IOSR Journal of Electronics and Communication Engineering (IOSR-JECE)
e-ISSN: 2278-2834,p- ISSN: 2278-8735.Volume 13, Issue 5, Ver. I (Sep.-Oct. 2018), PP 54-59
www.iosrjournals.org

Structure and temperature profile limits of optic fiber chalcogenide material based selenium

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Abstract: *The X-ray diffraction pattern of the system $Se_{80}S_{20-x}B$ where (x=0,2.5,5 and B=In or As) confirms the amorphous nature of the bulk and thin film of all samples. The addition of In or As to $Se_{80}S_{20}$ on the expense of S confirms this result.Also, the non-uniformity of SEM micrographs confirm the amorphous nature of the samples under test. The SEM micrographs of all samples after addition of In or As on the expense of S and even the addition of Er cover layer reveals no change in the amorphous structure. The thermal differential analysis (DTA) proves that the glass transition range is wide as 60^oC with onset temperature 106^0C for the sample $Se_{80}S_{20}$. This range becomes narrow as In or As replacing partially S. The addition of In on the expense of S reduces this range to be in the range 15^0C-20^0C. Also, replacing In by As this range becomes only one degree. The crystallization temperature (T_C) of the sample $Se_{80}S_{20}$ was 233^0C. The addition of In to $Se_{80}S_{20}$ on the expense of S reduced T_C to be 206^0C- 211^0C depending on the In ratio. The replacing of In by As with the same ratio decreases T_C to be 180^0C and 140^0C as As increases from 2.5 at % to 5 at %, respectively. The melting temperature range was $30^0C(530^0C$-$560^0C)$ for the sample $Se_{80}S_{20}$. This range decreased to be 15-20^0C as In replacing S partially. Finally, the thermal profile of the given chalcogenide material based selenium is a good selection to the production of optic fiber material to serve the international net communication well.*

Date of Submission: 08-10-2018 Date of acceptance: 22-10-2018

I. Introduction

Chalcogenide materials doped with various rare earth (RE) are extensively studied as potential materials for fiber optic amplifiers(1,2). The low phonon energy, good thermal stability, easy fiber fabrication and extended IR transmission had made chalcogenide glasses suitable host materials for rare earth ions (2). Chalcogenide glasses doped with rare earths are potential candidates for mid-infrared lasers for a variety of applications including environmental sensing, LIDAR and military counter measure ,powerful ,coherent,robust and compact sources. Sulfide, Selenide and Telluride glass have been shown to be chemically and mechanically durable to posses wide glasses forming regions and to have extended infrared transparency windows (3-5). Photoluminescence studies in bulk and thin film samples reports the overlap of optical transitions involving absorption and emission bands due to discrete levels of RE ions with conduction band and edge states of host materials (6,9). The efficiency of the luminescence is found to increase high at mid IR wavelength (7,9). Due to the high refractive index of these glasses, they exhibit high optical non-linearity(8-11). The aim of this work is to study and illustrate the structure and temperature profile limit of the system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or As) to be used as optic fiber cables covered with erbium(Er) cover layer.

II. Experimental technique:-

The chalcogenide samples of the system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or As) were prepared by melting quenching technique. Elements Se, S, In& As were weighted and mixed well using the ball milling method for each sample alone. The homogeneous mixture was placed in an evacuated (10^{-4} Pa) and capsulated silica tube. The silica tube containing each sample was heated at fixed temperature for fixed time. The sample $Se_{80}S_{20}$ and the samples contain In on the expense of S were melted at 500^0C for 8hours and quenching in Ice water. The samples containing As on the expense of S were melted at 800^0C for 8 hours and then quenching in ice water. The thin film samples were prepared from the bulk ingots using laser ablation on glass substrate. This was done using Nd:YAG pulsed laser deposition of wavelength 532nm under $55*10^{-4}$Pa vacuum, laser ablation was used under the same condition to cover each sample by Er layer. The thickness of the obtained thin films was determined using interferometry method. The structure of the obtained thin films was found to be amorphous as detected by X-ray hump and the non-homogenous SEM Images(fig(1,2)&(3,4)).The thermal profile of each sample was recorded using DTA analyzer ShimadZU DTA50 Japan at 10 degree/min heating rate.

