Semiconductor Physics, Quantum Electronics & Optoelectronics, 2025. V. 28, No 4. P. 394-399.

Semiconductor physics

Raman study of thin $TlInS_2$ films prepared by thermal evaporation

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Abstract. Thin (10…200 nm) $TlInS_2$ films are prepared by thermal evaporation on silicon and silicate glass substrates. Micro-Raman spectra measured at a rather moderate excitation (532 nm, 4 kW/cm^2) confirm amorphous character of the films. Narrow features emerging in the spectra at a higher excitation power density (40 kW/cm^2) testify crystallization of $TlInS_2$ on the film surface. The formation of crystallites is explained by thermal effect of the absorbed laser light. The film surface is shown to be heated above 150 °C by the 40 kW/cm^2 laser beam.

Keywords: amorphous films, Raman spectroscopy, crystallization, thermal treatment.

https://doi.org/10.15407/spqeo28.04.394
PACS 78.30.Ly

Manuscript received 03.03.25; revised version received 10.10.25; accepted for publication 26.11.25; published online 15.12.25.

1. Introduction

Crystalline $TlInS_2$ is a well-known semiconductor material with highly anisotropic layered structure. At low temperatures, it possesses ferroelectric properties and at 190…220 K, it exhibits an incommensurate phase [1–3]. It has been extensively studied [1–9] due to its promising applications in optoelectronics caused by its high photosensitivity in the visible spectral range, high birefringence, and broad transparency range as well as good thermoelectric characteristics and demonstration of photoinduced phenomena enabling memory effects [2, 10]. $TlInS_2$ is known to crystallize in various structures and polytypes, the most typical and extensively studied being the ones described by 6 C2h [4, 5] and 18 D4h [4] space groups. Important information on the crystal structure and lattice dynamics of $TlInS_2$ can be provided by Raman spectroscopy [4, 5, 8, 9, 11 and references therein].

Recent years demonstrate a growing interest of researchers to $TlInS_2$ thin films [12–15], similarly to the films of other I–III–VI_2 type materials mostly prepared by thermal evaporation [16–22]. Here, we report on a Raman spectroscopy study of thermally evaporated $TlInS_2$ thin films.

2. Experimental

$TlInS_2$ films were obtained by thermal evaporation of pre-synthesized crystalline $TlInS_2$ (the synthesis details are described in an earlier study [11]) onto silicate glass and crystalline Si substrates kept at room temperature. The evaporation temperature was close to 1050 K, the evaporation rate was 4 nm/s, and the pressure was ~$2\cdot10^{-4}$ Pa. The thickness d of the thicker films was evaluated by interferometry. The thickness of the thinner films was obtained by interpolation taking into account the evaporation time and was estimated to range from 10 to 200 nm. No post-preparation annealing of the films was performed.

Micro-Raman scattering measurements were performed at room temperature using a XPloRa Plus spectrometer (Horiba) equipped with a cooled CCD camera. Excitation was provided by a solid-state laser (λ_{exc} = 532 nm). The instrumental resolution was better than 2.5 cm^{-1}.

3. Results and discussion

Raman spectra of the $TlInS_2$ films on Si and silicate glass substrates measured at low excitation power density (P_{exc} = 4 kW/cm^2) are shown in Fig. 1. Broad maxima in the spectra (compared to those reported for single-crystalline $TlInS_2$ [9, 11]) are typical for non-crystalline materials, thereby characterizing the films as amorphous. The spectral position of the asymmetric broad maximum at 270…290 cm^{-1} for different samples correlates with the known Raman features of crystalline $TlInS_2$ corresponding to internal vibrations of In_4S_6 units in the $TlInS_2$ structure [5]. Evidently, one would hardly expect the existence of In_4S_6 units in amorphous $TlInS_2$ films. However, it is quite reasonable to assume that these maxima for the films with different thicknesses correspond to In–S bond vibrations, while broad lower-frequency

