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# Growth of the Bi–Sb superlattice

G Gładyszewski, P Mikołajczak, Z Mitura and M Subotowicz
Institute of Physics, Marie Curie-Skłodowska University, pl M Curie-Skłodowskiej 1, 20-031 Lublin, Poland

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**Abstract.** Bi–Sb multilayered thin films described in the paper were produced by evaporation in a diffusion vacuum system. X-ray diffraction measurements were the main method of investigating the structure of the films. Depending on the substrate temperature during the preparation of the samples, different types of texture were observed: (110) or/and (111). The samples produced at 170 K (showing strong (110) texture) were subjected to a detailed scrutiny. Computer-simulated x-ray spectra based on a statistical model of the superlattice structure were applied in the analysis.

## 1. Introduction

In investigations of the properties of metallic superlattices information about the structural parameters of the obtained samples, such as the superlattice period, grain sizes, slope of interface, etc is extremely important. Individual structures may be examined by means of the transmission electron microscopy (TEM), and in suitable cases high resolution can be achieved [1]. The surface of samples can be analysed by scanning electron microscopy (SEM). Quite recently, reflection high-energy electron-diffraction (RHEED) has aroused growing interest, since it makes possible the study of the growth of the films monolayer by monolayer [2, 3]. Secondary-ion mass spectrometry (SIMS) and Auger electron spectroscopy (AES) are used to analyse the composition depth profile of the films [4]. Other methods such as ion channelling and Rutherford backscattering spectroscopy (RBS) [5] give us information about the chemistry of the films as a function of depth, on a 100 Å scale of resolution, and about the location of the ions in the channels of the crystal. However, the structure is predominantly examined by the use of x-ray diffraction measurements [5]. In spite of applying advanced technology [6–8], samples show spectra differing considerably from those calculated for the model of an ideal superlattice structure. In order to make a more faithful interpretation of x-ray diffraction patterns a few models of non-ideal structure have been proposed [9–15]. The Bi–Sb superlattice was studied earlier by Jałochowski [16]. Mitura and Mikołajczak recommended the use of their statistical model of the superlattice structure [13]. They carried out numerical simulations of the growth of Pb–Ag and Bi–Sb superlattices and calculated relative x-ray diffraction spectra. In this paper we have analysed in detail the growth of Bi–Sb multilayered films and compared experimental results with the numerical calculations.

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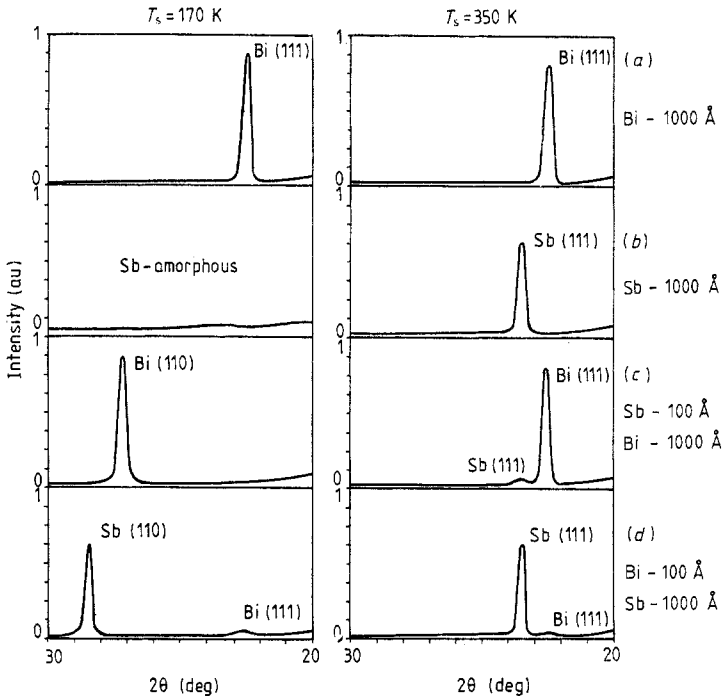


**Figure 1.** X-ray diffraction spectra of (*a*), (*b*) single and (*c*), (*d*) double layers of bismuth and antimony obtained at different substrate temperatures $T_s$ = 170 K and 350 K.

## 2. Experimental results

Thin-film layered samples were vacuum deposited in a diffusion-pump vacuum system equipped with a liquid nitrogen trap. The residual gas pressure during the evaporation was equal to $7 \times 10^{-7}$ Torr. A shutter (controlled by a microcomputer) and a water-cooled quartz oscillator were placed above the crucibles. It was possible to produce metallic superlattices in the full range of sizes and compositions of the unit cell (superlattice period). The evaporation rate was about 10 Å $s^{-1}$ for both elements. A computer-controlled system for the evaporation process has been thoroughly described before [17].