III. Result and discussions:-

i. X-ray diffraction:-

Fig [1] shows the X-ray diffraction pattern of the bulk samples of the system $Se_{80}S_{20-x}B$ where (x=0,2.5,5 and B=In or As). The result of fig [1] confirms the amorphous nature of the structure of all samples. This was revealed from the intense hump centered at $2\Theta=25^0$. The other less intense hump may be due to non-homogeneity in the powder mix of the samples. Fig [2] confirmed the amorphous nature of the thin film samples under test. The absence of the second hump in the case of thin film samples may be due to homogeneity of the sample thickness. Fig [3] shows the X-ray diffraction pattern after covering each sample surface by Er cover layer. The detected hump of fig [3] is characterized by low relative intensity, but with wide half width. This result confirms the amorphous nature of the Er cover layer as well as its formation in the nano-scale.

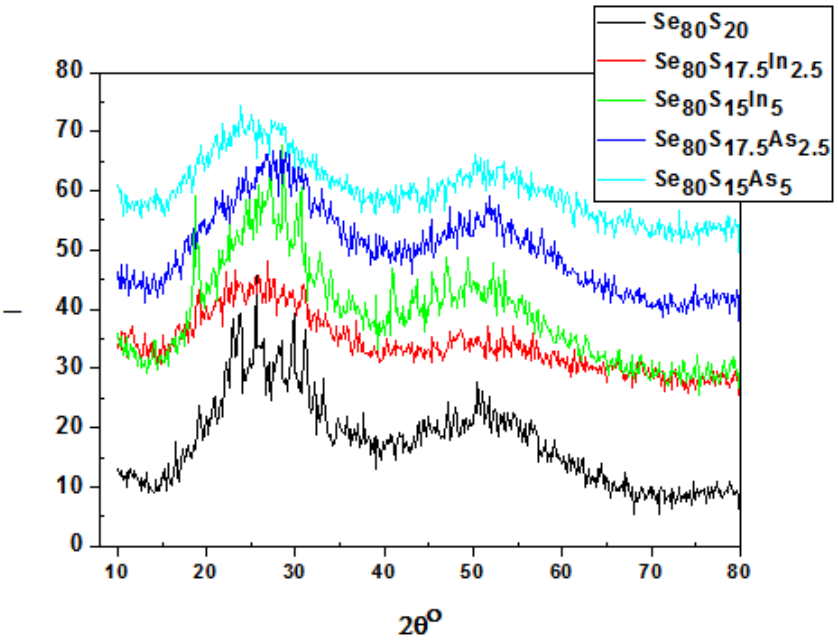

Fig [1] X-ray diffraction patterns of system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or AS) as bulk samples .

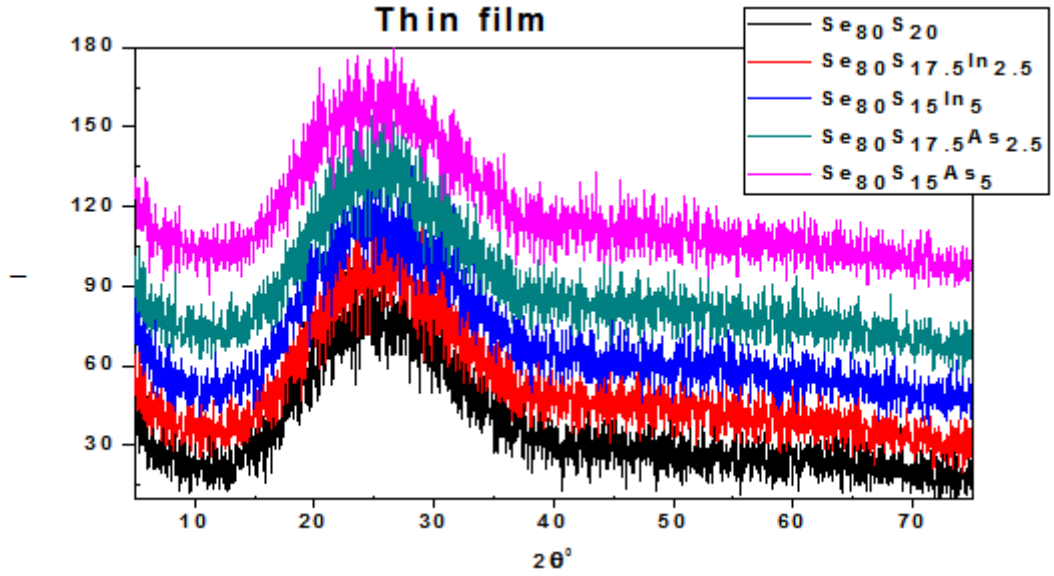

Fig [2] X-ray diffraction patterns of system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or AS)thin film deposited on a glass substrate.

