Photoplastic and photoinduced effects in amorphous $As_2Se_3:Sn_x$ thin films

D.V.HAREA, E.E.HAREA, E.P.COLOMEICO, M.S.IOVU

Institute of Applied Physics, Academy of Sciences of Moldova,
Str. Academiei 5, MD-2028 Chisinau, R. Moldova
dyanaharea@yahoo.com

Abstract: **The mechanical characteristics of amorphous As_2Se_3:Sn prepared by thermal evaporation in vacuum on glass substrates heated at T_{substr} = 100 °C are investigated. It was established that the hardness *H* of amorphous thin films is generally higher than the hardness of bulk samples of the same chemical composition. Because the composition of chalcogenide glass (ChG) determine the kind of structural units and the mean coordination number, in the present work the amorphous films of the $As_2Se_3:Sn_x$ (x=0÷10.0 at.% Sn) of chalcogenide system were investigated. The combination of depth sensing indentation and band-gap illuminations has been used to study the photoplastic effect in chalcogenide glasses. The prominent photoplastisity of thin films has been revealed through hardness deviations during nanoindentations under light illuminations from those ones which have been observed for the materials on the darkness. Some optical properties and the modification of optical parameters (optical band gap E_g, absorption coefficient α, refractive index *n*) under light irradiation and heat treatment of the amorphous $As_2Se_3:Sn_x$ thin films thin films, and the relaxation of photodarkening effect under the light exposure also was investigated.**

Keywords: **Chalcogenide glasses, hardness, photoplasticity, photostructural transformations, light-induced phenomena, Young's modulus.**

I. INTRODUCTION

For the development of modern nanotechnologies, nanostructured and functional materials, chalcogenide glasses (ChG) present a big interest. The amorphous films of ChG have been served as a base of many applications in photonics and optoelectronics, especially as inorganic photo-resists for sub-micron technology, optical diffractive elements, sensors and photonic crystals [1-3].

The effect of light-induced photo-structural transformations is characteristic for many amorphous chalcogenides films, and they have been served as a base of many applications in photonics and optoelectronics, especially as inorganic photo-resists for sub-micron technology [4-5]. Special interest is connected of doping of chalcogenide glasses (ChG) with metal impurities, which alter optical, photoelectrical and transport properties of the host material [6-8]. At the same time it was shown that doping of ChG by tin impurities assist in stabilizing the glassy matrix in respect to light exposure and thermal treatment [9, 10]. The interest in optical properties of amorphous semiconductors has been stimulated also by their possible applications as optical fibers amplifiers in telecommunication systems and integrated optics for fabrication of different diffractive elements with high resolution. It was shown that the addition of tin impurity in amorphous As_2Se_3 films can provide a pronounced effect on electrical, transport properties, optical and photo-induced phenomena [6-8, 10-13]. In this paper the experimental results on some mechanical properties, optical, and photo-induced characteristics of thermally evaporated amorphous $As_2Se_3:Sn_x$ (x=0÷10 at.% Sn) thin films (thickness *L*~2.0 μm). The transmissions spectra were used for calculate the absorption coefficient α, the optical band gap E_g, as well as the dependence of the refractive index on composition, and its modifications Δn under light irradiation and heat treatment.

A considerably information on the behavior of materials under irradiations can be obtained using the nanoindentation technique based on continuous recording of the nanoindentations process and the determination of the hardness and Young's modulus from the measured "load-indenter displacement" curve. Photoplastical effect was discovered about 60 years ago. The first work was dedicated to crystalline semiconductors [14]. Photoplastical effect is manifested by hardening of illuminated materials [15], as well as by their softening [16]. Photoplastical effect depends on a number of factors such as: radiation power, temperature and wavelength. Study of spectral dependence of photoplasticity showed the maximum effect under samples illumination with wavelength close to the band gap value $h\nu \geq E_g$ [17]. The photoplastic effect depends of the illumination power and light wavelength. With increasing illumination power the plastic deformation exhibits anomalous behavior. Studies of the spectral dependence revealed that the effect is maximized when the illumination wavelength is close to the bandgap of the crystal.

Photoplasticity in chalcogenide films were studied in [18-20], where the effect was observed during samples illumination with wavelength comparable with band gap value E_g. Nature of photoplastical effect is not carried out definitively yet. The effect was attributed to

thermal expansion of the film due to absorption of exciting light, as well as recombination of the photoexcited non-equilibrium electrons and holes. Investigation of photoplastical effect usually is performed under in-situ samples illumination during indentation [21,22] as well as their indentation after illumination [23].

