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Mechanical properties of photosensitive $AgInS_2$ sprayed thin films annealed under various atmospheres and temperatures

D.Gherouel, K.Boubaker*, M.Amlouk

Unité de Physique des Dispositifs à Semi-conducteurs, Faculté des Sciences de Tunis, Tunis El Manar University, 2092 Tunis, (TUNISIA)

ABSTRACT

This work aims to investigate the influence of heat treatment of photosensitive $AgInS_2$ sprayed thin films on the mechanical and structural properties. $AgInS_2$ ternary material has several important properties (Bandgap, electric resistivity, transmission–reflection spectra...) that guide it's to choice as an absorber in solar cells domain[1,2]. Hardness measurements have been carried out to determine the Vickers hardness of these films. The results revealed that annealing treatment enhances drastically the mechanical resistance to frontal penetrating solicitation (Hv hardness) may be due to the partial crystallization and phase transformations modified the microstructure of the based glassy material. The maximum gain was measured for samples treated at 400°C, having a hardness of 8.31GPa. However, when the annealing temperature was upper than 450°C the hardness is decreased may due to bulk defaults in the structure of the films which it is confirmed by XRD and AFM investigations.

INTRODUCTION

$AgInS_2$ thin films have been investigated for their interesting optical, mechanical and electrical performances. This paper deals with the fabrication of $AgInS_2$ thin films and the annealing effect on their mechanical properties. Commonly, $AgInS_2$ could be synthesized using several methods like microwave heating technique[3], electrodeposition process[4], sulfurization of Ag–In[5], thermal annealing of In_2S3–Ag2S[6] and chemical spray pyrolysis (SP)[7]. In this paper, the spray pyrolysis method is selected to prepare these layers. After that, the same films are subjected to a heat treatment using various temperatures (400°C, 450°C, 500°C) under two atmospheres (Selenium and air) for 1h in a programmable tubular and investigated a global approach to identify physical constants. Recently, we have determined the optical, thermal and structural properties of such films[1,2].

In this paper we investigate the mechanical parameters to propose as guides for industrial application. To date, the mechanical measurements of such ceramics have not yet studied.

CHARACTERIZATION TECHNIQUE

The mechanical properties were investigated along with hardness measurements which have been performed in the MA2I Laboratory (ENIT, Tunisia)[8]. This technique is based on hitting the targeted face of each sample by a common diamond-pyramidal-indenter under a prefixed load. The obtained imprints dimensions were subjected to geometrical standardized analyses yielding a scaled value: the Micro-Hardness Vickers (Hv).

The micro hardness Vickers *(Hv)* is giving by:

$$Hv = \frac{2\sin\alpha.F}{d^2} = \frac{1{,}854\ F}{d^2}(Kg.mm^{-2}) \quad (1)$$

with : $\alpha = 68$ °, F : the weight and d : the lattice parameter of the lozenge

