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Growth and Structural Characterization of Semiconducting Tin Telluride Thin Films by Novel Rapid Thermal Annealing Technique

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Abstract:-Tin Telluride is a representative IV-VI compound semiconductor of narrow band gap with potential use in IR detector. Thin films of SnTe were grown by a novel, cost effective rapid thermal annealing (RTA) technique. For this, elemental layers of Sn and Te were deposited on a quartz substrate and then subjected to rapid thermal annealing at optimized temperature and duration. The required experimental set up was designed and fabricated. Single phase formation of SnTewas confirmed from X-ray diffraction technique. The crystalline size were calculated. The duration and temperature of annealing were optimized. Further, the required excess Te-composition for single phase stoichiometric SnTe film was also optimized. It has been shown that thin films of single phase SnTe can be grown by rapid thermal annealing.

Index Terms: *Compound semiconductor, Thin films, Annealing, XRD, Conductivity*

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I. INTRODUCTION

The narrow band gap IV-VI compound semiconductors found various applications, as it is easy to make solid solutions of these compounds due to their low melting point and high solubility for metals of group IV [1]. SnTe is a representative IV-VI compound semiconductor of narrow band gap with potential use in its solid solutions with PbTe as IR detector [2,3].

SnTe thin films have been grown by various techniques like molecular beam epitaxy (MBE) [4],thermal evaporation [5],chemical reduction [6], solution-phase synthesis [7] etc. SnTe, when prepared from stoichiometric composition, is always deficient in tin due to the low chemical diffusion constant of Sn in SnTe below 300 ^{o}C, and the material is therefore always p-type extrinsic [8]. The presence of a large number of lattice defects, associated with the variation from stoichiometry, can cause considerable change in the electrical properties of SnTe thin films. To reduce Sn vacancies and to achieve stoichiometric SnTe composition of high quality, tin is added onto the samples normally by diffusing tin during annealing of the samples at high temperatures.The present work deals with the synthesis of thin films of SnTe with different stoichiometric ratio of Sn:Te as 1:1.2, 1:1.3 and 1:1.4 followed by different annealing conditions. Structural characterizations were performed to establish the formation of single phase SnTe. Annealing temperature and time were optimized.

II. EXPERIMENTAL

Thin films of SnTe were prepared by vacuum evaporation through resistive heating. For this, elemental layers of 5N pure Sn and Te were deposited onto quartz substrate at 150^{o}C in a vacuum of 5x 10^{-6} torr. The thickness of the film was controlled by a thickness monitor after optimizing distance between boat and substrate. After the deposition, the films were subjected to rapid thermal annealing in a specially designed setup as detailed in Fig.1. The basic requirements of RTA process viz. controlled rate of heating and cooling, control over heating duration, smooth movement of the sample in the heating zone, a non-reactive local surroundings, etc. are fulfilled as the film could be swiftly bring in to a high temperature region (400-600 ^{o}C) for a desired duration and could be taken out at desired rate. Structural characterization was performed by powder XRD technique using CuK$_{\alpha}$ radiation (λ= 1.5404 Å).

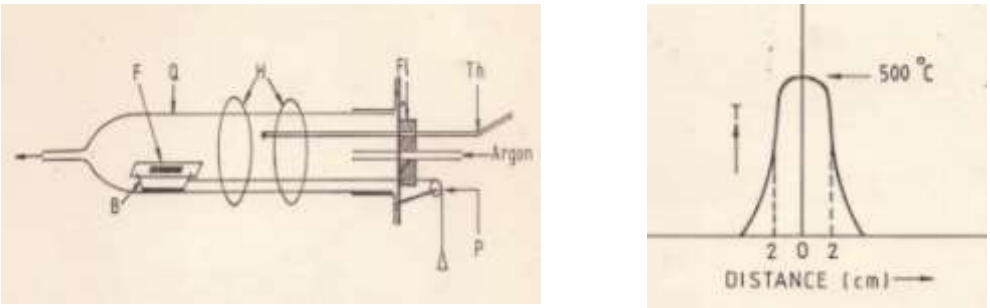

Figure 1: Experimental set up for rapid thermal annealing and its temperature profile. Q:Quartz tube with a provision of Argon gas flow, B: Quartz boat which can be moved swiftly with the help of weight and pulley(P) arrangement, H: two circular resistive heaters, F: SnTe Film, Fl: Flange, Th: Thermocouple

