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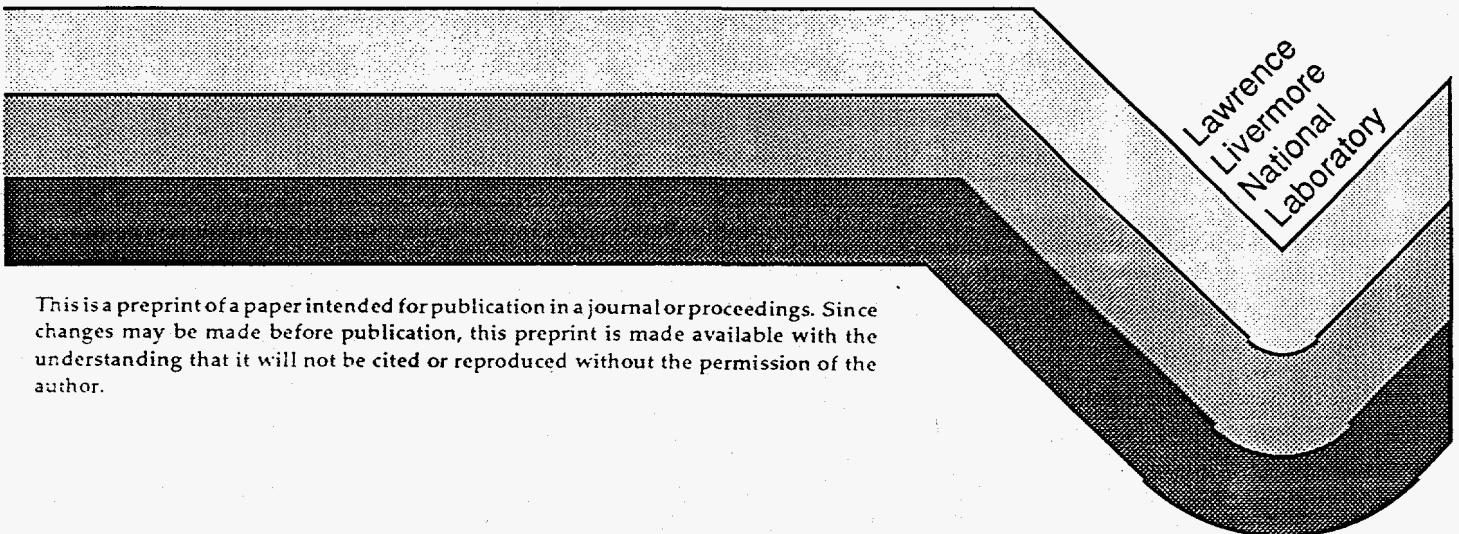
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**MULTILAYER THERMOELECTRIC FILMS:
A STRATEGY FOR THE ENHANCEMENT OF ZT**

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Multilayer Thermoelectric Films: A Strategy for the Enhancement of ZT

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ABSTRACT

The relative efficiency of a thermoelectric material is measured in terms of a dimensionless figure of merit, ZT. Although all known thermoelectric materials are believed to have $ZT \leq 1$, recent theoretical results predict that thermoelectric devices fabricated as two-dimensional quantum wells (2D QWs) could have $ZT \geq 3$. Multilayers with the dimensions of 2D QWs have been synthesized by alternately sputtering $\text{Bi}_{0.9}\text{Sb}_{0.1}$ and $\text{PbTe}_{0.8}\text{Se}_{0.2}$ onto a moving substrate from a pair of magnetron sources. These materials have been synthesized to test the thermoelectric quantum-well concept and gain insight into relevant transport mechanisms. This work focuses primarily on the scientific issues involved in producing the materials necessary to examine the possibility of enhancing ZT using quantum confinement. The techniques needed to measure the relevant electrical parameters of thermoelectric thin films are developed in this paper. Ultimately, if a quantum well enhancement of thermoelectrics is experimentally observed, devices based on this technology could be used to greatly expand the role of thermoelectrics in power generation and refrigeration.