Figure 1 : scheme of measurement technique of Vickers micro hardness

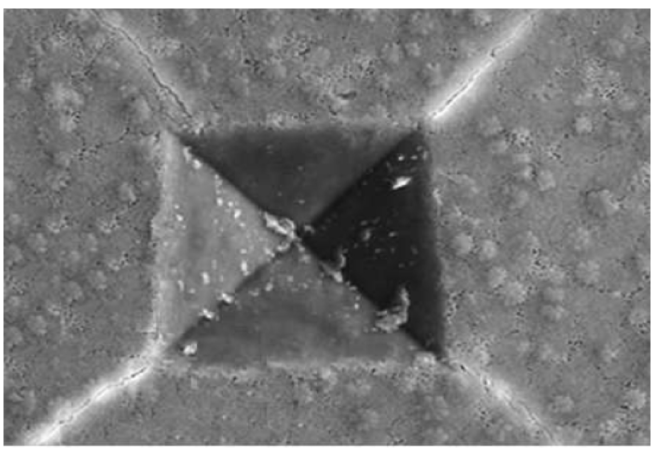

Figure 2 : Micrograph of a Vickers' indentation mark in sample $AgInS_2$

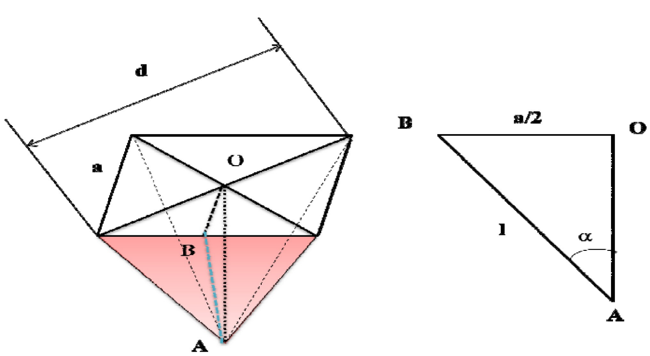

Figure 3 : The synoptic scheme of the micrograph of a Vickers' indentation mark

RESULTS AND DISCUSSIONS

XRD and AFM investigations on $AgInS_2$ thin films annealed in Se and in air

XRD patterns, 2D AFM morphological micrographs as well as the rms roughness and crystallite size of the annealed $AgInS_2$ sprayed thin films in air and in Se atmospheres using various temperatures lying in (400-500°C) domain for 1h in a programmable tabular were shown in Figures 4,5 and 6 have been the subject of recently works[1,2]. The XRD shows the crystallization in a tetragonal structure of $AgInS_2$ with the strong peak (112) and the presence others undesirables binaries phases such as In_2S_3 and Ag_2S. The same results have been obtained by the others authors[9-15]. In this study, it is noted that suitable structure of $AgInS_2$ thin films is reached using 450 and 400°C as appropriate heated temperatures for Se and air atmospheres respectively. For these latest treated films, minor secondary and undesirable phases are found.

Micro-hardness vickers (Hv) measurements

The targeted face of each sample was subjected to static indentation tabes1and 2 and Figure 7 at room temperature using a Vickers diamond pyramidal indenter (squared 136 ° summit angle pyramids). The curves traduce the fact that for low charge supply (m= 0.3Kg), the microhardness is lying in 3-8 GPa domain. This charge value is used to ovoid the contribution of the glass substrate due to the penetration of the diamond

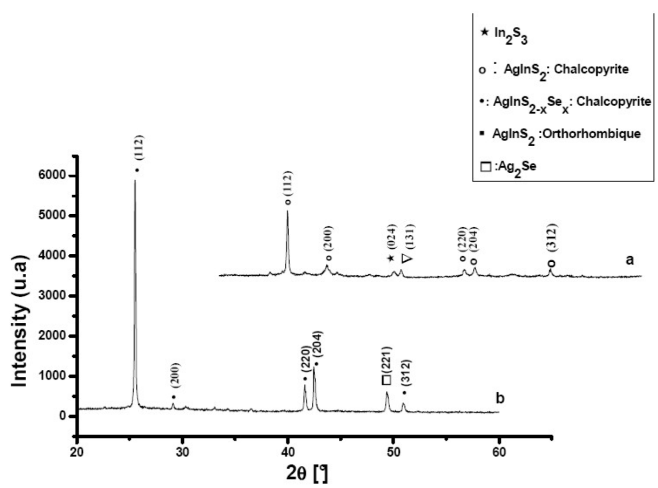

Figure 4: Typical X-ray diffractograms of AgInS2 annealed thin films in Se and air at 450C° and 400°C respectively corresponding to optimal conditions.

