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Study of annealing effects in Al–Sb bilayer thin films

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Abstract. In this paper, we present preparation and characterization of Al–Sb bilayer thin films. Thin films of thicknesses, 3000/1000 Å and 3000/1500 Å, were obtained by the thermal evaporation (resistive heating) method. Vacuum annealing and rapid thermal annealing methods were used to mix bilayer thin film structure. Results obtained from optical band gap data and Rutherford back scattering spectrometry showed mixing of Al–Sb bilayer system.

Keywords. AlSb; thin film; RBS; optical band gap.

1. Introduction

The III–V semiconductors are of great importance due to their applications in various electro–optical devices (Yu *et al* 1995). The semiconducting compound, Al–Sb, with an energy band gap of 1·62 eV is potentially a high efficiency solar material (Rittner 1954). The flash evaporation technique has been employed to grow Al–Sb films by Richards *et al* (1963). The stack elemental layer deposition method in case of Zn–Se films recently used (Singh and Vijay 2004a) suggested that RTA can be used for preparation of semiconductors on the basis of optical, electrical and structural studies of intermixing thin films. The thermal evaporation method in case of In–Sb used (Singh and Vijay 2004b) suggested that by annealing process intermixing is possible to prepare semiconductors. The isochronal heat treatment of bilayer results in the interdiffusion and reaction between the elements accompanied by consequent nucleation and growth (Raghvan *et al* 2000).

The RBS studies carried out by several workers to confirm mixing of bilayer (Holloway and Sinclair 1987; Niwa *et al* 1998; Kraft *et al* 2002; Dhar *et al* 2003), suggest that peak to valley ratio in the annealed sample decreases, which shows the intermixing of elements. The RBS studies of ion beam mixing bilayer carried out by many researchers (Niwa *et al* 1998; Kraft *et al* 2002; Dhar *et al* 2003), suggested phase transformation with ion doses. The active role of grain boundary and defects migration on annealing would be expected to occur during crystallization (Bhargava 1997). The increase in optical transmittance was related to increasing the substrate temperature (Kim *et al* 1999). There are three methods to prepare compound semiconductor systems: bilayer annealing (Singh and Vijay 2004a), rapid thermal annealing (Singh and Vijay 2004b) and ion beam mixing (Dhar *et al* 2003). The annealing and ion beam mixing were found to show inferior mixing effects compared to rapid thermal annealing. There has been no report on III–V elements of Al–Sb bilayer structure prepared by rapid thermal annealing and characterization of these films.

2. Experimental

2.1 *Sample preparation*

The samples of Al–Sb were prepared by thermal evaporation method using Hind High Vacuum coating unit at a pressure of 10^{-5} torr. High purity aluminum (99·999%) and antimony powder (99·5%) were used. The glass substrates were placed in the substrate holders above the boats carrying materials. The antimony having thicknesses, 1000 Å and 1500 Å, was first evaporated and later aluminum of constant thickness (3000 Å) deposited over these films to get Al–Sb bilayer structure. The thickness was measured *in situ* using a quartz crystal thickness monitor.

2.2 *Heat treatment*

A set of samples was annealed for 6 min by rapid thermal annealing process (RTA) using halogen lamp of 500 W to get homogenous mixture of Al–Sb film. A similar set of as deposited films was also annealed for 1 h in vacuum at a pressure of 10^{-5} torr up to 433 K for mixing the material.

2.3 *Absorption spectra*

The absorption spectra of as deposited, vacuum annealed and rapid thermal annealed films were recorded in the range 360–860 nm with the help of a Hitachi spectrophotometer model-330.

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2.4 *Rutherford backscattering spectra*

The RBS data have been recorded at the Institute of Physics, Bhubaneswar, using pelletron facility alpha (He^{2+}) particle beam having energy, 3 MeV at 160° backscattering angle with the help of surface barrier detector.

3. Results and discussion

3.1 *Optical absorption spectra*

Figure 1(a) shows the graph between wavelength vs absorption spectra of thin film set of Sb (1000 Å)–Al (3000 Å) as deposited, vacuum annealed and rapid thermal annealed. This graph shows that absorption increases in the case of vacuum annealed and rapid thermal annealed films when compared to as deposited films. It shows the variation in mixing of interface change with the method of preparation. Figure 1(b) shows the graph between wavelength vs absorption spectra of thin film set of Sb (1500 Å)–Al (3000 Å) as deposited, vacuum annealed and rapid thermal annealed. Here it was observed that vacuum annealed films show approximately same value of absorption but rapid thermal annealed films show drastic change in absorption curves. It shows better mixing in case of 1 : 3 ratios of two elements in films. Films of 1000–3000 Å thicknesses show band gap for rapid thermal annealed as well as vacuum annealed films very close to bulk value.

