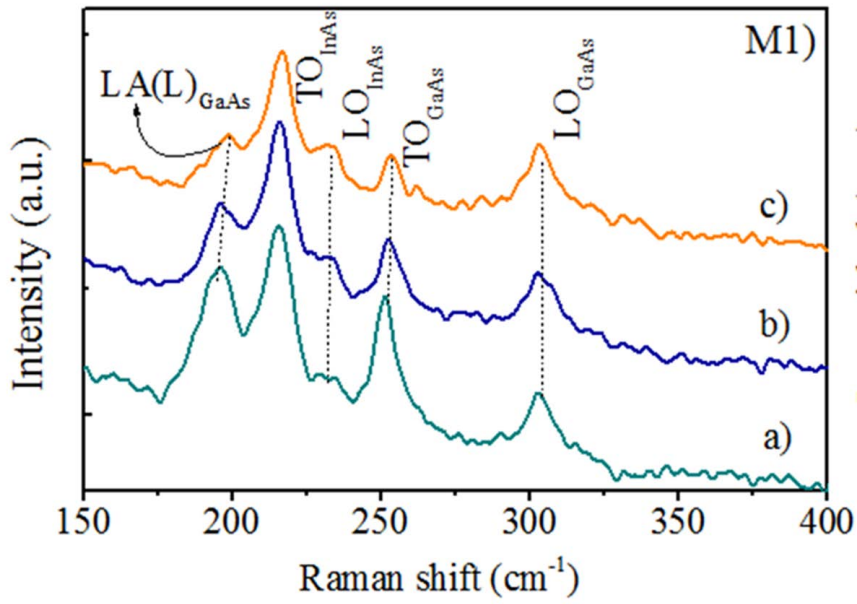


**Figure 4.** Raman spectra taken in cross section along the structure: **a)** near the surface, **b)** at 250 nm, and **c)** at 500 nm measured from the surface.

gradients among layers. Our approach becomes an alternative among the methods used by magnetron sputtering to control the atoms incorporated in the GaAs lattice.

## Conclusions

GaAs/In/GaAs/In/GaAs III-V semiconductor structures were obtained on silicon substrates (100) by a low-cost technique such as magnetron sputtering. The diffusion behavior of the atoms at the interfaces of the alternating GaAs and indium layers was determined from optical, compositional, and structural results. All the samples responded to the diffusion mechanism, which depends on the indium layer deposition time.

Raman analyses led to establishing LO and TO combined vibrational modes, which are characteristic of the substitution of indium atoms by gallium atoms. This corroborates the formation of the InGaAs ternary in some regions of the samples. Alternating semiconductor-metal layers (GaAs/In in this case), resulting in the formation of materials of interest.

Finally, the Magnetron sputtering method can be used for the preparation of semiconductor alloys with different species of metallic elements and

semiconductors, expanding the research spectrum for polycrystalline III-V semiconductor materials.

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## Conflict of interest

The authors declare having no conflict of interest

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## Estudio estructural y óptico de nanoestructuras de arseniuro de indio y galio preparadas por pulverización catódica por magnetrón

**Resumen:** Actualmente, la obtención de nanoestructuras basadas en materiales tipo III-V es costosa. Para ello se requieren enfoques de fabricación de nanoestructuras novedosos y económicos. En este trabajo, presentamos resultados sobre la fabricación de nanoestructuras que consisten en capas alternas de In y GaAs en un sustrato de Si mediante pulverización catódica con magnetrón. Además, caracterizamos las nanoestructuras producidas utilizando espectroscopía de masas de iones secundarios (SIMS), análisis de difracción de rayos X y espectroscopía Raman. La SIMS reveló variación en la concentración de átomos de In en las interfases de In/GaAs/In, y la difracción de rayos X reveló planos correspondientes a fases asociadas con GaAs e InAs debido a la difusión interfacial de a través de capas de GaAs. Finalmente, para estudiar la composición y la calidad de los cristales de las nanoestructuras fabricadas, se tomaron espectros Raman utilizando líneas de excitación láser de 452 nm, 532 nm y 562 nm en diferentes puntos de las nanoestructuras. Esto permitió determinar los modos ópticos transversales y longitudinales de GaAs y InAs, característicos de un comportamiento de dos modos. Se observó un modo vibratorio longitudinal acústico LA(Γ) de GaAs y un modo longitudinal acústico activado por desorden (DALA). Estos modos resultaron de la sustitución de átomos de Ga por átomos de In en altas concentraciones debido a la generación de vacantes de Ga (VGa) y/o As (VA). Estos análisis muestran que la pulverización catódica por magnetrón puede ser una técnica viable y de costo relativamente bajo para obtener este tipo de semiconductores.

**Palabras clave:** semiconductores ipo III-V; espectroscopía Raman; SIMS; rayos X.

## Estudo estrutural e óptico de nanoestruturas de índio e arseneto de gálio, preparadas por pulverização catódica por magnetron

**Resumo:** Atualmente, a obtenção de nanoestruturas baseadas em materiais do tipo III-V é cara. Isso exige abordagens de fabricação de nanoestruturas inovadoras e econômicas. Neste trabalho, apresentamos resultados sobre fabricação de nanoestruturas que consistem em camadas alternadas de In e GaAs em um substrato de Si por pulverização por magnetron. Além disso, caracterizamos as nanoestruturas produzidas usando espectroscopia de massa de íons secundários (SIMS), análise de difração de raios-X e espectroscopia Raman. O SIMS revelou variação na concentração de átomos de In nas interfases In/GaAs/In e a difração de raios X revelou planos correspondentes a fases associadas a GaAs e InAs devido à difusão interfacial de In através de camadas de GaAs. Finalmente, a fim de estudar a composição e a qualidade dos cristais das nanoestruturas fabricadas, os espectros Raman foram obtidos usando linhas de excitação a laser de 452 nm, 532 nm e 562 nm em diferentes pontos das nanoestruturas. Isso permitiu determinar os modos ópticos transversal e longitudinal de GaAs e InAs, característicos de um comportamento de dois modos. Foram observados um modo vibracional longitudinal acústico LA(Γ) de GaAs e um modo longitudinal acústico ativado por distúrbio (DALA). Esses modos resultaram da substituição de átomos de Ga por átomos de In em altas concentrações devido à geração de vagas de Ga (VGa) e/ou As (VAs). Essas análises mostram que a pulverização catódica por magnetron pode ser uma técnica viável e de custo relativamente baixo para obter este tipo de semicondutores.

**Palavras-chave:** semicondutores tipo III-V; espectroscopia Raman; SIMS; raios-X.

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