Journal of Optoelectronics and Advanced Materials Vol. 7, No. 2, April 2005, p. 619 - 624

PHOTO-INDUCED TRANSITION FROM ELASTIC TO PLASTIC BEHAVIOR IN AMORPHOUS As-Se FILMS STUDIED BY NANOINDENTATION

M. L. Trunov*, S. N. Dub[a], R. S. Shmegera[a]

Uzhgorod National University, 13, Kapitulna str., Uzhgorod 88000, Ukraine,
[a]Institute for Superhard Materials, 2 Avtozavodskaya str., Kiev 04074, Ukraine

The combination of depth-sensing indentation and band-gap illumination has been used to study the photoplastic effect (the reversible influence of light on the flow stress, hardness and plasticity) in chalcogenide glasses on a nanoscale. The prominent photoplasticity of thin As_{50} Se_{50} films has been revealed through deviations in the shape of load-displacement curves during nanoindentation under light illumination from those observed for the material in the darkness. The photoinduced changes in static mechanical properties such as nanohardness and elastic (Young's) modulus have been determined. The results show that Young's modulus increases by a factor of two under band-gap light illumination while nanohardness decreases drastically. As a result an essential plastic flow for the films under constant load has been observed. This observation is associated with a glass transition from initial elastic state with rigid and floppy phases to their essential modification induced by illumination at ambient temperature.

(Received August 8, 2004; after revision March 8, 2005; accepted March 23, 2005)

Keywords: Chalcogenide glass, Photoplasticity, Nanoindentation, Nanohardness, Young's modulus

1. Introduction

Light illumination of thin As-S(Se) films can greatly affect the properties such as surface morphology (photocontraction [1] or photoexpansion [2], microhardness [3]) and even induce mass transport [4]. Our previous investigations [5-7] have been concentrated on the phenomenon of photoinduced plasticity according to the data obtained by microindentation method, which uses a diamond Vickers pyramid as an indenter. The athermal increase in the plasticity of glasses has been achieved under the combined action of external stresses under the indenter and light illumination and as a result the glass viscous flow with a viscosity near 10^{12}-10^{13} Poise at ambient temperature has been observed [5,6]. Unfortunately, the successful use of microindentation technique to study photoinduced elastic to plastic transformations in glasses is limited by difficulties associated with monitoring the process of indentor penetration and relatively a large area of indented surfaces of the sample.

For the last decade the nanoindentation has become a commonly used technique to measure local mechanical properties and its changes under external influence from very small volumes of the material [8]. In the nanoindentation test the displacement of the indenter and the applied load are continuously monitored. This method allows us to record the penetration depth of a sharp indentor as

* Corresponding author: stm@tn.uz.ua

a function of applied load. As a result the material hardness and its changes are directly determined by analysis of the corresponding load–displacement curves independently from the residual imprint. Static mechanical properties such as Young's modulus and local (nano) hardness can also be obtained from the unloading curve [9]. In addition to the measurement of such a characteristic, the nanoindentation test may be used as a local mechanical probe to study the plasticity on a nanoscale as the mechanical response of such brittle materials as glasses. No results of nanoindentation studies of chalcogenide glasses and films are known to us except for casual (non-systematic) investigations of a pure a-Se [10].

The aim of this study is to demonstrate photoinduced changes in the nanoscale plasticity of $As_{50}Se_{50}$ films. Futhermore, it is shown that opposite changes in local mechanical properties (Young's modulus and nanohardness) take place due to band-gap light illumination. These unusual results are explained in terms of the two-phase (floppy and rigid) formation in the initial glass and their modification under the action of light.

2. Experimental

The experiments were performed on 1 μm thick films prepared by thermal evaporation of $As_{50}Se_{50}$ glasses from quasi-closed effusion cells with the rate of approximately 30 nm/s on unheated glass substrates. Both types (as-deposited and annealed) of the samples are irradiated by a beam of light with approximately band-gap energy from a laser diode (the wavelengh from 630 to 650 nm) with the intensity of < 1mW, so the power of the laser irradiation on the sample surface did not exceed 30 mW/cm^2. Nanoindenatation measurements were performed using a depth-sensing nanohardness tester (model Nanoindenter II, MTS Systems) with a Berkovich diamond pyramid as an indenter. The details of instruments and methods are given in [11]. Our load schedules comprised first a constant rate of 0.04 mN/s load ramp to the peak load of 2 mN, followed by holding at the peak load for a duration of 360 s and then constant-rate of 0.04 mN/s unloading. The nanohardness and Young's modulus were calculated from the load–displacement curves according to the Oliver and Pharr procedure [9] with a typical error of $\pm$ 0.1 GPa and of $\pm$ 1 GPa, respectively.