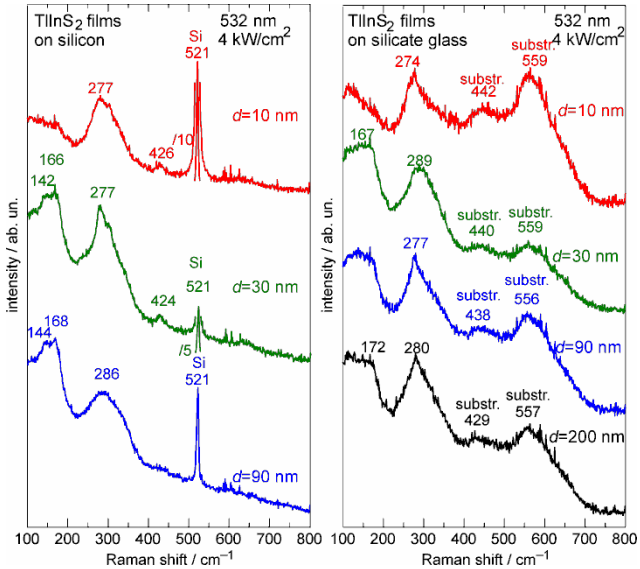

Fig. 1. Micro-Raman spectra of $TlInS_2$ films of different thicknesses d thermally evaporated on silicon and silicate glass substrates. The spectra were measured at the excitation with $\lambda_{exc} = 532$ nm and $P_{exc} = 4$ kW/cm^2.

features in the range of 140…175 cm^{-1} most likely origin from vibrations of Tl–S bonds. A weak maximum near 425 cm^{-1}, which is observed only for the films on Si substrates and is masked by the scattering from the substrate for the films on silicate glass, has not been reported for crystalline $TlInS_2$ [5, 9, 11]. Most likely, it is related to vibrations of homopolar S–S bonds in the films. The maxima in the spectra of the films of different thicknesses are similar. As expected, the thinner the films, the higher are the intensities of the substrate-related maxima in the observed Raman spectra (Fig. 1).

It should be noted that the spectra of the films shown in Fig. 1 drastically differ from those of thermally evaporated $TlInS_2$ films reported in a recent study [15], where the spectral features are more distinct and resemble those of crystalline $TlInS_2$ [5, 11]. Crystallinity of the $TlInS_2$ films from Ref. [15] (or at least presence of crystalline $TlInS_2$ phase in the films) is also supported by X-ray diffraction data showing narrow reflexes typical for $TlInS_2$. The reason why in our case, contrary to the study [15], thermal evaporation resulted in amorphous $TlInS_2$ films, is evidently related to the evaporation procedure. As follows from our recent study of $TlInSe_2$ films prepared by a similar procedure [23], the substrate temperature can play a crucial role. Namely, evaporation of $TlInSe_2$ onto substrates preheated to 300 °C resulted in polycrystalline films [19, 20], while evaporation onto non-preheated substrates led to formation of amorphous $TlInSe_2$ films [23]. In our case, evaporation of $TlInS_2$ films was performed onto cold substrates without any preheating. Meanwhile, the details of the evaporation procedure are described in a rather contradictory manner in Ref. [15]. Therefore, we were not able to find sufficient information to identify particular factors responsible for fabrication of amorphous or polycrystalline $TlInS_2$ films.

Other studies of $TlInS_2$ films reported earlier did not include Raman spectroscopy. However, the reported XRD data clearly confirmed the film polycrystalline nature [13]. The authors of [14] do not discuss the structure of the obtained $TlInS_2$ films, nor do they give the details of the substrate temperature during evaporation. Meanwhile, in an earlier study [12] claiming the films to be amorphous, the same films not subjected to any additional treatment were simultaneously stated to be crystallized in tetragonal, monoclinic, and rhombic structures. This, without any Raman or XRD data provided, makes the study [12] self-contradictory. Therefore, to the best of our knowledge, the present paper is the first one reporting on fabrication and study of amorphous $TlInS_2$ films.

Comparison of the measured Raman spectra of the $TlInS_2$ films (Fig. 1) with those of bulk $TlInS_2$ crystals (Fig. 2) shows that the positions of the intense broad maxima in the spectra of the amorphous films correlate to those of the much narrower peaks of crystalline $TlInS_2$. In particular, the broad band with the maximum position in the spectra of the films varying from 274 to 286 cm^{-1} (Fig. 1) corresponds to the two most intense features of crystalline $TlInS_2$ at 280 and 292 cm^{-1} assigned to "intramolecular" vibrations of In_4S_{10} tetrahedral groups in the $TlInS_2$ structure (Fig. 2). Evidently, the same broad band in the spectrum of the amorphous film also includes a contribution from vibrations corresponding to a less intense feature at 345 cm^{-1} in the spectrum of the $TlInS_2$ crystal. Meanwhile, the lower-frequency broad feature with the maximum at around 170 cm^{-1} (and sometimes an additional shoulder or maximum near 140 cm^{-1}) in the spectra of the films is evidently related to vibrations of Tl–S bonds in the amorphous film structure. In the spectrum of the $TlInS_2$ crystal it is represented by a peak at 140 cm^{-1} (Fig. 2), although a weak maximum at 172 cm^{-1} was also reported [9]. The weak high-frequency feature near 425 cm^{-1} in the spectra of the films prepared on Si substrate (it is masked by a broad substrate-related feature for the films on silicate glass) should most probably be ascribed to S–S vibrations, since the vibrations with participation of heavier Tl and In atoms

