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OPTICAL PROPERTIES OF X-RAY IRRADIATED Cu_6PS_5I-BASED THIN FILMS DEPOSITED BY MAGNETRON SPUTTERING

Cu_6PS_5I-based thin films deposited by magnetron sputtering were irradiated using wideband radiation of Cu-anode X-ray tube at different exposition times. Optical transmission spectra, Urbach absorption edge, and dispersion of refractive index for X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films were studied. The decrease of energy pseudogap and increase of refractive index with increase of X-ray irradiation time is observed.

Keywords: thin film, magnetron sputtering, X-ray irradiation, optical absorption, refractive index.
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Introduction

Cu_6PS_5I compounds belong to the superionic conductors with argyrodite structure [1]. Due to the high electrical conductivity, they are the promissing materials for the development of new types of solid-state batteries and supercapacitors [2]. At room temperature, Cu_6PS_5I crystal belongs to the cubic syngony ($F\bar{4}3m$ space group) [1]. Optical absorption, luminescence, Raman scattering, and refractive index dispersion for Cu_6PS_5I crystals were studied in Refs. [2-4].

In the recent years much attention has been devoted to studies of composites and ceramics based on Cu_6PS_5I superionic conductors [5–7]. Now it is important to obtain thin films on their base which would enable one to vary their physical properties and spheres of application. Cu_6PS_5I thin films for the first time were deposited onto silicate glass substrates by non-reactive radio frequency magnetron sputtering [8]. Structural studies show the formation of a homogeneous two-dimensional amorphous structure. A typical Urbach bundle is observed, temperature dependences of the optical pseudogap and the Urbach energy are described in the Einstein model. The influence of annealing and ionic implantation on the absorption edge parameters of Cu_6PS_5I thin films was investigated in Ref. [9, 10].

Electrical studies have shown that the total electric conductivity of the thin films increase with increase of Cu atoms content [11]. Besides, with Cu content increase, a red shift of the optical transmission spectra, as well as a typical Urbach bundle, explained by strong electron-phonon interaction, are observed. It is shown in Ref. [11] that the Urbach tail of the investigated thin films caused by the influence of different types of disordering and mainly determined by the structural disordering.

The aim of the present paper is to investigate the influence of X-ray irradiation on the optical transmission spectra, Urbach absorption edge parameters and refractive indeces of Cu_6PS_5I-based thin films.

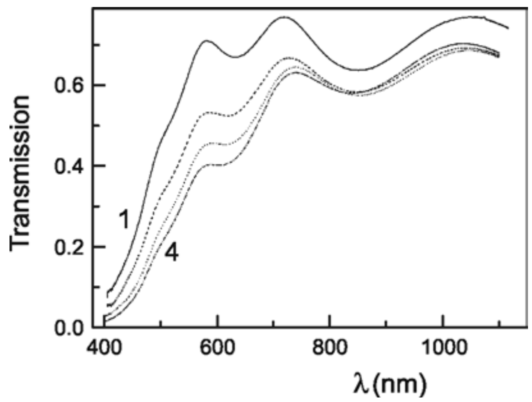
Experimental

Thin films were deposited from polycrystalline Cu_6PS_5I target onto silicate glass substrate at room temperature by non-reactive radio frequency magnetron sputtering. The structure of the deposited films was analyzed by X-ray diffraction and scanning electron microscopy (SEM). Diffraction patterns show the films to be amorphous. The thin films chemical composition was determined by the energy-dispersive X-ray spectroscopy. Structural studies performed by SEM confirm the formation of the uniform two-dimensional structure.

X-ray irradiation was performed for the different exposition times (30, 60 and 120 min) using wideband radiation of Cu-anode X-ray tube with approx 400 W of power applied (33 kV, 13 mA). Optical transmission spectra of $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films were studied at room temperature using MDR-3 grating monochromator. Spectral dependences of absorption coefficient and dispersion dependences of refractive index of thin films were calculated using the well-known method [12].