INTRODUCTION

The relative efficiency of a thermoelectric material is characterized in terms of a dimensionless figure of merit, ZT. The best known thermoelectric materials are heavily doped, mildly degenerate semiconductors and have $ZT \leq 1$. If materials with $ZT \geq 3$ could be developed, thermoelectric devices could be made that would have thermodynamic efficiencies close to that of an ideal Carnot engine. Such materials would make it possible to replace conventional heat engines and mechanical chlorofluorocarbon (CFC) refrigeration systems with solid state devices.

Conventional thermoelectric materials ($ZT \leq 1$) are currently used in applications where it is advantageous to have no moving parts, to increase reliability, to eliminate vibration, or to allow miniaturization. They have found a niche generating power in nuclear-fueled power sources for space exploration, in silent power sources for the military, and in solid-state electrical generators reclaiming waste heat from diesel engines. Conventional thermoelectric cooling devices are used to cool infrared detectors, CCD cameras in astronomical observatories, and portable food and beverage coolers. Given the environmental problems associated with CFC refrigeration and air conditioning, the demand for efficient solid-state alternatives should be great. We are pursuing the development of 2D QWs as a means of achieving higher efficiency. Theory predicts that thermoelectric devices fabricated as 2D QWs could have $ZT \geq 3$.

In this paper we develop the techniques needed to characterize thin film thermoelectric materials and describe the results of measurements of $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ multilayer films. Single layer films of these materials have also been deposited and characterized both electrically and structurally to gain a more thorough understanding of the issues involved in depositing high quality thin film thermoelectric materials.

BACKGROUND

Figure of merit determines efficiency

The thermodynamic efficiency of a thermoelectric power generator (η) and the coefficient of performance (β or COP) of a Peltier cooler are both calculated from the dimensionless figure of merit (ZT). The coefficient of performance is defined as the ratio of cooling to electrical power. Expressions for η and β at optimum current levels are:

$$\eta = \frac{T_h - T_c}{T_h} \frac{\sqrt{1+ZT} - 1}{\sqrt{1+ZT} + T_c/T_h} \quad (1)$$

$$\beta = \frac{T_c}{T_h - T_c} \frac{\sqrt{1+ZT} - T_h/T_c}{\sqrt{1+ZT} + 1} \quad (2)$$

where T_h is the temperature of the hot side of the device and T_c is the temperature of the cold side (Goldsmid 1964, Egli 1958). To achieve high values of η or β with a thermoelectric device, a material with a large ZT value must be developed.

Conventional Strategies for Maximizing ZT.

The dimensionless figure of merit, ZT , is determined by the Seebeck coefficient (α), electrical conductivity (σ), electronic thermal conductivity (κ_{el}), and lattice thermal conductivity (κ_{ph}).

$$ZT = \frac{\sigma \alpha^2}{\kappa_{ph} + \kappa_{el}} T \quad (3)$$

Good thermoelectric materials should have a large Seebeck coefficient (α), large electrical conductivity (σ), and small thermal conductivity ($\kappa_{ph} + \kappa_{el}$). Mildly degenerate semiconductors are found to have the best combinations of these intrinsic properties (Cornish 1959). Unfortunately, attempts to improve ZT by increasing σ are eventually counteracted by detrimental changes in κ_{el} as required by the Wiedemann-Franz law. Since α , σ , and κ_{el} are all sensitive to carrier concentration, ZT is optimized by changing the extent of doping. The optimum is usually in the vicinity of 10^{19} cm^{-3} . It is also desirable for thermoelectric materials to have a low lattice thermal conductivity (κ_{ph}). Semiconductor compounds made from elements with high atomic mass tend to have lower κ_{ph} than those made from lighter elements.

Basis for Thermoelectric Multilayer Films

Although there has been little improvement in ZT during the past thirty years, Hicks and Dresselhaus of MIT have recently developed a model for the thermoelectric properties of 2D QWs, which has led to some promising predictions (Hicks and Dresselhaus 1992, 1993a, 1993b, 1993c). In bulk form, $\text{Bi}_{0.9}\text{Sb}_{0.1}$ is a narrow-band semiconductor. This results in conduction by both electrons and holes which reduces ZT . Quantum confinement of $\text{Bi}_{0.9}\text{Sb}_{0.1}$ should increase the band gap separating the electrons and holes, thereby increasing ZT . The superlattice thus behaves like a single-band 2D QW. Along with changing the material to a one band material, there is a second enhancement in ZT which results from the 2D nature of the density of states. It is this contribution which account for the increase in ZT for the 1 band materials discussed in their earlier work (Hicks and Dresselhaus 1993b).