3.2 *Optical band gap*

The optical band gap of these films was calculated using the famous Tauc (1974) relation

$$\alpha h\nu = A(h\nu - E_g)^n,$$

where $h\nu$ is the photon energy, α the absorption coefficient, E_g the band gap, A a constant, $n = 0{\cdot}5$ for direct band gap material and $n = 2$ for indirect band gap material. Since Al–Sb is a indirect band gap material, so, in the present work, we have used $n = 2$. We have plotted the graph between photon energy ($h\nu$) vs $(\alpha h\nu)^{1/2}$. The intercept of straight line to energy axis is used to find the values of optical band gap of Al–Sb thin films.

The value of band gap is found to vary with treatment as well as depending upon thickness of Sb layer mix with Al layer as shown in table 1. For the samples with lower thickness of Sb with Al, the RTA treated samples give better mixing effects and band gap approaches to bulk value in good agreement with literature (Herczog *et al* 1958; Yu *et al* 1995). However, the samples with thickness ratio Sb : Al : 1 : 3 for both types of treated samples were found to be comparable with the band gap value of the bulk AlSb semiconductor, which suggests formation of single phase at the interface due to mixing of Al and Sb. The variation in band gap may be due to variation in intermixing interface with different treatments.

3.3 *Rutherford back scattering analysis*

The Rutherford back scattering is a single rapid, sensitive and non-destructive depth profiling technique and is most suitable for quantitative analysis to explain intermixing of bilayer. Figure 2 plotted between channel no. vs counts for Al–Sb as deposited, vacuum annealed and rapid thermal annealed bilayer thin films of thickness, 3000/1000 and 3000/1500 Å. It was observed that in case of vacuum annealed and RTA, the peak shifted towards higher energy side

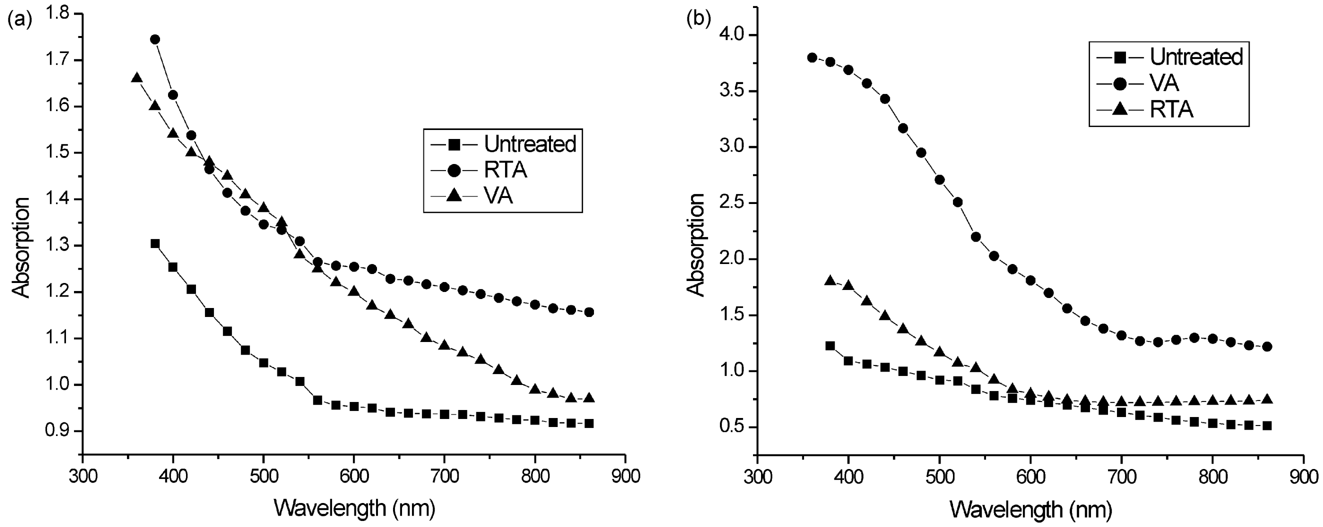

Figure 1. (**a**) Optical absorption spectra of as deposited, rapid thermal annealed and vacuum annealed Al–Sb bilayer thin films of thickness, 1000–3000 Å and (**b**) optical absorption spectra of as deposited, rapid thermal annealed and vacuum annealed Al–Sb bilayer thin films of thickness, 1500–3000 Å.