Results and discussion

Figure 1 presents the optical transmission spectra at various irradiation time at room temperature for X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films. With irradiation time increase the red shift of the short-wavelengt part of transmission spectra and interference maxima is observed.



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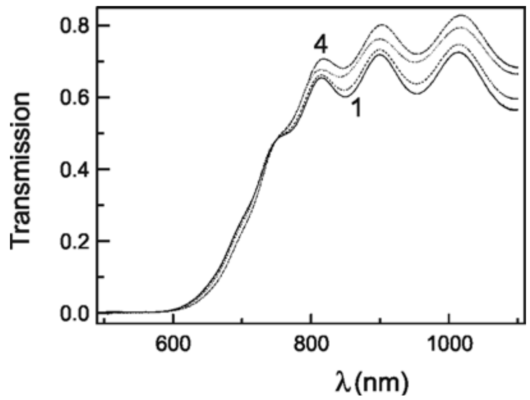

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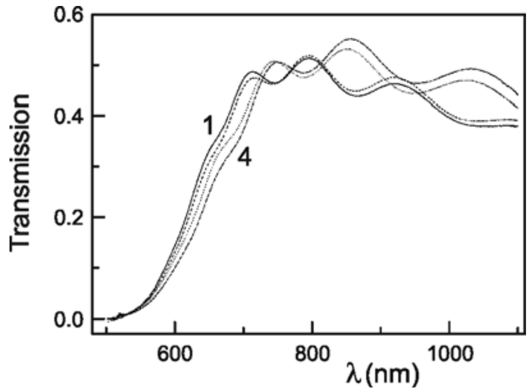

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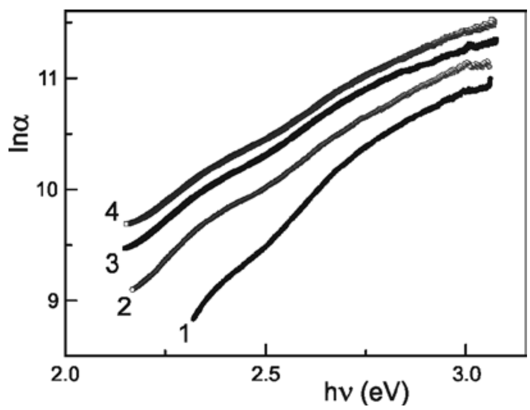
Fig.1. Optical transmission spectra of non-irradiated (1) and X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$ (a), $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ (b) and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ (c) thin films at various irradiation time: (2) 30, (3) 90 and (4) 210 min.

Spectral dependences of the absorption coefficient at various irradiation time at room temperature for X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films are shown in Fig.2.



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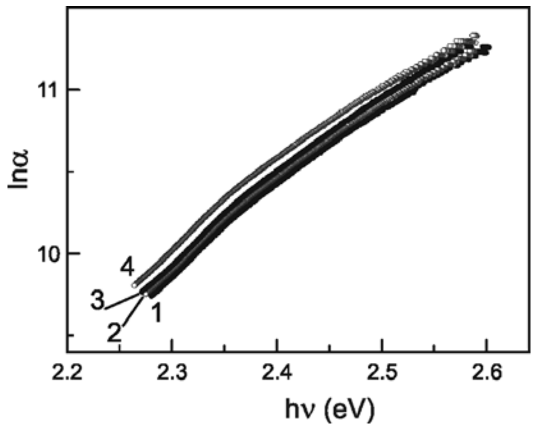