A prediction of ZT for the $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ system as a function of layer thickness was made with the Hicks-Dresselhaus model, assuming optimum doping. In this case, quantum theory indicates that it should be possible to achieve $ZT \geq 3$ with relatively thick $\text{Bi}_{0.9}\text{Sb}_{0.1}$ layers (approximately 75 angstroms) (Harman 1992).

DEPOSITION AND CHARACTERIZATION TECHNIQUES

Sputtering Deposition

Sputter deposition has been chosen to deposit these thermoelectric multilayers for several reasons. First, sputtering gives a steady deposition rate which allows uniform thickness layers in the sample. Second, it allows multicomponent materials to be deposited from a single source without many of the compositional shifts associated with thermal evaporation techniques. Finally, the technology for large scale deposition has been developed in industry and it has been shown that the large quantities of material needed to make multilayer thermoelectrics commercially viable can be produced relatively economically.

The multilayer thermoelectric films discussed here were all synthesized in a system originally designed to fabricate multilayer X-ray optics. The system is pumped with a cryogenic pump and usually achieves base pressures of approximately 10^{-7} torr after bake-out and before sputtering. Substrates are mounted on either a heated or water cooled region of a carousel which rotates below the two magnetron sputtering guns. The 2.5-inch diameter sputter sources are usually operated at an argon pressure of 0.01 torr and a power of 15 watts to give deposition rates directly under the gun of about 4 \AA/s . The sample is alternately swung under the two magnetron sputtering sources, one depositing a conduction layer and the other depositing a barrier layer. Single layer films can be made by running only a single sputtering source and either sweeping under it to cover a large number of substrates, or sitting the substrate directly below the gun.

Structural characterization

The structure of the films have been examined both using X-ray diffraction and TEM.

A Read thin film X-ray diffraction camera with a Cu X-ray tube was used to examine the phase of the material in the films. The Read camera is similar to a Debye-Scherrer camera except that it makes use of a wider photographic film which samples more of reciprocal space. The thin film sample is held fixed with the X-ray beam impinging at grazing incidence. Bragg diffraction of the Cu $K\alpha$ radiation is used to determine the structure. The large region of reciprocal space sampled makes this technique well suited for distinguishing between randomly oriented polycrystalline films, textured films, and epitaxial films.

Seebeck measurement

The Seebeck coefficient was determined as a function of temperature for these films from near the melting point down to liquid nitrogen temperature. The measurements were made by bridging the samples between two stages which can be cooled with liquid nitrogen and then heated with a boron nitride resistive heater element. The films were deposited on an insulating substrate to insure that the electrical properties of the substrate did not effect the values measured, and also kept the film isolated from the grounded stages. Fine (0.005") K type thermocouples were secured with silver paint to each end of the sample. A copper heat shield was bolted to the top of each stage over the sample to provide a uniform thermal environment. The chamber was then evacuated with a turbo pump to eliminate condensation on the sample at low temperatures and reaction with air at high temperatures. The thermocouple leads were connected to four amplifiers one monitoring the thermocouple voltage on each end of the film and the other two bridging the n and p type thermocouple leads. These amplifiers were then read by a computer running LabView software. Since the thermal EMF generated for each lead of a K type thermocouple with reference to platinum is very well known as a function of temperature, the computer can correct for the EMF generated in the leads used to measure the voltage across the sample. (Note that the Seebeck coefficient measurements reported in this paper will be referenced to platinum as is done in the ASTM standard. The absolute Seebeck coefficient relative to platinum is also shown on the graph so that the absolute value of the Seebeck coefficient can be easily read as the difference between the two curves.) From the four amplifier's input there are several ways to calculate the Seebeck coefficient, each involving three of the four inputs. This redundancy is used to assess the magnitude of small amplifier errors, as well as reduce these errors through averaging.

Besides computing the Seebeck coefficient and displaying and logging the result, the computer also controls the power to the heaters in each of the stages. This allows the computer to ramp the sample temperature up from liquid nitrogen temperatures while maintaining a fixed temperature difference between the two ends of the sample. The Seebeck coefficient is thus measured as a function of temperature.