V. EXPERIMENTAL

The glasses As_2Se_3: Sn_x (x = 0 to 10.0 at.% Sn) were synthesized from the starting elements of 6N (As, Se, Sn) purity by a conventional melt quenching method. The starting components elements As_2Se_3 and Sn were mixed in quartz ampoules and then evacuated to pressure of P~10^{-5} torr, sealed and heated to temperature T=900 ^oC at the rate of 1 ^oC/min. The quartz tubes were held at this temperature for 48 hours for the homogenization and then slowly quenched in the heating furnace. The amorphous As_2Se_3: Sn_x were obtained by thermal flash evaporation in vacuum (p = 5 • 10^{-5} Torr) of the initial synthesized material onto the glass substrate held at T_{substr}=100÷120 ^oC. The thickness of the amorphous films was in the range of d~1÷3.0 μm. The investigation of the mechanical properties of As_2Se_3: Sn_x (x = 0 - 10.0 at.% Sn) bulk and amorphous thin film samples was performed at PMT-3 device using Bercovici indenter. For optical transmission spectra measurements, a UV/VIS (λ = 300÷800 nm), the 61 NIR (λ = 800÷3500 nm) Specord's CARLZEISS Jena production. To initiate photostructural transformations in thin-film samples, the continuous He-Ne lasers with wavelengths λ = 633 nm was used as a source of light exposure. For data acquisition the experimental set-up included a digital build-in PC-card PCI-1713A connected with the registration module [24].

III. RESULTS AND DISCUSSIONS

The photoplastical investigation was performed using nanohardnes tester NHT CSM. The hardness was calculated from load-displacement curves by Oliver-Pharr method. In-situ illumination of samples was performed with green laser (λ = 532 nm) with power P = 50 mV/cm^2. For change the direction of incident laser beam the optical glass prism was used (Fig.1). Maximum indentation load was 5mN, which allowed maximal penetration depth does not exceed 15% of film thickness.

The hardness was calculated using the expression:

$$H_B = (1570 * P) / l^2, \qquad (1)$$

where P- is the applied load, and L – is the height of triangle of remaining imprints [25]. Both in the case of the bulk samples and for amorphous thin films the applied load was 10g, and the depth of deposited imprints does not exceeded 20% of films thickness.

Fig.2 shows variations in T_g(x) for the bulk As_2Se_3:Sn_x glasses, and the non-reversing heat $\Delta H_{nr}(x)$. One finds at low additive concentrations of Sn, T_g of the base glass to increase with x, suggesting that the base glass becomes more connected. However, as x approaches 5% of Sn, T_g show a threshold behavior [10].

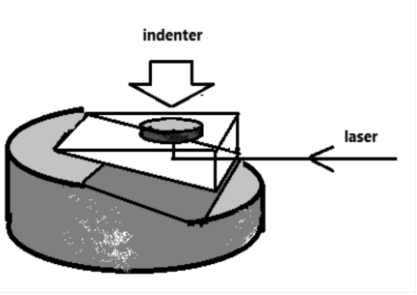


Fig.1. The experimental set-up for investigation of the photoplastical effect in chalcogenide glasses.

The isomer-shift of the line in the Mössbauer spectroscopy experiments has been previously assigned to Sn that is tetrahedral coordinated to 4 Se near-neighbors as in a $Sn(Se_{1/2})_4$ local structure [10]. Apparently, introduction of Sn additive in As_2Se_3 base glass promotes growth of $Sn(Se_{1/2})_4$ units and leads the base glass to become As-rich. The latter leads of forming $As_{2(}Se_{1/2})_4$ and As_4Se_4 structural units It was established that the hardness H of amorphous As_2Se_3:Sn_x thin films is generally higher than the hardness of bulk samples of the same chemical composition (Fig.3).

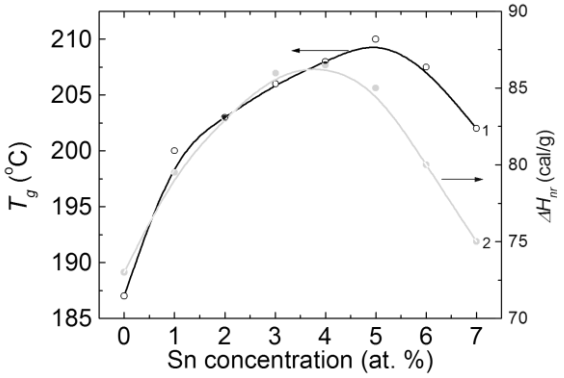


Fig.2. Variations in $T_g(x)$ (1), $\Delta H_{nr}(x)$ for As_2Se_3:Sn_x glasses. The smooth lines are computer fitting.