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III. RESULTS AND DISCUSSION

A large number of films with different thickness in the range of 0.4 to 0.8 µm and with different Sn:Te ratios were grown. XRD studies were made on all the films as a function of duration and temperature of RTP. CuK_{α} radiation was employed and a 2θspectrum in a range of 20 to 50 degree was recorded. In Fig. 2, curve (A) shows a typical XRD spectrum for as grown tin telluride precursor (thickness – 0.4 µm) with stoichiometric composition. It can be observed that there is only one peak at 2θ =26.3^{o}, corresponding to Te-phase, indicating that there is no formation of tin telluride under these conditions of deposition prior to RTA process.

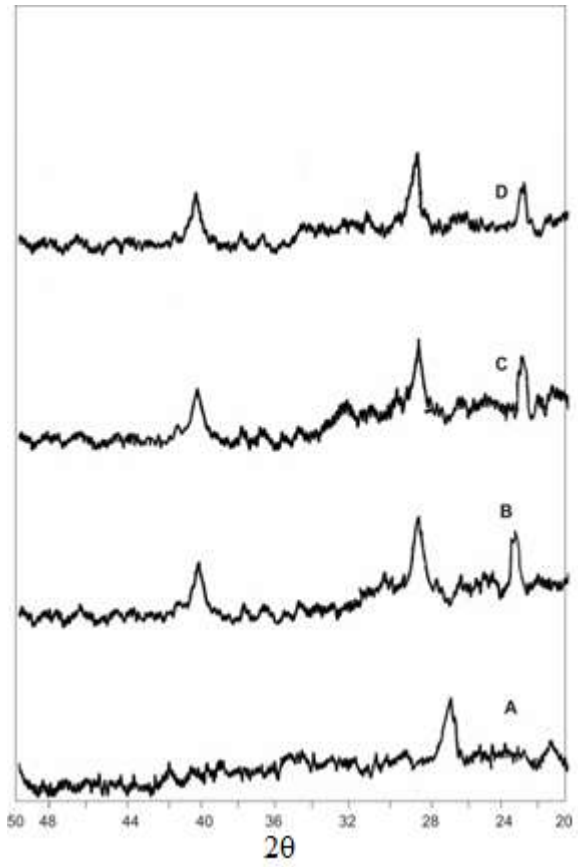

Fig. 2 XRD pattern of (A) as grown SnTe precursor, (B), (C) & (D) SnTe film thermally annealed at 450, 500 and 550 ^{o}C respectively for 30 sec.

Fig 2 (B,C,D) show the effect of annealing temperature on the structure and composition of the films. All these films were annealed for a fixed duration of 30 sec. at 450, 500, and 550^{o}C respectively. It can be observed that the annealing resulted in the occurrence of three peaks at 2θ = 23.2, 28.3 and 40.4 degrees. The peaks at 28.3 and 40.4 degrees correspond to the 200 and 220 plane of SnTe. This can be confirmed by estimating the lattice constants of SnTe by the relation,

$$d= a/\{ h^2 + k^2 + l^2\}^{1/2} \text{ ---------- (1)}$$

Where h, k and l are the indices of reflection and d is related to the Bragg angle θ as nλ = 2d sinθ and λ being the x-ray wavelength. The value of 'a' was estimated as 6.3 Å using 2θ values of 28.3 and 40.4 degrees, corresponding to h, k , and l values of 200 and 220 respectively, which is in agreement with the reported value of single crystals of SnTe [1]. It may be mentioned that the longer duration of RTA, particularly at temperature higher than 550 ^{o}C, always resulted in loss of material and the films become extremely thin. Moreover, at temperature greater than 600 ^{o}C, annealing even for 30 sec resulted in the loss of material. On the other hand, annealing below 400 ^{o}C was unable to produce SnTe phase as peaks at 28.3 and 40.4 degrees were not visible.