The films were illuminated from the bottom of samples through transparent substrates. Since the indented volume of films is much smaller than that affected by the laser irradiation, the changes in plasticity reported here were caused by the influence of light only. We have neglected the role of temperature increase due to illumination and assumed that no microstructural changes in the film material take place as the result of the high pressure created on the indenter tip.

3. Results and discussion

Fig. 1 shows an example of nanoindentation of the as-deposited $As_{50}Se_{50}$ film in permanent darkness (a), during first illumination (b), after illumination (c) and during second illumination (d). The same experiments, but for the annealed film were shown in Fig. 2 (a-c), respectively. In a series of nanoindentation under illumination the light was turned on during loading at total depth of 200-220 nm for the as-deposited film and 160-180 nm for the annealed one. In both cases it correponds to 25 s on the loading-time curve after the load to an indenter is applied.

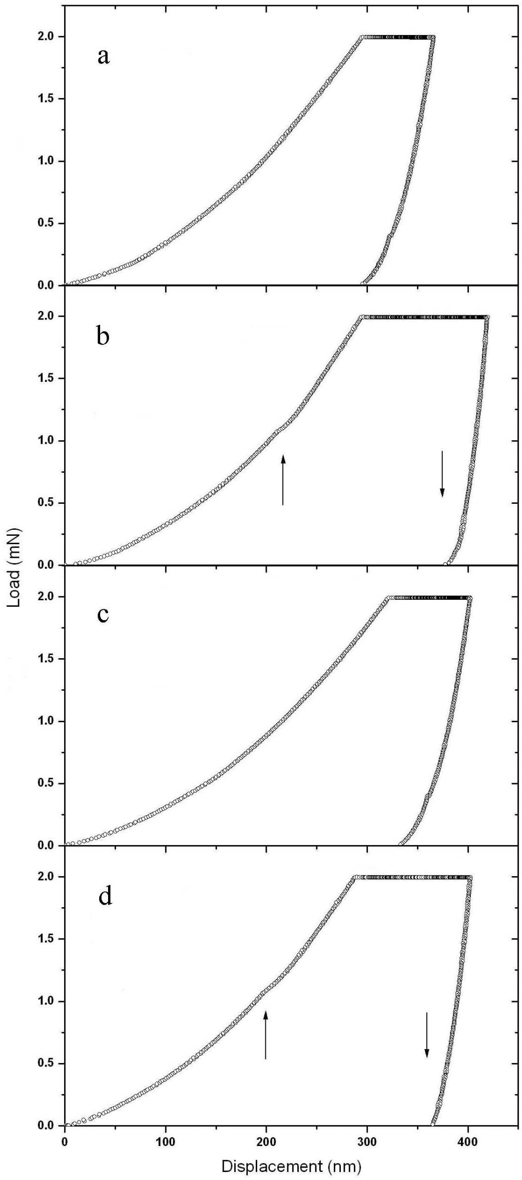

Fig. 1. Four types of load-displacement curves in nanoindentation of as-deposited $As_{50}Se_{50}$ film. The arrows show the beginning (↑) and the end (↓) of exposure. See text for details.

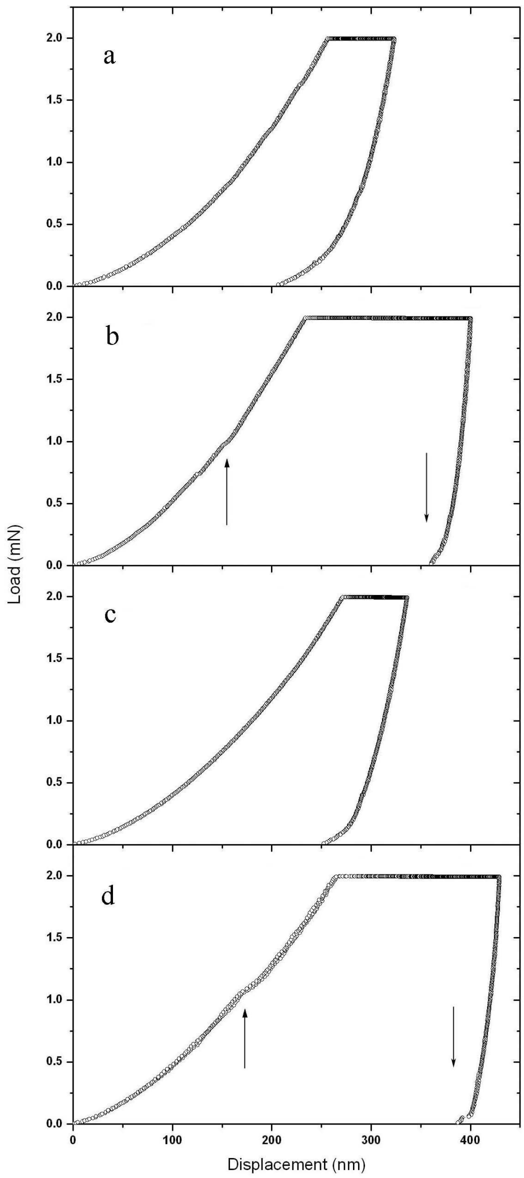

Fig. 2. Four types of load-displacement curves in nanoindentation of annealead $As_{50}Se_{50}$ film. The arrows show the beginning (↑) and the end (↓) of exposure. See text for details.