The experimental results of investigation of hardness for bulk and As_2Se_3: Sn_x amorphous thin films are presented in Fig.3, and are in good agreement with experimental results obtained earlier by Borisova [26]. The hardness values of bulk samples depending of Sn concentration in the As_2Se_3:Sn_x glasses and vary between 100 and 110 kg/mm^2. The hardness of the amorphous As_2Se_3:Sn_x thin films presents a non monotonous character in dependence on the Sn concentration. A sharp increasing of hardness is registered when the impurity concentration exceed the value of 3% ÷ 4% Sn. The hardness H of $(As_2Se_3)_{1-x}$:Sn_x films varies between H=115÷130 kg/mm^2. For both bulk samples and thin films the hardness is lower according to the data [27]. This fact may be connected with some technological specifics in preparing the ChG.

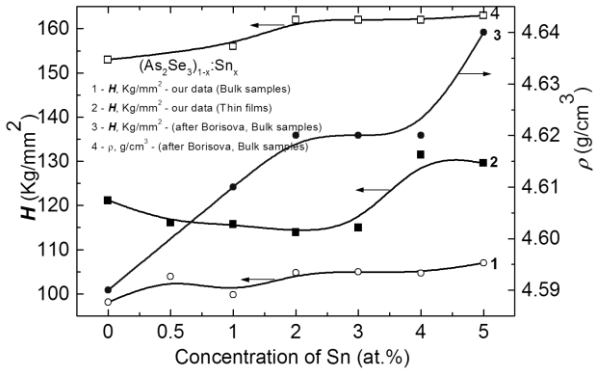


Fig.3. The hardness H (1 – bulk samples, 2 – thin films, the density ρ (3) for the $(As_2Se_3)_{1-x}:Sn_x$.

In the present paper we report the experimental results on investigation of the photoplastical effect in amorphous $As_2Se_3:Sn_x$ thin films. In the paper [10] on the base of the "slip-motion" model was shown that the photo-excited carrier play an important role in the processes of photoinduced phenomena in amorphous semiconductors. Althermal light-induced plasticity in cristalline semiconductors under light illumination causes decrease of hardness. This effect was interpreted in terms of increasing of the mobility of dislocations due to photoinduced increase of carrier concentration.

In our experiments the photomechanical properties of As_2Se_3:Snx was performed at NHT-SCM nanohardness tester. The essential difference of hardness value of investigated films, obtained using microhardness tester (Fig. 3.) and nanohardness tester (fig.4) is most probably due to different method of testers operation.

It should be noted, that PMT-3 microhardness tester calculates hardness values from projected area of relaxed imprints. The nanohardness testers operate dynamic mod of data collection, and calculate hardness values using projected imprint area when indenter is unloaded, but is not totally removed from material. During in-situ illumination of studied films, hardness decreasing was observed (Table 1 and Fig.4). Photoelastic effect becomes more visible when Sn concentrations increase. Under the illumination the nanohardness of amorphous $As_2Se_3:Sn_x$ thin films decrease. This effect is more pronounced for the compositions with Sn concentration more than 1.0 at. %. Decreasing of hahohardness under illumination also was observed for as-deposited and annealed amorphous $As_{40}Se_{60}$ thin films [20].

A microscopic model of this phenomenon is based on the assumption of the athermal decrease in the viscosity of the films during irradiation, which is close to the viscosity of vitreous semiconductors near their glass transition temperature T_g [19]. It was suggested that the photoviscous effect, that is, the athermal decrease of viscosity of non-crystalline chalcogenide upon illumination is the key for a considerable photoinduced effects in different amorphous chalcogenide films [21].

Table 1. The hardness vs. Sn concentration for amorphous $As_2Se_3:Sn_x$ thin films.