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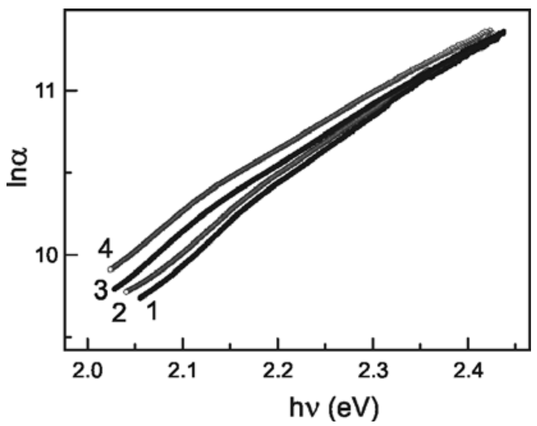

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Fig.2. Spectral dependences of the absorption coefficient of non-irradiated (1) and X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$ (a), $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ (b) and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ (c) thin films at various irradiation time: (2) 30, (3) 90 and (4) 210 min.

In Ref. [9] it is shown that the optical absorption edge for both as-deposited and annealed Cu_6PS_5I-based thin films in the region of its exponential behaviour are described by Urbach rule [13]

$$\alpha(h\nu,T)=\alpha_0\cdot\exp\left[\frac{h\nu-E_0}{E_U(T)}\right], \qquad (1)$$

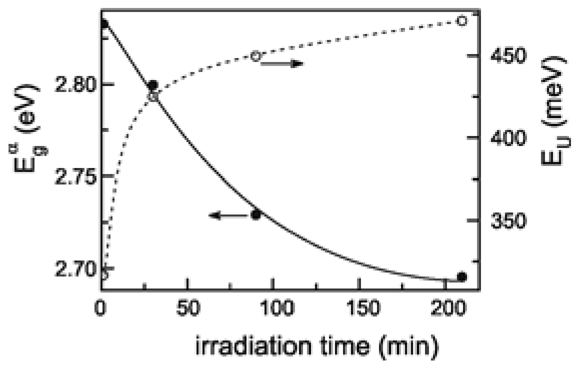
where $E_U(T)$ is the Urbach energy, α_0 and E_0 are the coordinates of the convergence point of the Urbach bundle, $h\nu$ and T are the photon energy and temperature, respectively. In the X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films we also observed the Urbach behaviour of the optical absorption edge. It should be noted that the optical absorption edge for X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films is shifted to the long-wavelength region with irradiation time increase.

Table 1

Optical parameters of non-irradiated and X-ray irradiated Cu_6PS_5I-based thin films

Irradiation time (min)	0	30	90	210
$Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$				
n	2.295	2.302	2.307	2.313
E_g^α (eV)	2.874	2.831	2.738	2.694
E_U (meV)	329	438	462	484
$Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$				
n	2.380	2.382	2.384	2.386
E_g^α (eV)	2.521	2.521	2.519	2.512
E_U (meV)	256	257	260	269
$Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$				
n	2.815	2.832	2.862	2.886
E_g^α (eV)	2.283	2.271	2.256	2.237
E_U (meV)	254	265	286	306

The observed variation of the optical absorption edge leads to the energy pseudogap E_g^α decrease and Urbach energy E_U increase with irradiation time increase (for $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$ from 2.874 to 2.694 eV (by 0.18 eV or 6.3%) and from 329 to 484 meV (by 155 meV or 47.1%), respectively; for $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ from 2.521 to 2.512 eV (by 0.009 eV or 0.4%) and from 256 to 269 meV (by 13 meV or 5.1%), respectively; for $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ from 2.283 to 2.237 eV (by 0.046 eV or 2.0%) and from 254 to 306 meV (by 52 meV or 20.5%), respectively). The dependences of E_g^α and E_U for X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films on irradiation time are presented in Fig. 3.



