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E.K. Polychroniadis
Ahmet Yavuz Oral
Mehmet Ozer *Editors*

2nd International Multidisciplinary Microscopy and Microanalysis Congress

Proceedings of InterM, October 16–19, 2014

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Preface

The second International Multidisciplinary Microscopy and Microanalysis Congress & Exhibition (INTERM 2014) provided all these scientists the opportunity to meet, present their work, discuss and mutually interact in order to enhance and promote their research work.

This volume, published by Springer, includes all the papers presented at this Congress, held in Liberty Hotels Lykia Oludeniz, Turkey, October 16–19, 2014.

On behalf of the organizing committee we would like to thank all the plenary and invited speakers for their valuable contribution and especially Professor Gustaaf Van Tendeloo (EMAT, University of Antwerp, Belgium) for his excellent opening lecture.

We would also like to thank TURA Tourism for their support in the organization of the Congress, as well as our sponsors: JEOL Ltd., NanoMEGAS, and PVA TePla. Finally, we would like to thank the publishers for the quality of this edition.

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Part I
Applications of Microscopy in the Physical Sciences

Electron Microscopy Study of Thermoelectric $(Bi_xSb_{1-x})_2Te_3$ Thin Film

Aikaterini Breza, Christos B. Lioutas and John Giapintzakis

Abstract In the present work, transmission electron microscopy (TEM) techniques were used in order to study the morphology and investigate the structural properties of a $(Bi_xSb_{1-x})_2Te_3$ (BST) thin film. The sample was fabricated on silicon substrate by pulsed laser deposition (PLD) method. Towards this aim, cross sectional and planar view samples were prepared, suitable for High Resolution Electron Microscopy. Results revealed a polycrystalline, c-axis oriented film with coherent boundaries between neighboring columnar grains.

1 Introduction

In the past decades, thermoelectric films have received increasing interests due to their application on electronic devices in the micro- heating and cooling areas [1]. Bismuth telluride based thin films have gained growing interest since they have attractive thermoelectric properties and can potentially contribute to saving energy issues [2]. Generally, the performance of thermoelectric materials is characterized by the dimensionless figure-of-merit, ZT, defined as ZT = S2σT/κ, where S is the Seebeck coefficient, σ is the electrical conductivity, T is the absolute temperature and κ is the total thermal conductivity with contributions from the lattice thermal conductivity (κL) and the electronic thermal conductivity (κe). Theoretical

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approach has shown that nanostructuring Bi_2Te_3 alloys enhances their performance compared to the corresponding bulk materials, as quantum confinement effect leads in reduction in thermal conductivity [3]. In this direction, researcher groups have fabricated several types of low-dimension structures and confirmed that spatial confinement significantly improves ZT [4–9].

From a structural point of view, bismuth antimony telluride has the same rhombohedral tetradymite structure as Bi_2Te_3 of the space group R-3m. It could be described as a hexagonal unit cell consisting of five covalently bonded atomic lamellae stacked in the rhombohedral [111] direction [10]. It is worth noting that nanostructuring these materials is due this anisotropic character. In this work, we report on the morphological characteristics and structural properties of a BST thin film.

2 Experimental Details

The BST thin film that we studied in this work, was deposited on n-type Si (1 0 0) single crystal substrate by PLD method. UV-excimer laser pulses with wavelength $\lambda = 248$ nm, pulse duration $\tau_L = 25$ ns, pulse repetition rate of 10 Hz and fluence $\Phi = 2$ J cm^{-2} were employed for target ablation. For depositions 700 laser pulses were used. The substrate was heated to 350 °C. The BST target was a polycrystalline pellet with density higher than 90 % of the theoretical density. The structural features of the film were studied by TEM using a JEOL 2011 transmission electron microscope, operating at 200 kV and having a point resolution of 1.94 Å. Specimens suitable for cross-sectional and planar view TEM observations, were prepared using well-known techniques, including mechanical griding followed by ion milling with Ar ions of energy of 4 keV (Gatan PIPS).

3 Results and Discussion

The main features of the film are shown on a typical cross-sectional picture of the film presented in Fig. 1a. As it can be seen from the lower part of the film, it consists of almost columnar crystals, growing from the substrate up to the surface of the sample, of sizes from 80 to 150 nm, while its thickness is measured at 330 nm. A closer look at the interface with the Si substrate reveals the presence of an amorphous SiO_2 layer, having thickness of about 6.5 nm. Study of the corresponding electron diffraction pattern (Fig. 1b) confirms the epitaxial grown of the film, which is c-axis of all crystals parallel to [001]Si direction. Furthermore, all diffraction spots derived from the film were identified to belong to $(Bi_{0.5}Sb_{0.5})_2Te_3$ compound (72-1835#PDF).