Resistivity measurement

The resistivity of the samples has been measured using a four point probe technique. An alternating current source (frequency typically between 10 and 200 Hz) has been set up so as not to induce a thermal gradient in the samples from the thermoelectric effect. The current through the outer pair of contacts is monitored by passing it through a reference resistor and measuring the voltage across the resistor with a lock-in amplifier. The voltage across the inner electrodes is similarly monitored with a lock-in amplifier. Using alternating current and the lockin amplifiers also helps reduce electrical interference. The thin film samples to be measured are deposited on insulating substrates which have four parallel

nickel strips running from edge to edge across the sample. These nickel strips serve as electrical contacts. The spacing between the inner pair of strips has been made the same as the width of the deposited film. Thus, the resistivity of the film is the resistance of the film multiplied by the thickness. The film resistance is the resistance of the reference resistor multiplied by the ratio of the voltage measured across the inner electrodes to that across the reference resistor. The same vacuum system used for the Seebeck measurements is used here, but the sample is mounted on a single stage. Again for ease of operation the temperature is ramped up from liquid nitrogen temperature by the computer as it collects, reduces, and displays the data. The thickness of the samples can be accurately determined by RBS so that the resistivity can be calculated from the measured resistance.

RESULTS

The experiments discussed here were conducted to experimentally examine the theoretically predicted quantum well enhancement of the thermoelectric properties of $\text{Bi}_{0.9}\text{Sb}_{0.1}$. It was also necessary to deposit single layer films of both the $\text{Bi}_{0.9}\text{Sb}_{0.1}$ conducting layer and the $\text{PbTe}_{0.8}\text{Se}_{0.2}$ barrier layer in order to understand the effects of making thermoelectric materials in thin film form, and separate this from the effect of the multilayer structure. It has already been observed in the literature (Volklein 1986, 1987) that thin films do not have the same thermoelectric properties as bulk materials. Results from single layer films of pure Bi will also be discussed, to better understand the $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films.

Structure

Read thin film X-ray diffraction measurements confirmed that all the deposited films, both single layer and multilayer, are polycrystalline, but oriented. The Bi diffraction pattern agrees with the rhombohedral structure of bulk Bi. It is interesting to note that Bi is quite close to a simple cubic structure with a distortion along the body diagonal (Ashcroft and Mermin 1976). Sb has the same structure and is just 5% smaller allowing the two elements to form a solid solution for all compositions. Bi and $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films are indistinguishable using the Read thin film camera except for a small shift in lattice spacing apparent at high angle diffraction. All the samples of Bi and $\text{Bi}_{0.9}\text{Sb}_{0.1}$ deposited on silicon ((100) with the native oxide) sapphire (1102) and glass are textured polycrystalline films with the c axis in the growth direction. This is an interesting observation since these are not the densest packed planes in the structure. It is possible that the material favors this growth direction because deposition stress in the plane of the film forces the elongated direction of the pseudo cubic unit cell out of the plane.

PbTe has a NaCl structure with a unit cell size very close to twice that of the pseudo cubic structure mentioned above for $\text{Bi}_{0.9}\text{Sb}_{0.1}$. This relationship can also be seen from the match of the (111) direction dimension for PbTe with the dimension of the bottom of the unit cell of Bi or $\text{Bi}_{0.9}\text{Sb}_{0.1}$ in the hexagonal

representation. Since Se can substitute for Te, the Read thin film camera shows $\text{PbTe}_{0.8}\text{Se}_{0.2}$ films have the PbTe structure. These films deposited on sapphire also show the same texturing as found for $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films with the (111) direction preferentially aligned with the growth direction.

For $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ multilayers the growth direction is found to be parallel to the (100) direction of the $\text{PbTe}_{0.8}\text{Se}_{0.2}$ layer. The presence of the multilayer structure forces both the $\text{PbTe}_{0.8}\text{Se}_{0.2}$ and $\text{Bi}_{0.9}\text{Sb}_{0.1}$ layers to deposit in a direction different than they would in a single layered film.