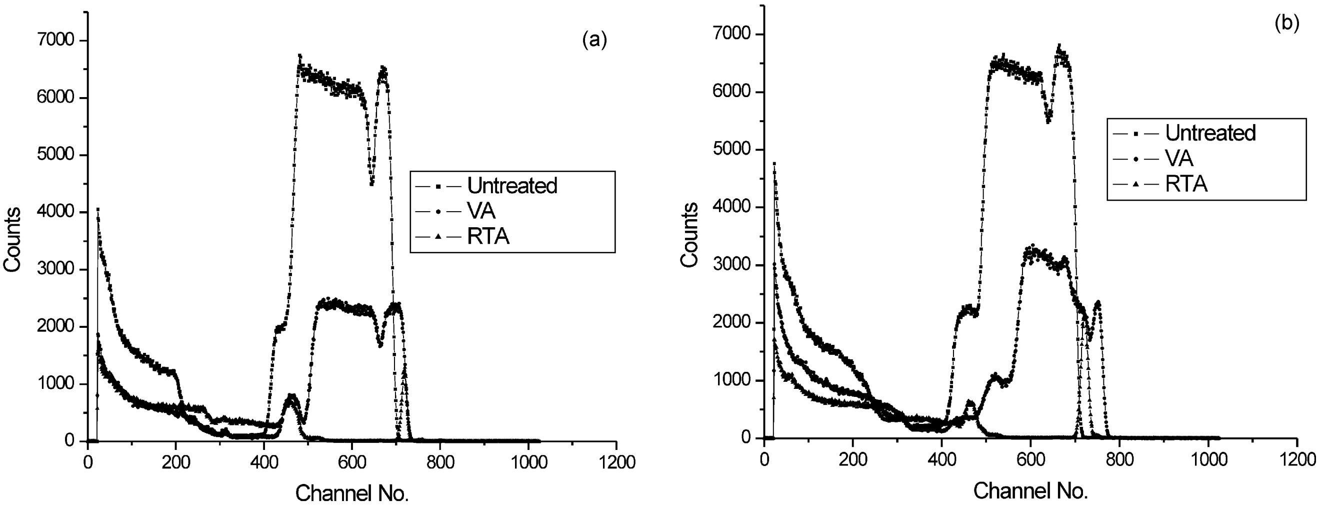

Figure 2. (**a**) RBS spectra for as deposited, vacuum annealed and rapid thermal annealed Al–Sb bilayer thin films of thickness, 1000–3000 Å and (**b**) RBS spectra for as deposited, vacuum annealed and rapid thermal annealed Al–Sb bilayer thin films of thickness, 1500–3000 Å.

Table 1. Variation of optical band gap with heat treatments.

Thickness (Å) of Al–Sb films	Band gap (eV) of vacuum annealed films	Band gap (eV) of RTA films
3000–1000	1·76	1·67
3000–1500	2·10	2·06

and also reduction in intensity of peak was observed, which shows the mixing effect in binary system. In figure 2(b), better mixing effects are observed in rapid thermal annealed film compared to vacuum annealed ones. It means interdiffusion occurs between the Al/Sb bilayers and the concentration of Al enhanced due to higher diffusion coefficient of aluminum than antimony. The downward peak to valley ratio is also observed (Holloway *et al* 1987) in the case of Ti–Si bilayer structure. Similar observations are found in our optical band gap data for 1 : 2 ratio of thickness of films. In figure 2(a), the RTA spectra having 1 : 3 thickness ratio of bilayer films gives better results compared to vacuum annealed samples. In all spectra we have observed that RTA annealed samples show more diminished peak intensity and it may be due to more mixing at interface. The peak of RTA annealed samples almost disappear and that shows more mixing than in vacuum annealed films. The observed value of our optical band gap also agrees with bulk band gap of the material and this supports the mixing. Similar results are found in case of Zn–Se RTA annealed system (Singh and Vijay 2004a) and in In–Sb annealed system (Singh and Vijay 2004b).

4. Conclusions

On the basis of these results, we may say, in thin films with lower Sb thickness, RTA and vacuum annealing give better mixing based on the result that band gap values are close to bulk value.

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