Initially, we examined growth conditions of single layers of bismuth and antimony at different substrate temperatures. In single layers of Bi, a strong (111) texture was found (see figure 1(*a*)). Single layers of Sb evaporated at 170 K turned out to be amorphous (figure 1(*b*)). Then we repeated evaporations of Sb and Bi, but this time using thin buffer layers of the other element. X-ray diffraction spectra of these samples are shown in figure 1(*c*, *d*). On the basis of these spectra it can be seen that a (110) texture of Bi and/or Sb layers has been forced by thin buffer layers. Probably for the same reason the multilayered samples obtained at 170 K also had the (110) texture. Samples obtained at higher temperatures showed both (110) and (111) texture. At 350 K the (111) type of texture was far more preponderant (see figure 2).

In conclusion to this section we would like to point out that we obtained samples with quite different types of texture by changing only the temperature of the glass (amorphous) substrate. A similar effect (different types of texture) was presented by

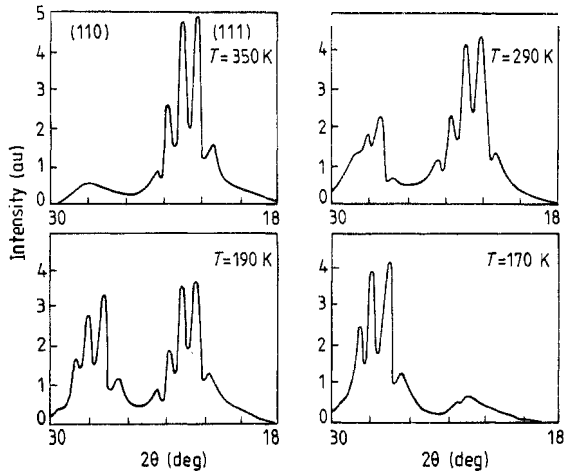


**Figure 2.** Experimental x-ray spectra of multilayered Bi–Sb films obtained at different substrate temperatures.

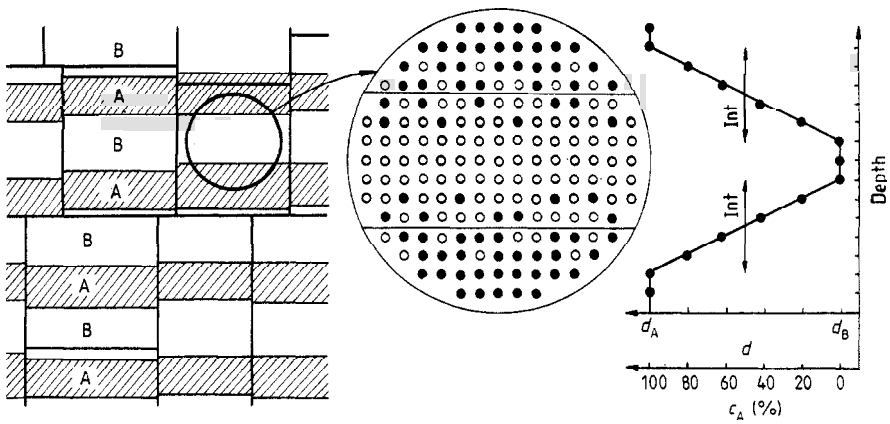


**Figure 3.** The concept of the model of a non-ideal superlattice structure; $c_A$ is the concentration of element A in an actual plane, $d$ is the actual interplanar space, $d_A$ and $d_B$ are interplanar spaces for pure elements A and B, and Int is the thickness of the interdiffused interface region.

Durbin *et al* [18], but in their case it was obtained by applying different types of substrates at the same temperature.

## 3. Analysis of the Bi–Sb superlattice structure

The analysis was carried out for samples with (110) texture because this type of texture is forced mutually by both components, which is more interesting from the crystallographic point of view. Multilayered films were examined by comparing experimental x-ray