TEM images of the multilayer samples confirm that the samples are chemically modulated. The growth structure of the film is columnar with grains approximately 1000Å in diameter. There appears to be good crystalline continuity between the layers, but some waviness associated with the columnar structure.

Single layer films of Bi and $\text{Bi}_{0.9}\text{Sb}_{0.1}$

Measurements were first made on single layer films to establish a baseline for comparison with multilayer films. The resistivity of a single layer Bi film is shown in figure 1. We have found that annealing the samples by ramping the samples to successively higher temperatures during measurement reduces the resistivity. At the highest annealing temperatures near the melting point, the high temperature resistivity continues to fall but the low temperature resistivity begins to rise. Even after annealing the film to just below the melting point, the resistivity at room temperature remains approximately five times higher than bulk material. For single layer $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films the resistivity measured was found to behave like the pure Bi films being approximately five times higher than the literature values for bulk material at room temperature (figure 2). For both films the resistance decreases with increasing temperature which is opposite the behavior of the bulk material. This has previously been observed by Volklein (1986, 1987) in thin $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films and attributed to grain boundary and surface scattering of the charge carriers.

The Seebeck coefficient for Bi and $\text{Bi}_{0.9}\text{Sb}_{0.1}$ are shown in figures 3 and 4. For the Bi film the Seebeck coefficient of the as deposited film is near that of the bulk material. Following annealing, the high temperature Seebeck coefficient drops in magnitude while at low temperature the Seebeck coefficient increases. For the $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films there is an increase in the magnitude of the Seebeck coefficient at all temperatures with annealing.

The reduction in resistivity and increase of the Seebeck coefficient in these films upon annealing can be attributed to reduction in the scattering from grain boundaries caused by grain growth. Samples grown at elevated temperatures have the same electrical properties as the films which have been annealed.

Single layer $\text{PbTe}_{0.8}\text{Se}_{0.2}$ films

$\text{PbTe}_{0.8}\text{Se}_{0.2}$ has been chosen as a barrier layer for the multilayer films since its band gap and resistance are higher

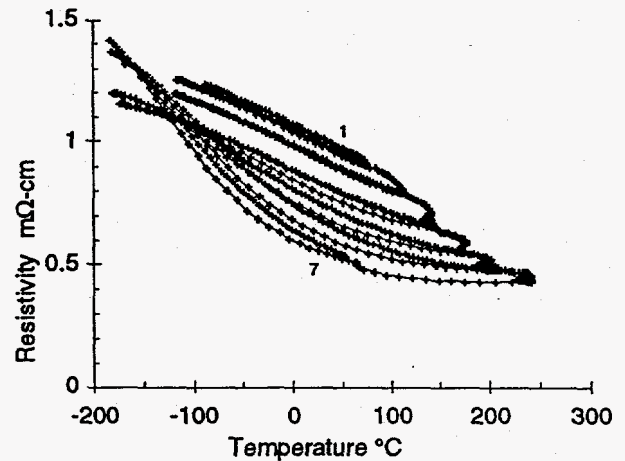


FIGURE 1: TEMPERATURE CYCLING RESISTIVITY MEASUREMENT ON A 5400Å THICK Bi FILM. CYCLE 1 AND 7 ARE LABELED.

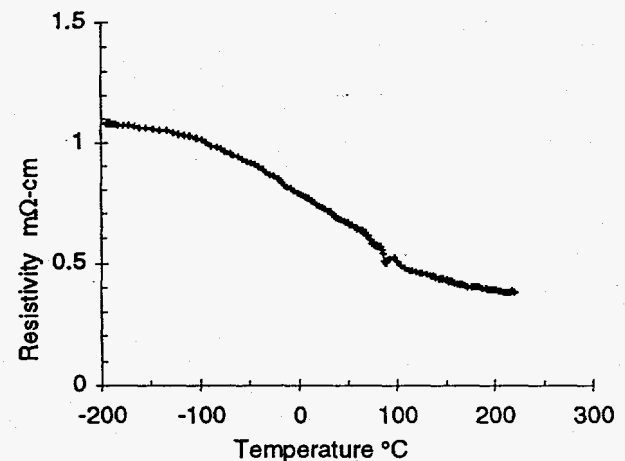


FIGURE 2: RESISTIVITY OF A WELL ANNEALED 670Å THICK $\text{Bi}_{0.9}\text{Sb}_{0.1}$ FILM.

than $\text{Bi}_{0.9}\text{Sb}_{0.1}$, and it has a similar structure with similar lattice dimensions so that the layers should grow epitaxially across the layer interfaces. Single layer films of this material show a high resistance, which upon annealing to 250°C, drops to only 100 mΩ-cm. The deposited films also show a large n type Seebeck coefficient which reaches $-600\mu\text{V}/^\circ\text{C}$ after annealing to 250°C.