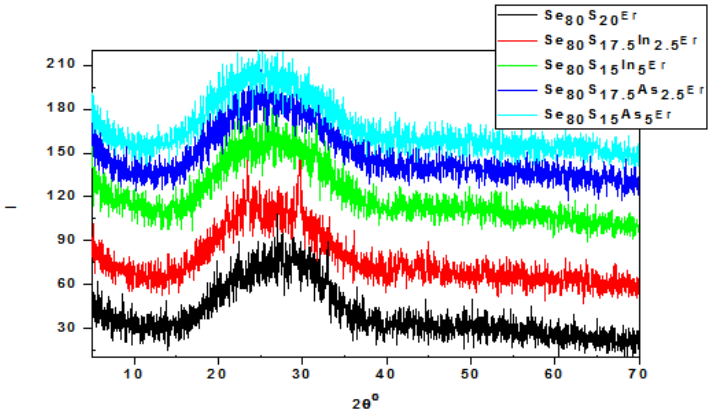

Fig [3] X-ray diffraction patterns of system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or AS)thin film after covering Er layer.

i. Scanning electron microscopes micrographs(SEM)

The non-uniformity of SEM micrographs confirmed the amorphous nature of the thin film samples structure of this system fig [4]. These results are a good support to the X-ray result. The addition of In or As on the expense of S with the same ratio enhance the ordered condition of the amorphous samples nature. Fig [5] show the addition of the Er cover layer to the thin film samples surfaces enhance its reflectivity as its surface become more smoothing.