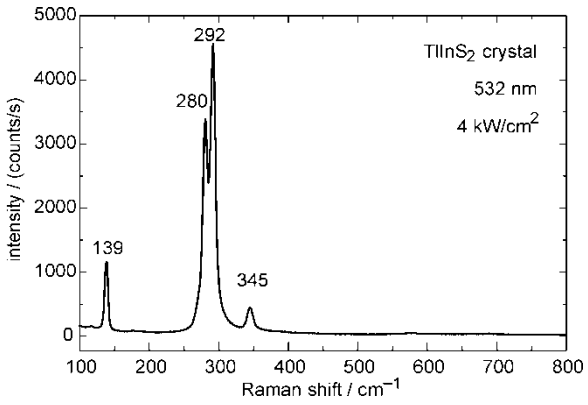

Fig. 2. Micro-Raman spectrum of $TlInS_2$ crystal measured in $Z(XX+XY)\bar{Z}$ scattering configuration at the excitation with $\lambda_{exc} = 532$ nm and $P_{exc} = 4$ kW/cm^2.

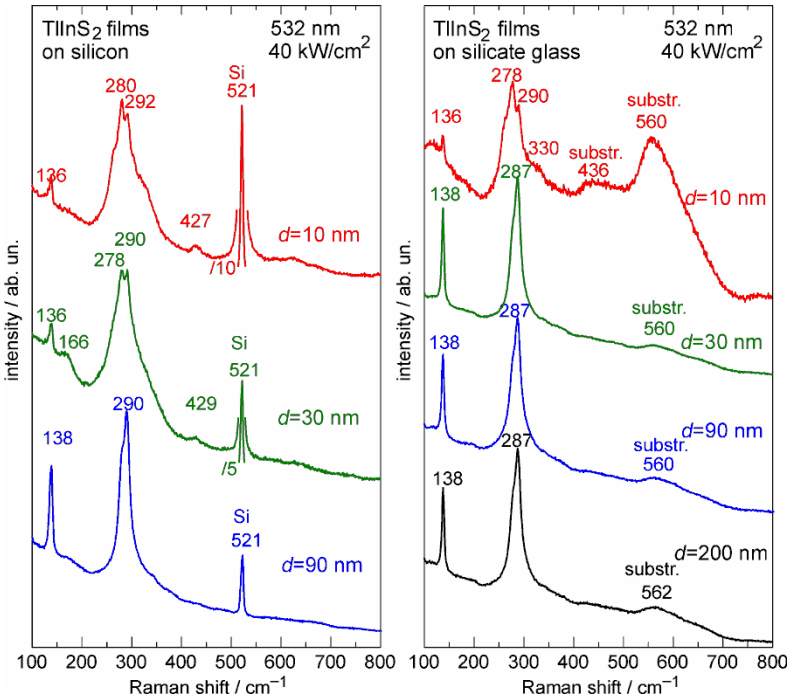

Fig. 3. Micro-Raman spectra of $TlInS_2$ films of different thicknesses d thermally evaporated on silicon and silicate glass substrates. The spectra were measured at the excitation with $\lambda_{exc} = 532$ nm and $P_{exc} = 40$ kW/cm^2.