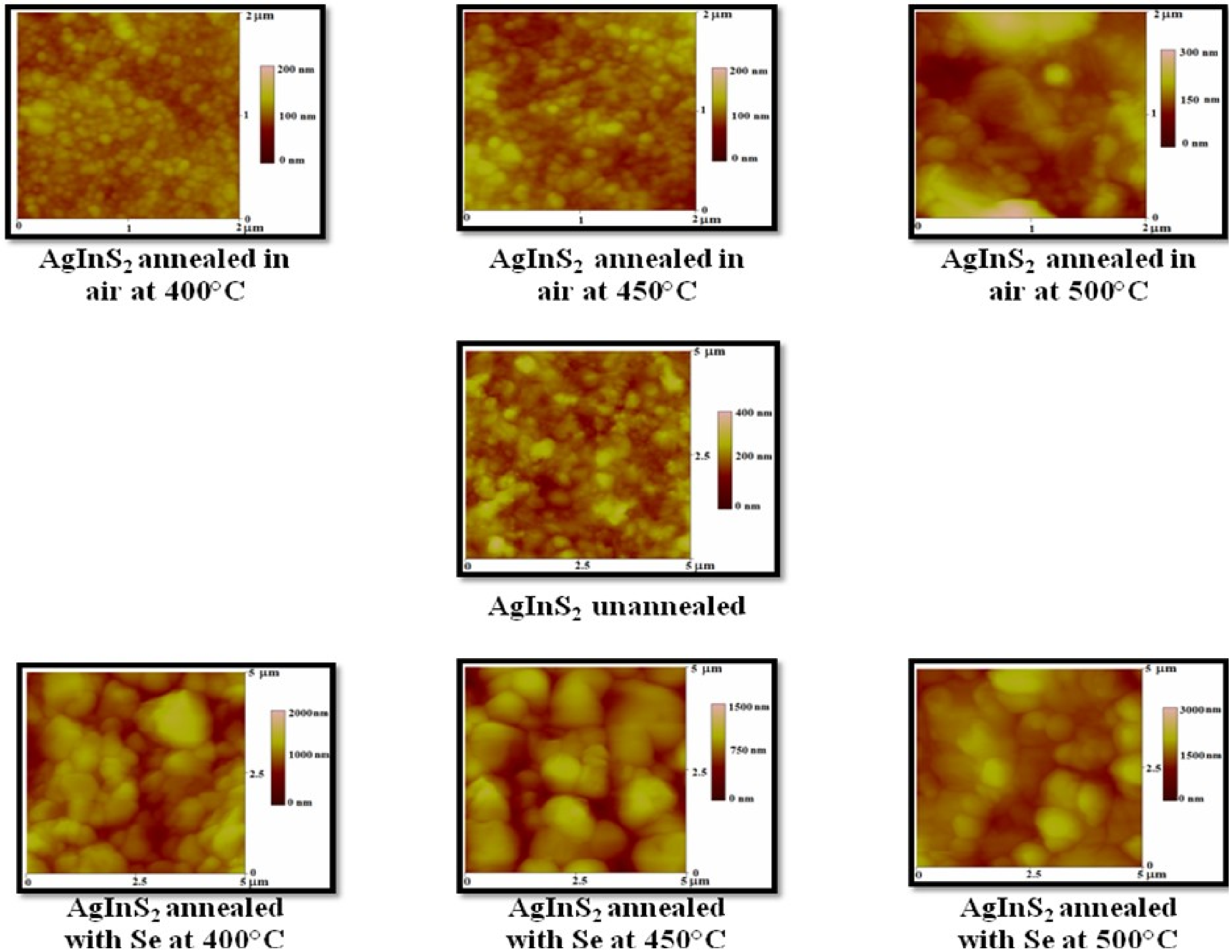

Figure 5 : Comparison of the AFM (2D) micrographs of AgInS$_2$ annealed in air and in Se atmosphers at various temperatures