High Resolution TEM study was performed in order to clarify the structure of the interfaces between the nano-columns. A typical micrograph of a boundary

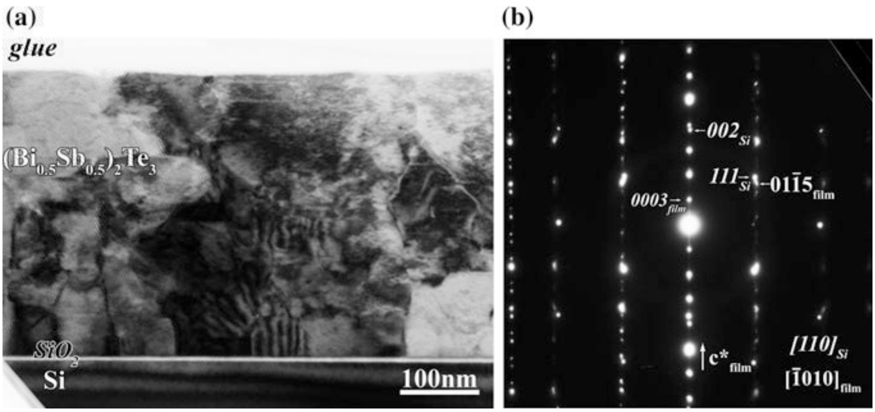

Fig. 1 **a** Bright-field image of film and substrate and **b** the corresponding electron diffraction pattern

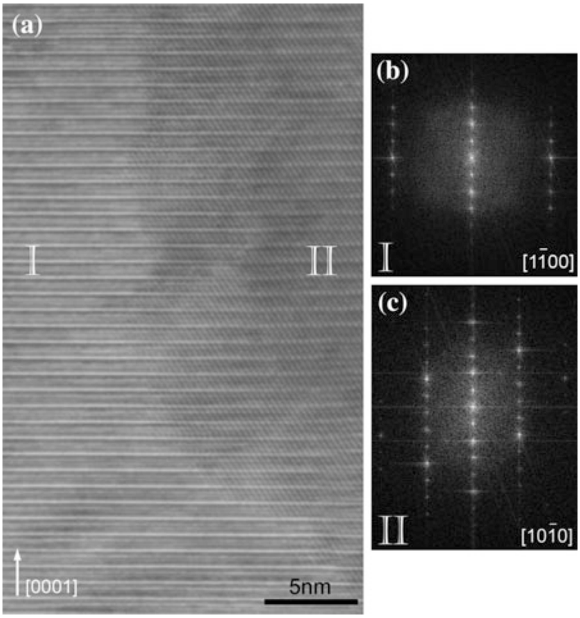

Fig. 2 **a** A typical coherent boundary is shown, while **b** and **c** FFTs correspond to crystallites I and II respectively and reveal the orientation relation between the columns

between two columns is given in Fig. 2a. It is worth noting that (0001) planes are coherent throughout the boundary. As identified by the corresponding FFTs (Fig. 2c, d), crystallites I and II are observed along [1-100] and [10-10] zones, respectively.

A planar-view image of the film is given in Fig. 3a, revealing the polygon shape of the crystals, observed along [0001] direction. In the corresponding electron diffraction pattern (Fig. 3b), diffraction rings confirm that it is polycrystalline.

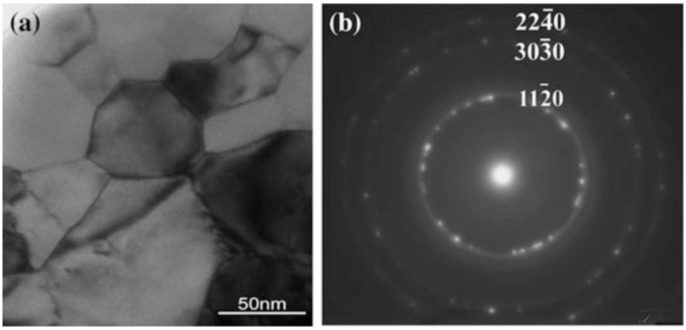

Fig. 3 **a** Bright-field image of the multigrains **b** the corresponding SAED of film

At the first view there is no preferential orientation of a-axis of the crystallites, regarding the substrate. Although, additional measurements of the angles between adjacent crystals, shows a tendency to form angles in good agreement with Coincide Site Lattice (CSL) theory.