Nr.	Film composition	*H* (MPa) As-deposited films	*H* (MPa) Illuminated films
1	As_2Se_3	1780	1768
2	$As_2Se_3:Sn_{0.5}$	1762	1783
3	$As_2Se_3:Sn_{1.0}$	1734	1614
4	$As_2Se_3:Sn_{2.0}$	1735	1628
5	$As_2Se_3:Sn_{3.0}$	1745	1656
6	$As_2Se_3:Sn_{4.0}$	1774	1660
7	$As_2Se_3:Sn_{5.0}$	1732	1607

In order to investigate the modification of the optical parameters (optical band gap E_g, absorption coefficient α, refractive index n) under light irradiation, the transmission spectra $T=f(\lambda)$ of the amorphous $As_2Se_3:Sn_x$ thin films with different amount of Sn were measured. It was demonstrated that increasing of Sn concentration increase the absorption coefficient α and decrease the optical band gap E_g [28]. At the same time Sn concentration in amorphous $As_2Se_3:Sn_x$ thin films and light illumination with increase the refractive index n.

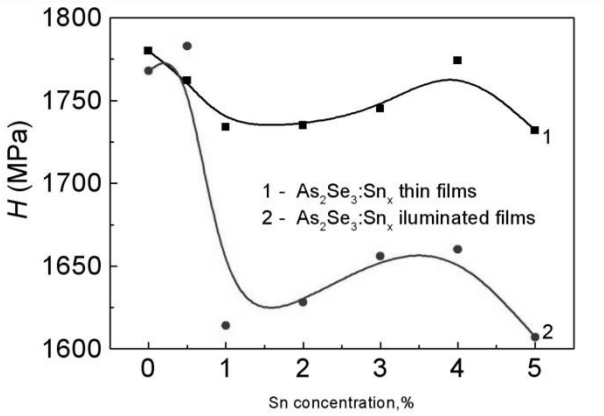
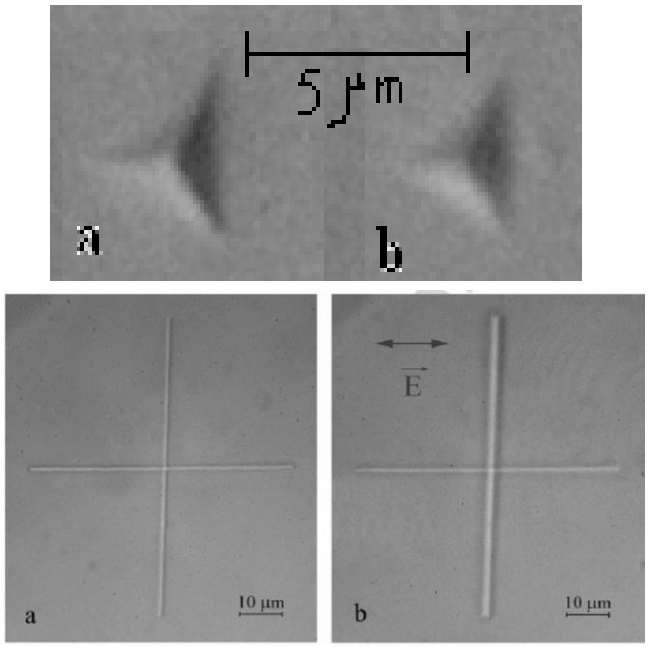


Fig.4. The hardness vs. Sn concentration for amorphous $As_2Se_3:Sn_x$ thin films.

Fig.5 represents the changes of the surface morphology of the as – deposited amorphous $As_2Se_3:Sn_{0.1}$ thin film under the indentation load after light illumination (a) and before light illumination (b, upper patterns). For comparison, in the downside of the Fig. 5 the pictures of the light-induced anisotropic plasticity in amorphous $As_{20}Se_{80}$ thin films are shown: a – the image of non-irradiated film surface, and b – the image of this surface by irradiated linearly polarized laser beam (λ = 633 nm) [19]. In both cases it is observed that after light irradiation the plasticity of the investigated amorphous films increase. For explanation of this phenomena the authors [19] the mechanical model of anisotropic plasticity in chalcogenide glasses have been proposed, according to which the anisotropic softening consists in the weakening of mechanical compliance in the direction to be orthogonal to the light polarization.

Fig.6 represents the dispersion curves of the refractive index $n=f(\lambda)$ for the amorphous $As_2Se_3:Sn_{1.0}$ thin films: 1 – as-deposited (curve 1), as-deposited upon light exposure (curve 2), as – deposited upon heat treatment (curve 3), and as – deposited upon heat treatment and light exposed (curve 4). From the Fig.5 we can observe that the maximum modification of the refractive index Δn take place for the fresh as – deposited amorphous films upon light exposure $\Delta n = 0.078$ (curves 1 and 2). For the annealed amorphous thin films the modification of the refractive index Δn under light exposure are smaller $\Delta n = 0.016$ (curves 1 and 4). These features may be explained by the fact, that for the annealed amorphous films the structure of the films became more stable.