Multilayer films

The multilayer films have a $\text{Bi}_{0.9}\text{Sb}_{0.1}$ thickness of 143Å and a $\text{PbTe}_{0.8}\text{Se}_{0.2}$ thickness of 115Å. Multilayer films have just slightly higher resistivity than the single conducting layer samples. This means that if one assumes that all the observed conduction is in the conduction layers and not in the barrier

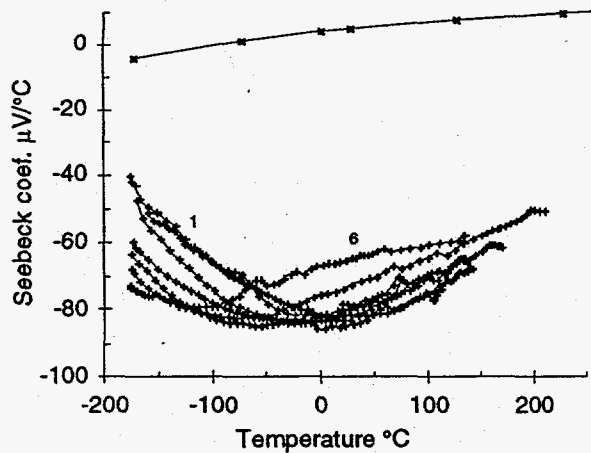


FIGURE 3: SEEBECK COEFFICIENT MEASUREMENT FOR THE Bi FILM SHOWN IN FIGURE 1 WITH REFERENCE TO Pt. (+): Bi DATA (X): ABSOLUTE DEFINITION OF SEEBECK RELATIVE TO Pt. THE FIRST AND LAST TEMPERATURE CYCLE ARE LABELED.

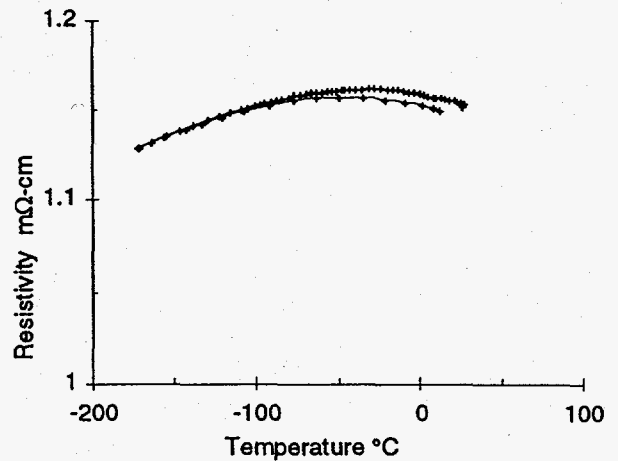


FIGURE 5: RESISTIVITY OF A 17000 Å THICK $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ MULTILAYER FILM.

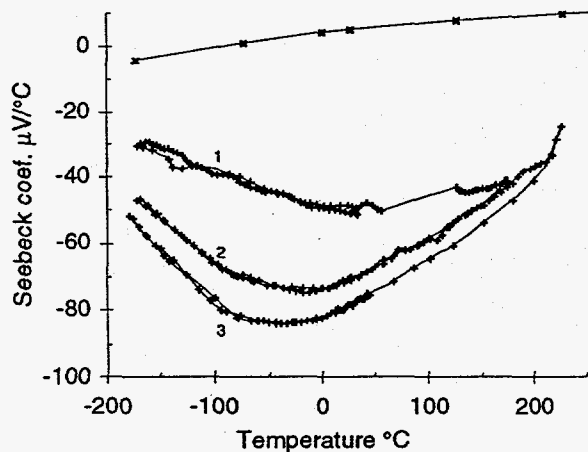


FIGURE 4: SEEBECK COEFFICIENT MEASUREMENT FOR THE $\text{Bi}_{0.9}\text{Sb}_{0.1}$ FILM SHOWN IN FIGURE 2 WITH REFERENCE TO Pt. (+): $\text{Bi}_{0.9}\text{Sb}_{0.1}$ DATA (X): ABSOLUTE DEFINITION OF SEEBECK RELATIVE TO Pt. TEMPERATURE CYCLES ARE NUMBERED SEQUENTIALLY.