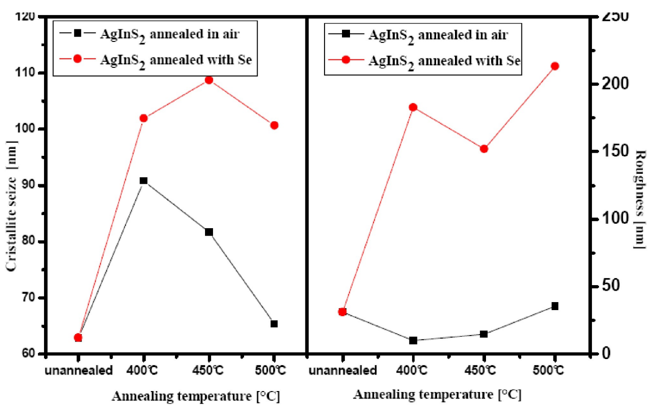

Figure 6 : Comparison of the roughness and the crystallite size of AgInS$_2$ thin films heated at various temperatures under two different atmospheres

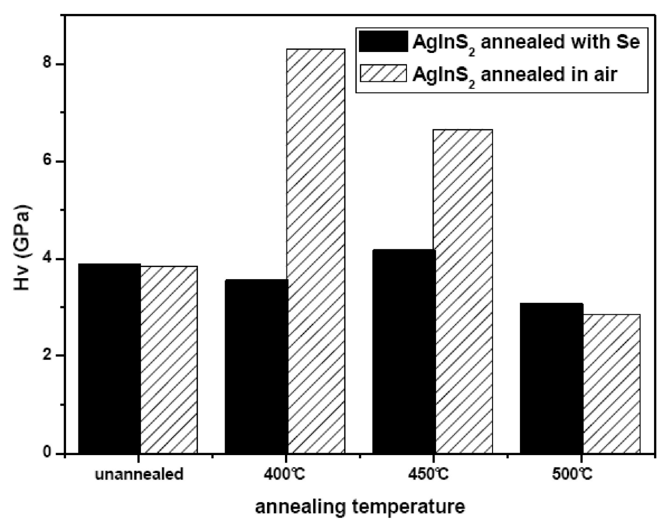

Figure 7 : Variation of Hv of AgInS$_2$ annealed at various temperatures

pyramidal indenter beyond the layer[14].

From the results plotted in Figure 6, it is noted that best values of roughness as well as the crystallite size have been reached related to AgInS2 thin films heated at 450°C under Se atmosphere and those treated at 400°C in air.

TABLE 1 : Values of Vickers hardness (Hv) of $AgInS_2$ films annealed with Se

	$AgInS_2$ unannealed	$AgInS_2$ annealed with selenium		
		400°C	450°C	500°C
Hv (GPa)	3.85	3.54	4.15	3.06

TABLE 2 : Values of Vickers hardness (Hv) of $AgInS_2$ films annealed in air

	$AgInS_2$ unannealed	$AgInS_2$ annealed in air		
		400°C	450°C	500°C
Hv (GPa)	3.85	8.31	6.66	2.86

The variation level of microhardness in terms of the heat temperature suggests the beneficial effect of this process to increase the hardness of these ternary ceramics. Indeed, the treatment under Se especially at 450°C (4.15 GPa) as well as at 400°C (8.31GPa) in air reinforce the hardness of these films and increased the surface as well as bulk structure by a possible molecular motion via a partially taking Se/O the place of S and minimized the porosity and voids and cracks inside the films. This behavior has been obtained by other authors in others compounds under appropriate conditions[15-19]. This treatment using such temperature leads to an enough solicited change in the crystalline state of $AgInS_2$ material. This mechanical study is consistent with XRD and AFM results described above.

CONCLUSION

The Micro-Hardness Vickers (Hv) of the $AgInS_2$ sprayed thin films annealed in air and with selenium are increased with the increase of the annealing temperature may be due to the improvement of the structure of heated films under air and Se atmospheres using appropriated temperatures. This study seems to be so interesting since a macro characterization like Hardness vickers is used as a powerful technique to evaluate the physical quality of ternaries ceramics based on Ag-In-S(Se,O).

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