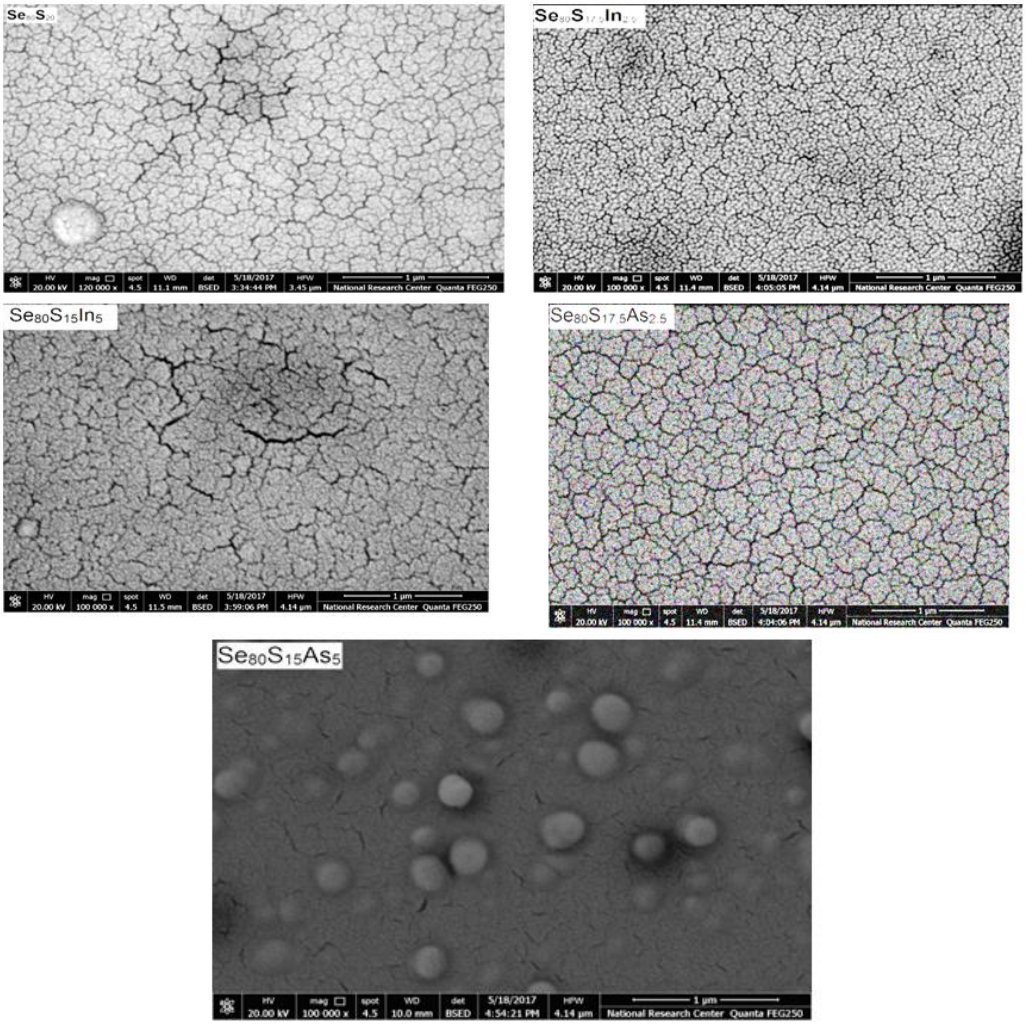

Fig [4] scanning electron micrograph image of chalcogenide system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or AS) thin film.