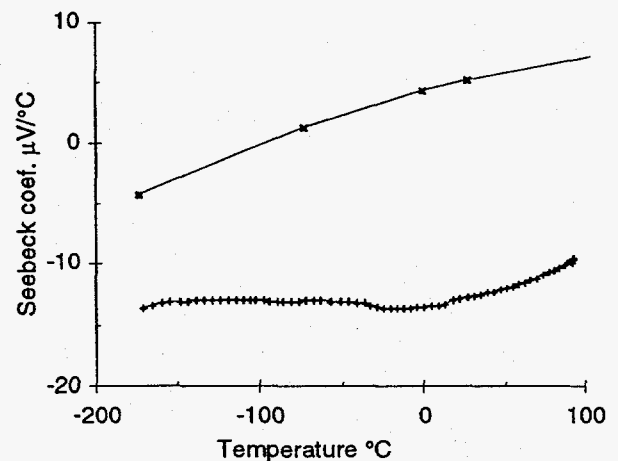


FIGURE 6: SEEBECK COEFFICIENT FOR THE $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ MULTILAYER FILM SHOWN IN FIGURE 5 WITH REFERENCE TO Pt. (+): $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.8}\text{Se}_{0.2}$ DATA (X): ABSOLUTE DEFINITION OF SEEBECK RELATIVE TO Pt.

layers, the resistivity of the conducting layers is slightly lower than in the bulk films. The Seebeck coefficient in these multilayer films however is reduced by a factor of 5 from that found in the bulk $\text{Bi}_{0.9}\text{Sb}_{0.1}$ films. Depositing the sample onto a heated substrate produces better quality multilayers as imaged with TEM, however the resistivity is not decreased further and the Seebeck coefficient is considerably lower still.

The large reduction in the Seebeck coefficient in these multilayer films results from material in the barrier layer doping the $\text{Bi}_{0.9}\text{Sb}_{0.1}$ conduction layer.

DISCUSSION

In this ongoing effort to measure possible enhancement of ZT from 2D quantum confinement in multilayer structures reliable electrical characterization is important. Techniques for

accurately measuring the thermoelectric properties of thin films as a function of temperature from the melting point of the films down to liquid nitrogen temperatures have been developed.

No evidence of an increase in the figure of merit from quantum confinement has been observed so far. It is likely that the poor performance of the multilayer materials made to date is caused by diffusion of the barrier layer material into the conduction layer. This interdiffusion can add p type carriers to the n type conduction layers which would drastically reduce the Seebeck coefficient as is observed. This doping caused by interdiffusion may make it necessary to choose materials for the conduction and barrier layers which do not dope each other.

The results presented here expose an additional materials issue which needs to be addressed in the ongoing work to produce thermoelectric multilayer quantum wells. The high resistivity observed in single layer thin films both here and in the literature presents a problem for applications of thin film technology. In order to reduce the resistivity of the films which is caused by grain boundary scattering the number of grain boundaries must be reduced. This can be achieved by growing the films epitaxially on single crystalline substrates. Work is currently underway to deposit films epitaxially on BaF_2 . Since high temperature deposition will have the adverse effect of large interdiffusion an effort will have to be made to produce a clean substrate interface to allow epitaxial growth.

If it is possible to grow such epitaxial films on the proper substrate at below room temperatures it may be possible to limit interdiffusion during deposition. This may allow us to continue to use the $\text{Bi}_{0.9}\text{Sb}_{0.1}/\text{PbTe}_{0.9}\text{Se}_{0.2}$ system to conclusively determine if it is possible to enhance the performance of thermoelectric materials using quantum well confinement.

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