Since the material is elastic when indented in the permanent dark and taking into consideration the results obtained earlier from the microindentation experiments [12], an increase in the penetration depth was expected when indenting under illumination. Though the slope of the load-displacement curve changed almost immediately when the light was turned on (as shown by arrows in Figs. 1 and 2) and no significant effect was recorded during loading (see Fig. 1 b, d and Fig. 2 b, d, respectively).

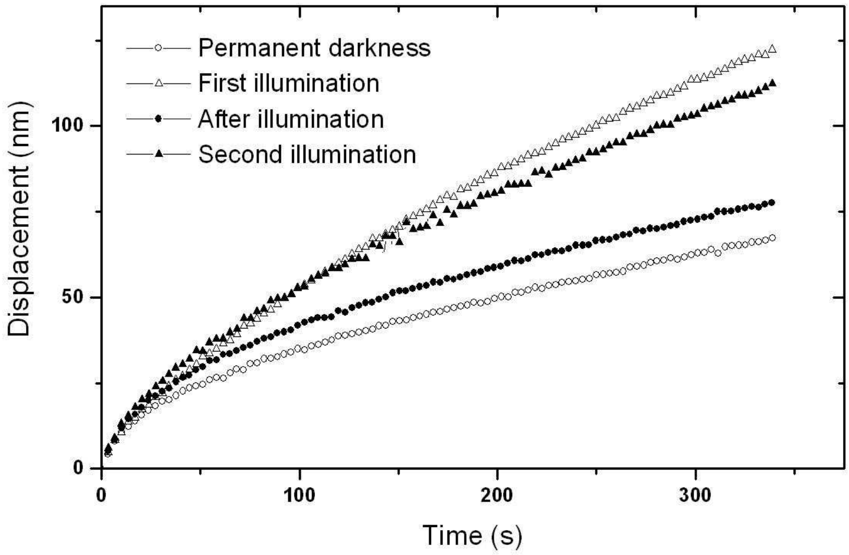

Fig. 3. Displacement-time (creep) curve of the load-hold process for as-deposited $As_{50}Se_{50}$ film. The data are normalized at the zero point.

Figs. 1 and 2 show that the light causes a drastic increase in the penetration depth when indenting under illumination and keeping the load on the indenter constant. For the as-deposited film total depths (after recovery) for the load of 2 mN during holding time of 360 s before and after illumination were 295 nm (Fig. 1a) and 333 nm (Fig. 1c), respectively, whereas the illumination increased the depth to 378 nm in the first cycle (Fig. 1 b), and to 365 nm in the second one (Fig. 1 d). The appropriate value for indentation of annealed films under the same load and holding time parameters changed from 205 nm (Fig. 2 a) and 250 nm (Fig. 1 c) to 360 nm (Fig. 2 b) and 390 nm (Fig. 2 d), respectively.

Nanohardness and Young's modulus and their changes under illumination were calculated. The results obtained during the series of measurements are summarized in Table 1.

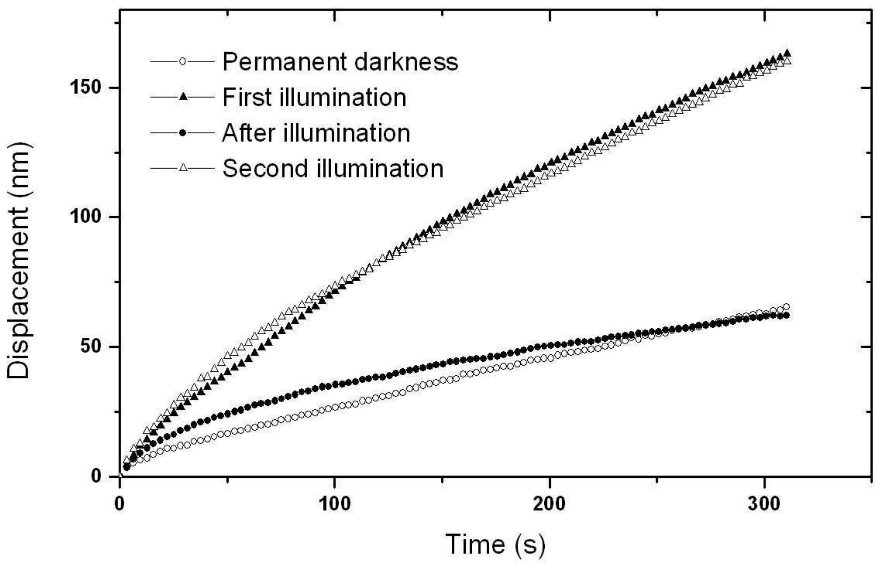

Fig. 4. Displacement-time (creep) curve of the load-hold process for annealead $As_{50}Se_{50}$ film. The data are normalized at the zero point.