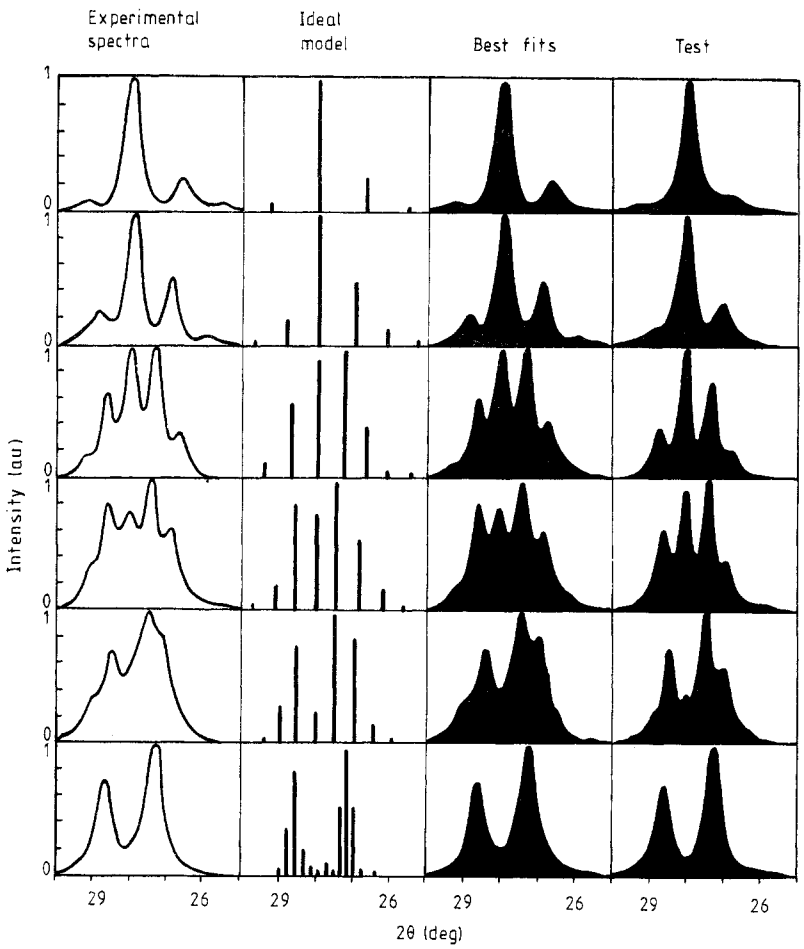


**Figure 4.** Comparison of experimental and calculated x-ray spectra of the Bi–Sb (110) superlattice. Unit cell composition of the samples determined by calculations is (from top to bottom) 10/12, 14/16, 19/23, 20/28, 27/31 and 60/80. 'Best fits' were obtained with the parameter values $\sigma_{AT} = 1.5$, $45 < \bar{G} < 50$, $I = 0$. The parameter values of 'test' were $\sigma_{AT} = 3.5$, $\bar{G} = 65$, $I = 4$.

spectra and computer x-ray spectra simulated by the Monte Carlo method. In simulations we followed the model as presented in [13]. Figure 3 shows the concept of this model. The application of diffraction theory leads to the following formula in calculating the intensity $I(\theta)$ of x-ray scattering by a sample, if the $\theta$–$2\theta$ diffraction technique is used:

$$I(\theta) = L(\theta) \sum_{k=1}^{K} \left| \sum_{j=1}^{G_k} P_{kj}(\theta) \exp\left(\frac{i4\pi x_{kj} \sin\theta}{\lambda_x}\right) \right|^2$$

where the $L(\theta)$ factor is dependent on the polarisation, Lorentz and geometric factors; the $P_{kj}(\theta)$ factor depends on scattering functions, Debye–Waller coefficients, and densities of atoms in the $j$th plane of the $k$th grain; $x_{kj}$ is the coordinate of this plane in the growth direction. $G_k$ is the number of all monolayers in the $k$th grain and $\lambda_x$ is the x-ray

wavelength. The following parameters depending on the superlattice structure appear in the model: $\bar{m}$, $\bar{n}$ are the average numbers of evaporated monolayers in one unit cell, $\sigma_{AT}$ is the standard deviation of the numbers of monolayers in one unit cell, $\bar{G}$ is the average number of all monolayers in one grain, $\sigma_G$ is the standard deviation of the grain thickness, $I$ is the number of deformed distances in one metal at the interface. A chemical (interdiffused) interface has been used in this work. Computer simulations executed for different superlattices showed that it was not possible to introduce a general algorithm for determining the aforementioned parameters for any superlattice by using an analysis of x-ray spectra. However, it was possible to work out such a method independently for every superlattice. The analysis of computer spectra for the Bi–Sb (110) superlattice proved that large changes in the spectra were caused by small changes of $\bar{m}$, $\bar{n}$ and $\bar{G}$, but changes of $\sigma_{AT}$ and $I$ influenced the spectra only slightly. Also worth noting is the fact that by decreasing $\bar{G}$ and increasing $\sigma_{AT}$, an increase in peak halfwidths was obtained. The increase in $\sigma_{AT}$ also caused large deformations of satellite peak shapes. Taking into account the above remarks, we analysed the Bi–Sb (110) structure in the following way (see figure 4).

(i) Preliminary step. Using the model of an ideal superlattice we determined $\bar{m}$, and $\bar{n}$ for each experimental sample. Simultaneously, preliminary simulations showed that $I < 3$ should be assumed.

(ii) Estimation of $\sigma_{AT}$ and $\bar{G}$. It was assumed that $I = 0$ and $\sigma_G = 1/2\bar{G}$. The simulations pointed to the estimation: $0 < \sigma_{AT} < 3$, $40 < \bar{G} < 60$.

(iii) Testing. The handling of different parameter values ($I > 3$, $\sigma_{AT} > 3$, $\bar{G} > 60$) provided spectra with features quite different from those typical of experimental samples.

## 4. Conclusion

We have analysed the growth of the Bi–Sb superlattices on glass (amorphous) substrates at different temperatures. We found that (110) texture is forced mutually by both components when the substrate is kept at 170 K. The analysis of the structure was carried out for these samples. Experimental x-ray diffraction spectra were compared with those simulated on the basis of the statistical model of superlattice structure. The best comparisons were obtained with the following parameter values: $I = 0$ (sharp interface), $\sigma_{AT} = 1.5$ monolayers, average grain sizes $45 < \bar{G} < 50$ monolayers.

The method of analysing the superlattice structures based on comparing experimental and simulated x-ray spectra seems to be very promising in spite of the difficulties caused by the need to estimate many parameters. The application of this method together with other parallel investigations (electron microscopy, RHEED, etc.) can reduce these difficulties and prove its value.

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