The peak at 23.2 degree, however neither corresponds to any phase of SnTe, nor does it correspond to individual elements viz. Sn or Te. This suggests that there is a formation of intermediate phase Sn_xTe_y also in addition to pure SnTe phase. The presence of intermediate phases like Sn_xTe_y are presumably rich in Sn, because of the fact that tellurium has higher vapor pressure as compared to tin. This was confirmed by growing the films from Sn as Te excess precursors. The films grown from excess Sn always resulted in the extra peak at 23.2 degree, whereas the films grown from the excess Te was free from this peak. In order to grow single phase SnTe films, the base precursor films were fabricated with excess of Te. Various compositions of SnTe in the ratio of 1:1.1 to 1:1.5 were tried. The films grown from less than 1:1.2 always resulted in a peak at 23.2 degree, whereas films grown from composition greater than 1:1.4 resulted in additional peaks corresponding to Te. The only compositional range which resulted in single phase SnTe was 1:1.2 to 1:1.4. Fig 3 (A), (B), (C), and (D) show the effect of increasing annealing temperature on the structure of films with Sn:Te as 1:1.4. These films were annealed for a fixed duration of 30 sec. at temperatures 450,500,550 and 600 ^{o}C, respectively. It can be observed that the films were always single phase with preferred orientation of 200 and 220 planes. The peak height, however, increase and the peak width decreases with the increase of annealing temperature, indicating that the RTA temperatures improves the crystallinity of the thin films.

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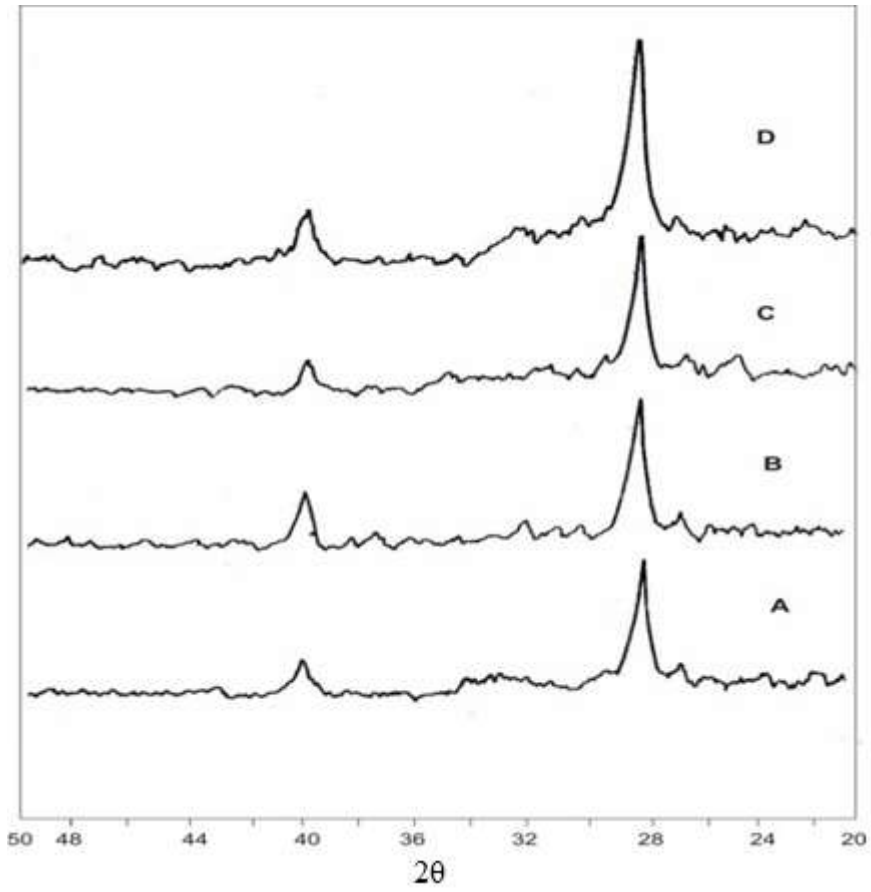

Fig.3 XRD of SnTe film with excess of Te (Sn:Te= 1:1.4). These films were annealed at different temperatures (A) 450^{o}C, (B) 500^{o}C, (C) 550^{o}C and (D) 600 ^{o}C.

The grain size of these films was estimated using well known Scherer relation

$$L = \lambda/D \cos \theta \text{----------(2)}$$

Where L is the grain size and 'D' is the width of the peak at half maxima.The values of the grain size estimated from different planes at different RTA temperatures are given in Table 1.