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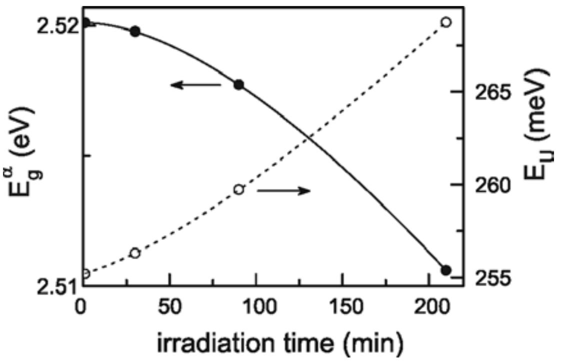

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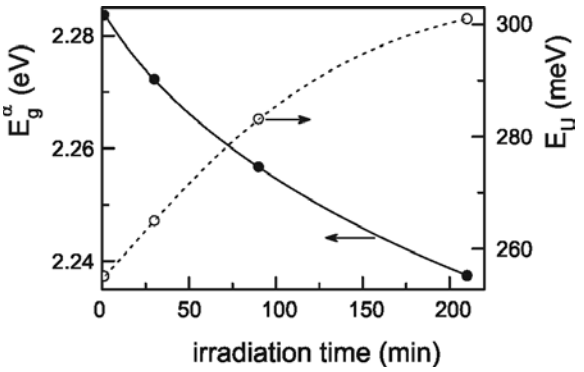

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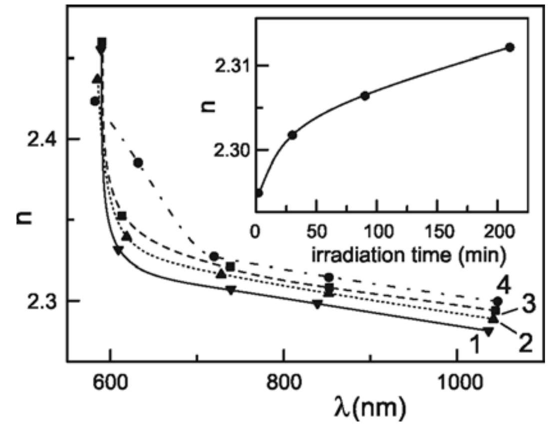
Fig.3. Dependences of the energy pseudogap E_g^{α} (α=5×10^4 cm^{-1}) and Urbach energy E_U on X-ray irradiation time for $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$ (a), $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ (b) and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ (c) thin films.

It is well-known that Urbach energy E_U is characterised the disordering level of investigated system and is described by the equation [14]

$$E_U = (E_U)_T + (E_U)_X, \qquad (2)$$

where $(E_U)_T$ and $(E_U)_X$ are the contributions of temperature and structural disordering to E_U, respectively. The Urbach energy E_U increasing is the evidence of the increase of the structural disordering due to the X-ray irradiation.

Dispersion dependences of the refractive index for the X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films at various irradiation time are presented in Fig.4. In the transparency region a slight dispersion of the refractive indeces for the X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films is observed, increasing with approaching the optical absorption edge. With irradiation time increase the nonlinear increase of the refractive indeces in the X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films is revealed. The X-ray irradiation leads to the refractive index increase from 2.295 to 2.313 (by 0.018 or 0.8%) for $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, from 2.380 to 2.386 (by 0.006 or 0.3%) for $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and from 2.815 to 2.886 for $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ (by 0.071 or 2.5%), at λ=1 μm.



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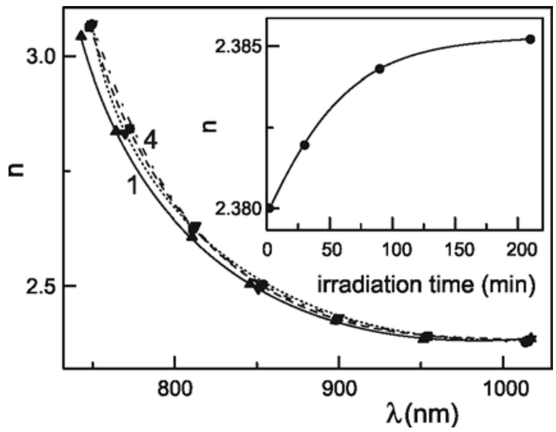