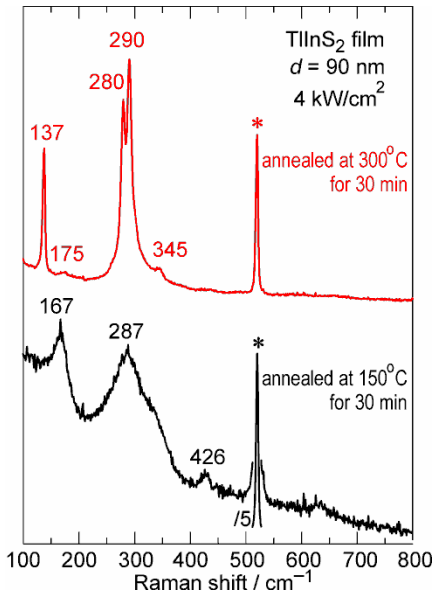

Fig. 4. Micro-Raman spectra of $TlInS_2$ films with the thickness $d = 90$ nm on Si substrates annealed for 30 min at 150 °C and 300 °C. The spectra were measured at the excitation with $\lambda_{exc} = 532$ nm and $P_{exc} = 4$ kW/cm^2. Asterisks mark the 521 cm^{-1} peak from the Si substrate.

are expected to have much lower frequencies. However, vibrations of S–S bonds are more likely to be expected at somewhat higher frequencies, around 460…490 cm^{-1}, in particular, for amorphous chalcogenides [24, 25].

In the Raman spectra of the same $TlInS_2$ films measured at a higher $P_{exc} = 40$ kW/cm^2, relatively sharp peaks at 136…138 cm^{-1} as well as in the range of 278…290 cm^{-1} are revealed (Fig. 3). These peaks may be related to rapid localized crystallization of the films under irradiation by a laser beam. For the thinner films (evaporated on Si substrates with $d = 10$ nm and 30 nm and on silicate glass substrate with $d = 10$ nm), one can clearly see two peaks in the range of 278…290 cm^{-1}, where the most intense Raman features of crystalline $TlInS_2$ are revealed (see Fig. 2). Evidently, the orientation of crystallites formed in the irradiated area is random. One may expect therefore that Raman modes of all the allowed symmetries will be revealed in the spectra. The sharp features at 136…138 cm^{-1} and 278…290 cm^{-1} emerging after the irradiation are the most intense for $TlInS_2$ in this spectral range [4, 5, 7, 11] and are readily observed therefore.

It can be also seen that the crystalline-like features in the spectra of the films measured at $P_{exc} = 40$ kW/cm^2 are considerably narrower for the thicker films, while for the thinner films they partially retain the "amorphous-like" features of the low-P_{exc} spectra, namely the broad high-frequency wing of the feature centered at 278…290 cm^{-1} and the weak feature near 425 cm^{-1} (Fig. 3). Besides, it is seen that the features typical for amorphous materials are retained for the thinner films on Si substrates much better than for the films on silicate glass substrates. Evidently, the reasons for this are related to the mechanism of film transformation under illumination by a tightly focused laser beam.

The photoinduced crystallization of $TlInS_2$ in the amorphous film can result from the material heating due to absorption of light in the laser spot. Recently, we applied Raman spectroscopy to observe a similar effect for $TlInSe_2$ films. In these films $TlInSe_2$, TlSe, and In_2Se_3 crystallites were formed under intense illumination [23]. On the other hand, photoinduced formation of $TlInSe_2$ crystallites in an amorphous As_2Se_3-based material under intense laser illumination was reported earlier for Tl–In–As–Se glass [26] as well as formation of II–VI chalcogenide nanocrystals in amorphous As_2S_3-based [25] or As_2Se_3-based [27] films was demonstrated. In these cases, the photoinduced crystallization was based on a nonthermal mechanism of photofluidization of the amorphous material (a drastic drop of its viscosity) upon illumination, which also led to an accompanying effect of photoinduced mass transport with formation of a pit in the laser spot on the amorphous film surface [25–27].

Meanwhile, in our case, the film surface remained undamaged after Raman measurements and, contrary to the earlier studies [25–27], no signs of mass transport were observed in the optical microscope used for the Raman measurements. The mechanism of localized crystallization in the illuminated films during Raman measurements at an increased P_{exc} must be purely thermal. The illuminating light is absorbed by the sample surface that causes an increase of temperature of the amorphous

film in the laser spot thereby locally enhancing the atom mobility and resulting in a reconfiguration of the film structure with formation of more energetically favourable crystallites.