Table 1

RTA temperature (^{o}C)	Grain size (Å) (200)
450	230
500	303
550	303
600	303

D.C. Conductivity

The d.c. conductivity of all the films was measured as a function of temperature in the range liquid nitrogen - room temperature. The variation of d.c. conductivity (lnσ vs 1/T) for the films grown by RTP with Sn:Te ratio as 1:1.2, 1:1.3 and 1:1.4 are shown in the above figure as A, B and C respectively. The results of similar studies with the film grown by conventional evaporation technique having a composition 1:1.3. is depicted in curve D.

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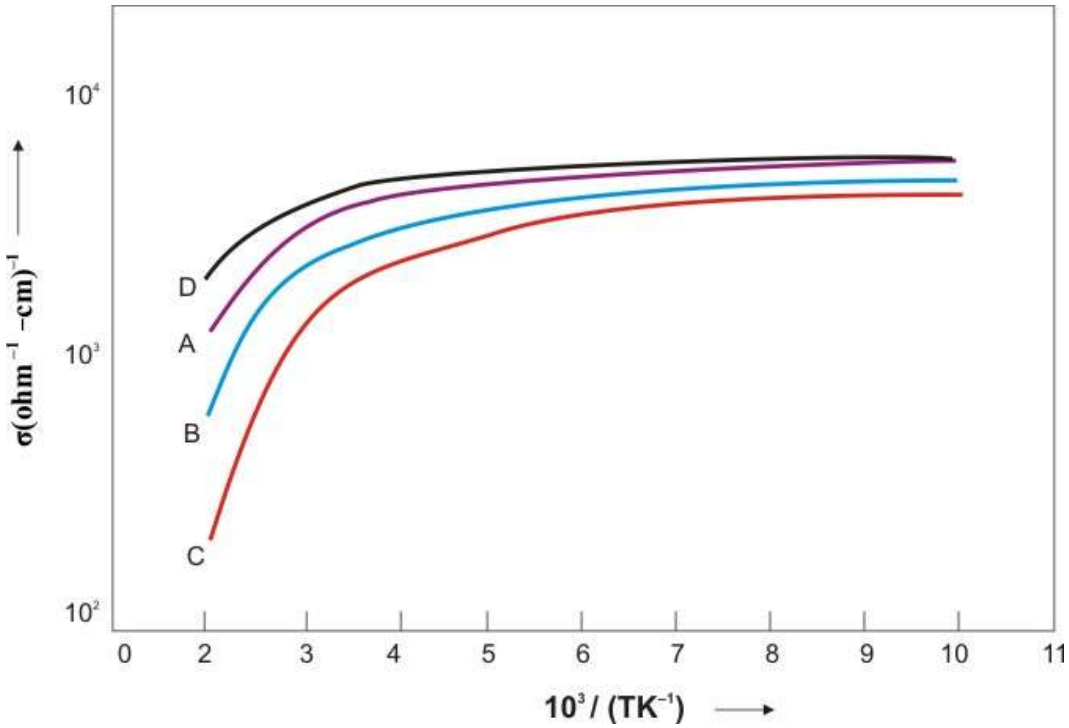

Fig.4. dc conductivity of SnTe films grown by RTA technique with initial ratio of Sn:Te as (A) 1:1.2, (B) 1:1.3 and (C) 1:1.4 . (D) SnTe film grown by conventional thermal deposition technique with Sn:Te ratio as 1:1.3.

As shown in the figure, the conductivity is observed to be a weak function of the carrier concentration in the low temperature region. However, in the high temperature region, a significant increase in 'σ' occurs with increasing carrier concentration i.e. with decreasing tin content. This result is understandable because the concentration of light holes in the films with higher content of tin is less and therefore the effect of heavy holes, which have lower mobility will be more pronounced. It is also observed from the figure that the onset of heavy hole conduction starts at a lower temperature in the films with higher tin contents.

IV. CONCLUSION

It has been demonstrated that a simple RTA technique can be used to grow SnTe film. The optimized annealing temperature range is 450-550 ^{o}C for annealing duration 30 sec. A lower temperature does not support formation of SnTe phase while higher temperature resulted in loss of material. Further, stoichiometric SnTe always resulted in Te deficient films. For single phase SnTe, an excess of Te with a starting composition Sn:Te as 1:1.2 to 1:1.4 was required. The dc conductivity of RTA grown films are comparable to those in grown from conventional thermal evaporation technique. In conclusion, it has been shown that single phase SnTe films can be grown by a simple RTA technique.

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