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The plot $(n^2-1)^{-1}$ *vs.* $(h\nu)^2$ (Fig.7) allow to determine the oscillator parameters by fitting a straight line to the experimental points. For as - deposited amorphous $As_2Se_3:Sn_{1.0}$ thin films, by extrapolating the fitting line towards $(h\nu)^2=0$, one can obtain the static refractive index $n(0)$=2.321 and the static dielectric constant $\varepsilon_s = n^2(0)$ = 5.386. For as – deposited thin films upon the light exposure these values are higher $n(0)$=2.338 and $\varepsilon_s = n^2(0)$ = 5.464, respectively.

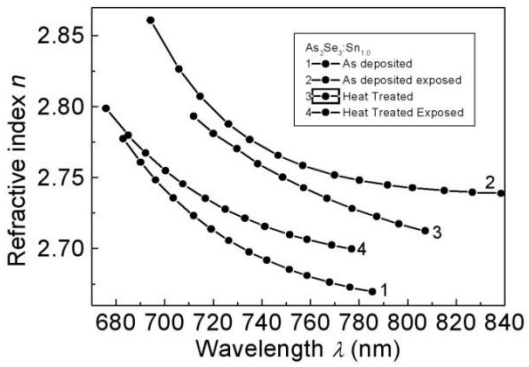

Fig.6. The dispersion curves of the refractive index $n=f(\lambda)$ for the amorphous $As_2Se_3:Sn_{1.0}$ thin films: 1 – as-deposited (curve 1), as-deposited upon light exposure (curve 2), as – deposited upon heat treatment (curve 3), and as – deposited upon heat treatment and light exposed (curve 4).

The dispersion of the refractive index is related to the electronic absorption spectrum through the Wemple equation based on the single electronic oscillator model [29]:

$$(n^2-1)=\frac{E_d E_0}{E_0}-(h\nu)^2, \qquad (2)$$

where E_0 is the average electronic energy gap, and E_d is the dielectric oscillator strength.

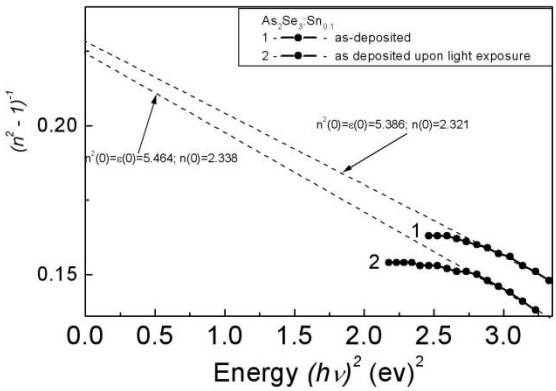

Fig.7. The dependence $(n^2-1)^{-1} = f((h\nu)^2)$ for as – deposited amorphous $As_2Se_3:Sn_{1.0}$ thin films (1) and as – deposited upon light exposure (2).

Large values of the refractive index n are obtained for smaller E_0 and for large E_d and leads to a large dispersion throughout the chalcogenide glass material. From equation (2) we obtain

$$(n^2-1)^{-1}=\frac{E_0}{E_d}-(\frac{1}{E_0 E_d})(h\nu)^2 \qquad (3)$$

Using the plot $(n^2-1)^{-1}$ *vs.* $(h\nu)^2$, for amorphous $As_2Se_3:Sn_{1.0}$ thin films the E_0 and E_d were calculated with the values E_0 = 3.094 eV and E_d = 13.571 eV for as –

deposited films, and with the values E_0 = 2.906 eV and E_d = 12.972 eV for as –deposited films upon light exposure, respectively. The value of E_0 = 3.094 eV is higher than the value of the optical band gap E_g = 1.82 eV calculated from the Tauc plot. For the amorphous $As_{12.6}Ge_{23.8}S_{63.6}$ the relationship between the values of E_0 and E_g was established a relationship $E_0 \approx 2 \times E_g$ [30].

The photoinduced phenomena of amorphous As_2Se_3:Sn_x thin films were investigated by measuring of the relaxation of the relative optical transmission $T(t)/T(0)=f(t)$ curves under the light exposure (λ=630 nm nm) at room temperature (Fig.8).