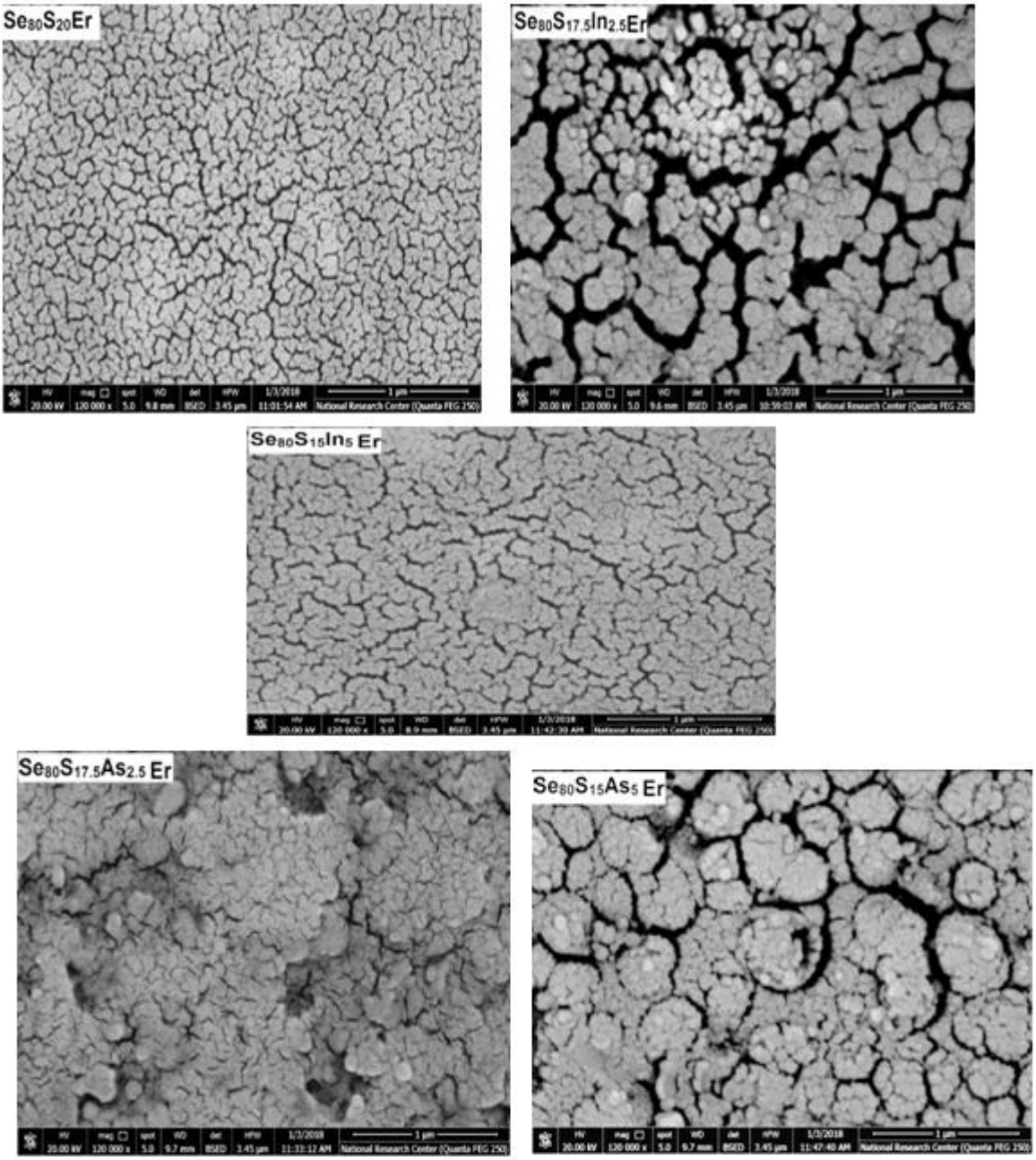

Fig [5] scanning electron micrograph image of chalcogenide system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or AS) thin films covered with Er layer

ii. The temperature profile limits:-

Fig[6] shows the differential thermal analysis (DTA) thermograms of the chalcogenide samples of the system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or As) recorded at heating rate 10 degree /min.

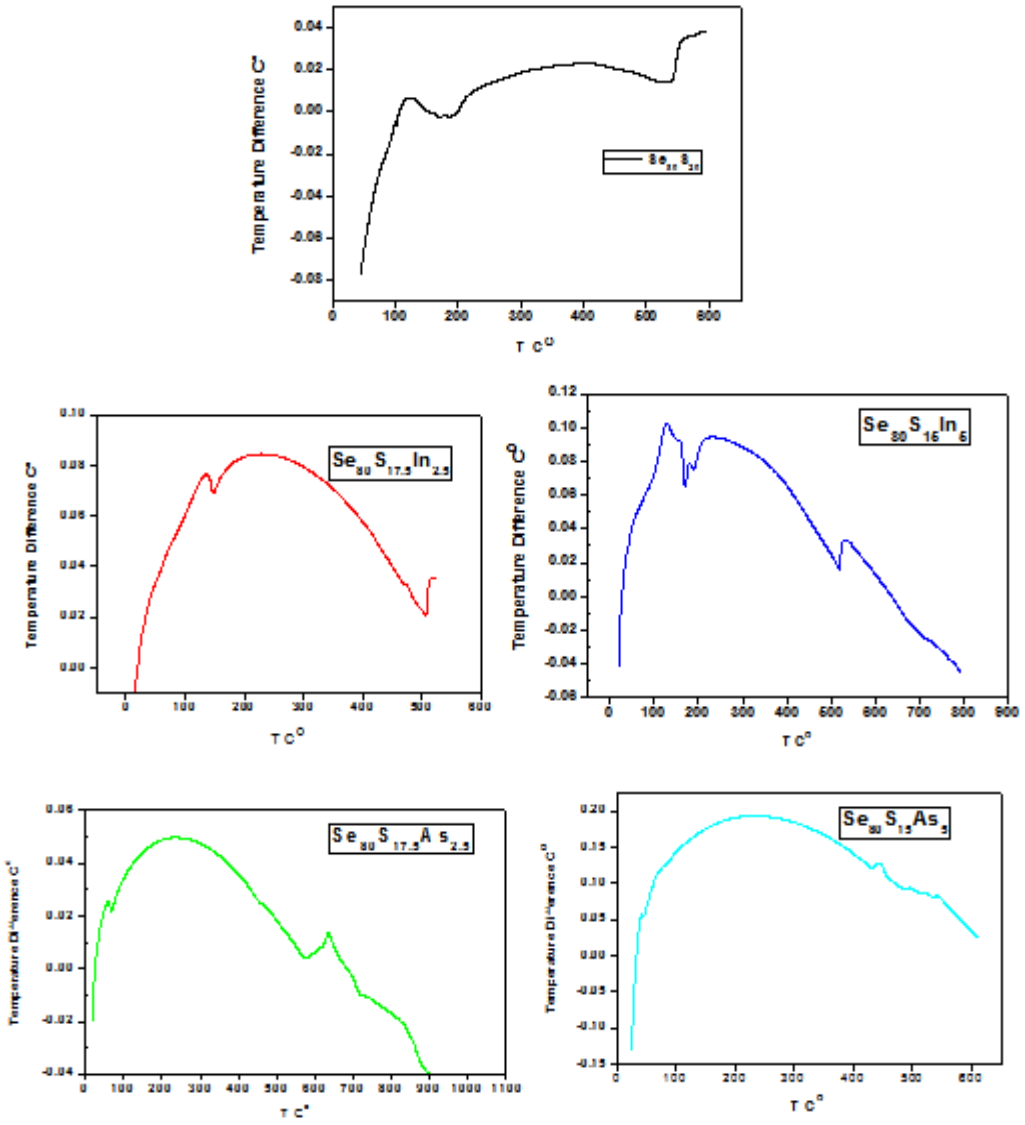

Fig [6] DTA thermograms of chalcogenide system $Se_{80}S_{20-x}B_x$ where (x=0,2.5,5 and B=In or As.