4 Conclusion

We investigated by means of TEM the characteristics of a thermoelectric $(Bi_xSb_{1-x})_2Te_3$ thin film grown by pulsed laser deposition technique. Throughout the examined areas, the hexagonal $(Bi_{0.5}Sb_{0.5})_2Te_3$ compound (72-1835#PDF) was found. TEM micrographs and EDs showed that the 330 nm-thick film is polycrystalline and consists of columnar crystals of 80–150 nm width with orientations close to that predicted from CLS theory. HRTEM images revealed the epitaxial relationship between film and substrate, which is $[0001]_{film}//[001]_{sub}$.

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Structural Characterization of Layers for Advanced Non-volatile Memories

K. Giannakopoulos, J. Giannopoulos, P. Bousoulas, E. Verrelli and D. Tsoukalas

Abstract Non-volatile memory cells are the devices with the most aggressive scaling on the market. For this reason the accurate characterization of their layer stacks is of great importance. We present a review of our recent work on a large variety of such stacks, for charge-trap and resistive memories, which have been characterized structurally with Transmission Electron Microscopy and Conducting Atomic Force Microscopy; we discuss the features of their structure on their function as memory elements.

1 Introduction

The technology for creating advanced memory cells plays a central role in the miniaturisation of electronic devices; new process nodes usually enter production with the manufacturing of memory cells. At this moment the 16 nm memory cells are in production. The recent slowdown of the transistor scaling has partially been compensated by the improvements in memory performance and by their integration on various chips. There is a fierce competition between memory manufacturers which is governed by their ability to introduce new processes on time that are improving the speed, the reliability and the density of the memory cells, while keeping the production and development cost at a low level.

Non-volatile memories are at the centre of this technology and during the last years Solid State Disks have become mainstream computer components. Flash memories have dominated this vast field for a long time, and because their scaling

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has also been slowed down, the interest of the scientific community is shifting increasingly towards other technologies, such as the Resistive Memories. The resistive memory is currently the most promising non-volatile memory technology, as it offers low-power, reliable and denser memory cells with the option to exploit not only single level cell storage (storing one bit of information) but also multilevel cells, a feature that multiplies the effective information density storage.

From a Materials Science point of view, advanced non-volatile memories consist of nanometer scale stacks that can be processed with a large number of methods, in order to achieve the best balance of their electrical properties. Insulating, semiconducting and metallic properties are pushed to their limits while there is a constant search for new materials and device geometries. Of course, the introduction of new materials and process is not easily welcomed by the manufacturers, as it may lower the production yield and therefore increase significantly the cost of the devices. They prefer to work with "CMOS compatible" materials and processes, i.e. materials that they know better, such as Si and its compounds (Silicon Dioxide, Silicon Nitride) that can be integrated in the existing processes and that will not cause cross-contamination concerns in their ultra-clean production environments.

Whatever the approach for creating non-volatile memories, structural characterization is a critical part of the research effort that is required for their development. Because of the complicated nature of their functional properties (retention time, write/erase speed etc.), the structural and morphological data of these devices must be combined with other materials' properties and esp. the electrical, in order to understand their operation.

2 Flash Memories

Flash memories consist of at least 3 layers that are deposited on a semiconducting substrate (usually Si) covered by an electrode (or gate) where the write/erase voltage (VG) is applied (Fig. 1). In these 3 layers, the middle layer is the charge storage layer (floating gate or charge trap layer), where the charge is being stored and the other two layers are the insulating layers that block the transfer of electrons to and from the middle layer. The insulating layer next to the substrate (called "tunneling" layer) is thin enough to allow the control of the tunneling of the charges to the substrate, i.e. the charging and discharging of the charge storage layer; the tunneling layer is usually SiO_2 (thermally grown on the Si) because of the unique stability of this material, a crucial property for this position in the stack. The second insulating layer, the "blocking" or "control" oxide, has also very critical properties for the operation of the memory cell, as this is the medium through which the current may flow to the gate electrode. The device operates with the application of a voltage to the top electrode; charge is then injected from the substrate into the charge storage layer. The presence of charge near the Si substrate affects its local conductivity; if we replace the gate stack of a Field Effect Transistor (FET) with this stack, we have a transistor that can also store a bit permanently (see Fig. 1); it will