We performed an experiment to check directly the effect of thermal treatment on the amorphous $TlInS_2$ film structure. The Raman spectra of the 90-nm thick $TlInS_2$ films annealed during 30 min. at 150 °C and 300 °C are shown in Fig. 4. In order to avoid additional heating of the samples during the Raman measurements, low value P_{exc} = 4 kW/cm^2 was chosen. As can be seen from Fig. 4, annealing of a film at 150 °C does not result in any visible changes of the Raman spectrum compared to the one for the as-prepared sample (Fig. 1), clearly confirming its amorphous structure. However, thermal treatment at a higher temperature (300 °C) leads to drastic changes in the spectrum with appearance of distinct peaks of crystalline $TlInS_2$ at 137, 280, 290, and 345 cm^{-1}, very much similar to those of single-crystalline $TlInS_2$ (Fig. 2) and to the spectra of the films measured at high P_{exc} (Fig. 3). This clearly confirms that the effect of $TlInS_2$ crystallization induced by high-P_{exc} illumination is driven by the thermal mechanism. One may conclude that illumination of $TlInS_2$ films with P_{exc} = 40 kW/cm^2 results in the sample heating above 150 °C.

As mentioned above, photoinduced crystallization of the thinner (10…30 nm) films is retarded compared to that of the thicker (90…200 nm) films. Moreover, this retardation is more pronounced for the films evaporated onto Si substrates compared to the films on silicate glass substrates. We believe that this is due to the fact that heat dissipation occurs faster for the thinner films due to the higher thermal conductivity of the substrate compared to that of the film material [28]. Hence, the laser light energy absorbed by a thinner film is dissipated faster and the temperature required for crystallization cannot be achieved so fast. Moreover, as crystalline silicon has much higher thermal conductivity than silicate glass [28], the thinner films on Si substrates dissipate heat even faster.

Comparing the obtained data with the results of a similar study of $TlInSe_2$ films with similar characteristics prepared by the same technique [23], one should mention that for $TlInSe_2$ the temperature of 150 °C is sufficient to initiate film crystallization. Besides, while heating (both photoinduced and applied directly) of the $TlInSe_2$ films leads to formation of several crystalline phases ($TlInSe_2$, TlSe, and In_2Se_3) [23], a single phase ($TlInS_2$) is formed in our case.

4. Conclusions

$TlInS_2$ films with the thicknesses from 10 to 200 nm were obtained by thermal evaporation on silicon and silicate glass substrates kept at room temperature. As follows from the micro-Raman spectra measured at λ_{exc} = 532 nm and a laser power density P_{exc} = 4 kW/cm^2, the structure of the films is amorphous, contrary to the data of other authors [15] for the films evaporated onto heated substrates. To our knowledge, this is the first study of amorphous $TlInS_2$ films reported so far.

Raman spectra measured at a higher P_{exc} = 40 kW/cm^2 exhibit narrow features, which evidence formation of $TlInS_2$ crystallites on the film surface. The film crystallization in the laser spot is shown to originate from the thermal effect of a tightly focused laser light absorbed by the film, which facilitates diffusion of atoms in the illuminated area and rearrangement of the film structure. Low-P_{exc} Raman measurements performed for the 90-nm $TlInS_2$ film annealed for 30 min at 300 °C confirm this conclusion, showing remarkably similar crystallite-related peaks. Meanwhile, the Raman spectrum of a similar film annealed for 30 min at 150 °C completely retains the features characteristics for amorphous films. Hence, laser light illumination by P_{exc} = 40 kW/cm^2 heats the film surface to the temperature above 150 °C. Crystallization is slower for thinner films due to the higher heat dissipation because of the higher thermal conductivity of the substrates with respect to the film material.

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Loya V.Y.: investigation.
Suslikov L.M.: supervision, resources.
Voynarovych I.M.: investigation.

Раманівське дослідження тонких плівок $TlInS_2$, отриманих методом термічного напилення

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Анотація. Тонкі (10…200 нм) плівки $TlInS_2$ отримано методом термічного напилення на підкладках з кремнію та силікатного скла. Мікрораманівські спектри, виміряні при помірній інтенсивності збуджуючого світла (532 нм, 4 кВт/см^2), підтверджують аморфний характер плівок. Вузькі особливості, які з'являються у спектрах при вищій густині потужності збудження (40 кВт/см^2), свідчать про кристалізацію $TlInS_2$ на поверхні плівки. Утворення кристалітів пояснюється термічним ефектом поглиненого лазерного світла. Показано, що поверхня плівки нагрівається лазерним променем потужністю 40 кВт/см^2 до температури понад 150 °C.

Ключові слова: аморфні плівки, раманівська спектроскопія, кристалізація, термічна обробка.