The obtained values of the temperature limits for each stage were gathered together and tabulated in table[1].

Table [1]

Samples	Onset T_g c°	offset	T_C C°	Onset T_m c°	offset
$Se_{80}S_{20}$	106	162	233	530	560
$Se_{80}S_{17.5}In_{2.5}$	126	150	211	496	511
$Se_{80}S_{15}In_5$	136	155	206	502	522
$Se_{80}S_{17.5}As_{2.5}$	48	49	180	580	600
$Se_{80}S_{15}As_5$	37	38	140	455	470

The results of table [1] show that the glass transition temperature range of the sample $Se_{80}S_{20}$ was 106^0C-162^0C, with onset- offset temperature difference 60^0C. The addition of 2.5 at % In to $Se_{80}S_{20}$ on the expense of S reduce this range to be 25^0C.This range becomes 20^0C as In addition become 5 at %.

The replacement of In by As with the same ratio to the sample $Se_{80}S_{20}$ reduce the glass transition temperature range to be only one degree. This means that, the addition of In or As to the sample $Se_{80}S_{20}$ on the expense ofSsave the energy of disorder – order transformation. The detected crystallization temperature (T_c) was 233^0C for the sample $Se_{80}S_{20}$. Adding 2.5 at % or 5 at % In decreases T_c to be 211^0C and 206^0C respectively. On the other hand, replacing In by As leads to decrease T_c to be 180^0C and 140^0C as As increased from 2.5 at % to 5 at % respectively. According it is clear that , the addition of In or As to $Se_{80}S_{20}$ save energy of melting process.

Also, the results of table [1] show that, the melting temperature range is 30^0C (530^0C-560^0C) for the sample $Se_{80}S_{20}$. This range was decreased to be in the range 15-20^0C as In added on the expense of S by the ratio 2.5 at%(502^0C-522^0C) .

The replacing of In by As with same ratio lead to change this range to be 15-20^0C. The interesting result is that the glass transition range of the chalcogenide optic fiber material based selenium is wide enough to keep it in the glassy state , which is low cost. On the other hand, the melting temperature range is suitable since its onset temperature is high and in turn save.

Generally , the thermal profile of the selected chalcogenide material based selenium is a good selection to be used as an optic fiber material to be used all over the world without any drawbacks.

IV. Conclusion

The X-ray diffraction pattern confirm the amorphous nature of all samples under test and after adding Er cover layer. The SEM micrographs confirm this fact. The Er cover layer increases the smoothing of the samples surfaces and increase its reflectivity to restore the losses of the light within the optic fiber cables. The temperature profile limits illustrate that the addition of In to the sample $Se_{80}S_{20}$ on the expense of S reduces the glass transition range to be in the range 20-25^0C depending on the In ratio. Also, replacing In by As with the same ratio reducing this range to only one degree. The crystallization temperature of the sample $Se_{80}S_{20}$ was 233^0C. This temperature becomes 211^0Cand 206^0C as In replacing S by the ratios 2.5 at % and 5 at % ,respectively. The replacement of In by As with the same ratios, reducing T_c to be 180^0C&140^0C respectively. The melting temperature range was 30^0C(530-550^0C) for $Se_{80}S_{20}$. Adding In or As on the expense of S leads to decrease the range to be in the order of 15-20^0C depending on the additives type and its ratio. Accordingly , the amorphous nature of the optic fiber cables together with the temperature profile of the chalcogenide selenium based material must be a good candidate to serve and save the international communication net all over the world.

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M.M.El-zaidia. " Structure and temperature profile limits of optic fiber chalcogenide material based selenium." IOSR Journal of Electronics and Communication Engineering (IOSR-JECE) 13.5 (2018): 54-59