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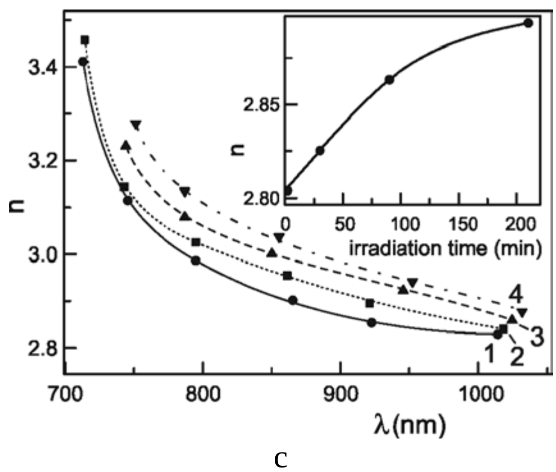

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Fig.4. Refractive index dispersions of non-irradiated (1) and X-ray irradiated $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$ (a), $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ (b) and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ (c) thin films at various irradiation time: (2) 30, (3) 90 and

(4) 210 min. The insets show the dependences of refractive indeces on X-ray irradiation time.

Conclusions

$Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films were deposited onto silicate glass substrates by non-reactive radio frequency magnetron sputtering. The influence of X-ray irradiation on optical properties of $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ and $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$ thin films was investigated. With irradiation time increase the decrease of energy pseudogap as well as increase of Urbach energy and refractive index values were observed. It was determined that with irradiation time increase the darkening and densification of the thin films occur. The increase of Urbach energy indicates on increase of structural disordering contribution into E_U induced by the influence of X-ray irradiation.

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ОПТИЧЕСКИЕ СВОЙСТВА ОБЛУЧЕННЫХ РЕНТГЕНОВСКИМИ ЛУЧАМИ ТОНКИХ ПЛЕНОК НА ОСНОВЕ Cu_6PS_5I, НАНЕСЕННЫХ МАГНЕТРОННЫМ РАСПЫЛЕНИЕМ

Тонкие пленки на основе Cu_6PS_5I, нанесенные методом магнетронного распыления, облучались при различных временах экспозиции с использованием широкополосного излучения медного анода рентгеновской трубки. Изучены спектры оптического пропускання, урбаховский край оптического поглощения и дисперсия показателей преломления облученных рентгеновским излучением тонких пленок $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ и $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$. Обнаружено уменьшение енергии псевдозапрещенной зоны и увеличение показателя преломления при увеличении времени рентгеновского облучения.

Ключевые слова: тонкая пленка, магнетронное распыление, рентгеновское излучение, оптическое поглощение, показатель преломления

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ОПТИЧНІ ВЛАСТИВОСТІ ОПРОМІНЕНИХ РЕНТГЕНІВСЬКИМИ ПРОМЕНЯМИ ТОНКИХ ПЛІВОК НА ОСНОВІ Cu_6PS_5I, НАНЕСЕНИХ МАГНЕТРОННИМ РОЗПИЛЕННЯМ

Тонкі плівки на основі Cu_6PS_5I, нанесені методом магнетронного розпилення, опромінювалися при різних часах експозиції з використанням широкосмугового випромінювання мідного аноду рентгенівської трубки. Вивчено спектри оптичного пропускання, урбахівський край оптичного поглинання та дисперсію показників заломлення опромінених рентгенівським випромінюванням тонких плівок $Cu_{5.6}P_{1.7}S_{4.9}I_{0.8}$, $Cu_{6.4}P_{1.1}S_{4.6}I_{0.9}$ та $Cu_{8.0}P_{0.7}S_{3.6}I_{0.7}$. Виявлено зменшення

енергії псевдозабороненої зони та збільшення показника заломлення при збільшенні часу рентгенівського опромінювання.
Ключові слова: тонка плівка, магнетронне розпилення, рентгенівське випромінювання, оптичне поглинання, показник заломлення.

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