The variation of nanohardness as a function of light presence or absence shows a decrease in nanohardness during illumination. The final penetration depth increases under illumination. The creep behavior of the tested samples, during the hold period, is shown in Figs. 3 and 4.

It may be noted that creep is much higher under illumination for both types of the film (see the creep column in Table 1). Moreover the light illumination allows the plastic flow to occur practically completely which dominates over the elastic deformation, because the relations between total penetration depth h_t and the depth after recovery h_r for as-deposited and annealed films are close to 1 (see the h_r/h_t column for the films indented under illumination and the shape of unloading curves on Fig. 1 b, d and Fig. 2 b, d). This fact confirms that the photoinduced plasticity in chalcogenide glasses on a nanoscale takes place.

Table 1. Data obtained from the measurements carried out on 1 μm $As_{50}Se_{50}$ film. H and E are nanohardness and Young's modulus, respectively. $\Delta H/H$ is the relative hardness change (in %) due to photoplasticity, h_t is the total penetration depth and h_r is the depth after recovery.

.

H (GPa)	E (GPa)	h_r/h_t	$\Delta H/H$ (%)	Creep (nm)	Comments
			as-deposited film		
0.63	28	0.80	27	70	permanent darkness
0.46	40	0.90		125	first illumination
0.52	25	0.82	< 4	75	after illumination
0.50	38	0.93		110	second illumination
			Annealed film		
0.81	28	0.63	39	60	permanent darkness
0.50	54	0.90		160	first illumination
0.76	25	0.75	42	60	after illumination
0.44	53	0.90		160	second illumination

The data obtained here (Table 1) show an increase in Young's modulus under irradiation of the films by a factor of 1.5 for the as-deposited film and even by factor of 2 for annealed ones. It indicates that different deformation mechanisms are activated as the combined action of the light illumination and the external stress is applied.

The microscopic mechanism of the above changes is not completely understood and must be investigated by direct structural methods. Nevertheless, on the basis of the obtained results a macroscopical model may be proposed. In short, we assume that chalcogenide glasses have two phases, rigid and floppy, at the initial stage simultaneously. Switching on the light during nanoindentation modifies the floppy phase which is responsible for the plastic flow of glasses whereas modification of the rigid phase defines the change (increase) in Young's modulus of the material on a nanoscale.

We do not consider here the initial dimensions of these phases and the degree of their change as well as a definite modification mechanism. Note that only within such a conception one can explain the simultaneous increase in plasticity and rigidity of the chalcogenide glass observed under light illumination. After illumination nanohardness and Young's modulus also decrease (see Table 1) that agrees well with known results on the photoinduced decrease in elastic constants on the macroscale [13].

4. Conclusions

The mechanical response to the light illumination of As-Se films has been investigated by nanoindentation tests. We have made the first observation of the photoplastic effect (the increase in plasticity with light) of chalcogenide glasses on a nanoscale.

References

[1] C. A. Spence, S. R. Elliott, J. Non-Cryst. Solids **97/98**, 1215 (1987).
[2] H. Hisakuni, K. Tanaka, Appl. Phys. Lett. **65,** 2925 (1994).
[3] I. Manika, J. Teteris, J. Non-Cryst. Solids **90**, 505 (1987).
[4] A. Saliminia, T. V. Galstian, A. Villeneuve, Phys. Rev. Lett. **85**, 4112 (2000).
[5] M. L. Trunov, A. G. Anchugin, Pis'ma Zh. Tekh. Fiz., **18**, 78 (1992). (in Russian); [Sov. Tech. Phys. Lett. **18,** 158 (1992)].
[6] M. L. Trunov, J. Non-Cryst. Solids **192/193**, 431 (1995).
[7] M. L. Trunov, V. S. Bilanich, J. Optoelectron. Adv. Mater. **5**, 1085 (2003).
[8] M. R. VanLandingham, J. Res. Natl. Inst. Stand. Technol. **108**, 249 (2003).
[9] W. C. Oliver, G. M. Pharr, J. Mater. Res. **7**, 1564 (1992).
[10] B. Tang, A. H. W. Ngan, J. Mater. Res. **18** 1141 (2003).
[11] N. G. Olson, C. Leung, X. Wang, Experimental Techniques **12,** 51 (2002).
[12] M. L. Trunov, V. S. Bilanich, Thin Solid Films **459**, 228 (2004).
[13] S. R. Elliott, J. Non-Cryst. Solids **81**, 71 (1986).