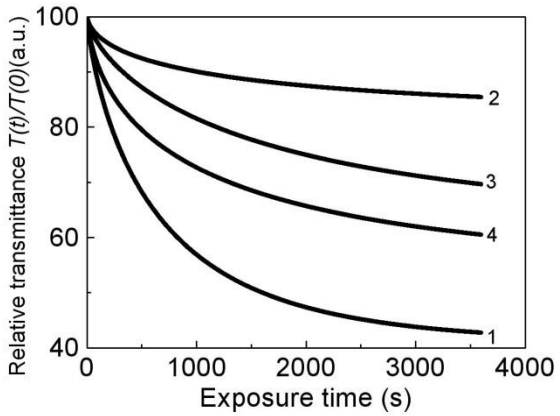

Fig.8. Photodarkening kinetics of as-deposited As_2Se_3:Sn_x films with exposure time. x (at. %Sn) = 0 (1); 1.0 (2); 3.0 (3), 5.0 (4).

At a constant light intensity the presented dependences characterize the decay of the film optical transmittance with the increase of the dose of absorbed photons. From Fig.5a it is shown that even 1.0 at.% of tin impurity significantly reduces the fotodarkening effect in as-deposed amorphous As_2Se_3 films. The decrease of photodarkening is characteristic for amorphous As_2Se_3 films doped with tin amount up to 2.0 at.% Sn. For higher concentrations of tin the effect of reducing of phodarkening by impurity decreases. To obtain a unified basis for comparison of the transmission relaxation $T(t)$ curves we used so called stretched exponential presentation for the relaxation curves in Fig.5a and Fig.5b in the form:

$$T(t)/T(0) = A_0 + A\exp[-(t-t_0)/\tau]^{(1-\beta)} \qquad (4)$$

Here t is the exposure time, τ is the apparent time constant, A characterizes the exponent amplitude, t_0 and A_0 are the initial coordinates, and α is the dispersion parameter ($0<\beta<1$). For explanation of the photodarkening process in the investigated amorphous thin films, a "slip-motion" model for a-$As_2Se(S)_3$ has been involved [31], in which the photoexcited charge carriers in extended states are considered as responsible for photodarkening.

This effect is particularly important at high Sn contents in our case up to x=2.0 at.% Sn, when impurity approaches the dissolution limit and actually changes the number of structural units in the glass. In the untreated films, as it was pointed out earlier [8], the changes in the structure after introducing Sn impurity occur first of all in the layers, that is why the slip mobility is retained, while the enlargement of the interlayer distance promotes the slip motion slightly decreasing the time constant of the process.

Alternatively, it is likely that Sn goes largely in the layers and buckles them, since Sn possesses a larger (1.40 Å) covalent radius than As (1.20 Å). The Sn-Se bond strength (47.4 kcal/mole) exceeds [24] that of As-Se one (41.7 kcal/mole). The enhanced interlayer interactions come at the expense of reduced interlayer ones. The increased inter-layer spacing reflects a loss of inter-layer interactions. The buckling or loss of planarity of the layers would naturally suppress the slip motion of the layers that is thought to contribute to photodarkening as suggested by Shimakawa and collaborators [31].

The reduction in optical gap of As_2Se_3 glass upon alloying Sn, most likely, results from a broadening of the valence band. The top of the valence band in these glasses has contribution from Se lone-pair electrons. The lone pair band broadens because of a pressure effect. The size mismatch produced by doping Sn in the layers produces stresses that are primarily localized on the bridging Se atoms, the most forgiving and flexible connections (coordination number of 2) in the backbone. This is akin to applying an external pressure, and as shown by different authors the band-gap of crystalline As_2Se_3, indeed, decreases upon applying an external pressure. The same physical mechanism occurs in the glass with chemical doping serving to clamp the Se local environments.

CONCLUSION

In this paper are investigated the mechanical characteristics of amorphous As_2Se_3:Sn_x (x=0÷5.0 at.% Sn) chalcogenide system prepared by vacuum evaporation on glass substrates. It was established that the hardness H of amorphous thin films is generally higher than the hardness of bulk samples of the same chemical composition. By applying in-situ stress measurement of photomechanical changes in chalcogenide films based on As_2Se_3:Sn_x we have found essential dynamic changes in films plasticity.

Photodarkening and its relaxation under light exposure in amorphous films of As_2Se_3:Sn (0.5 to 5.0 at.% Sn) was studied in dependence on the Sn concentration. It was shown that tin impurity effectively reduces photodarkening and the degree of reduction becomes stronger up to 2.0 at.% Sn, than photodarkening is increased again.

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