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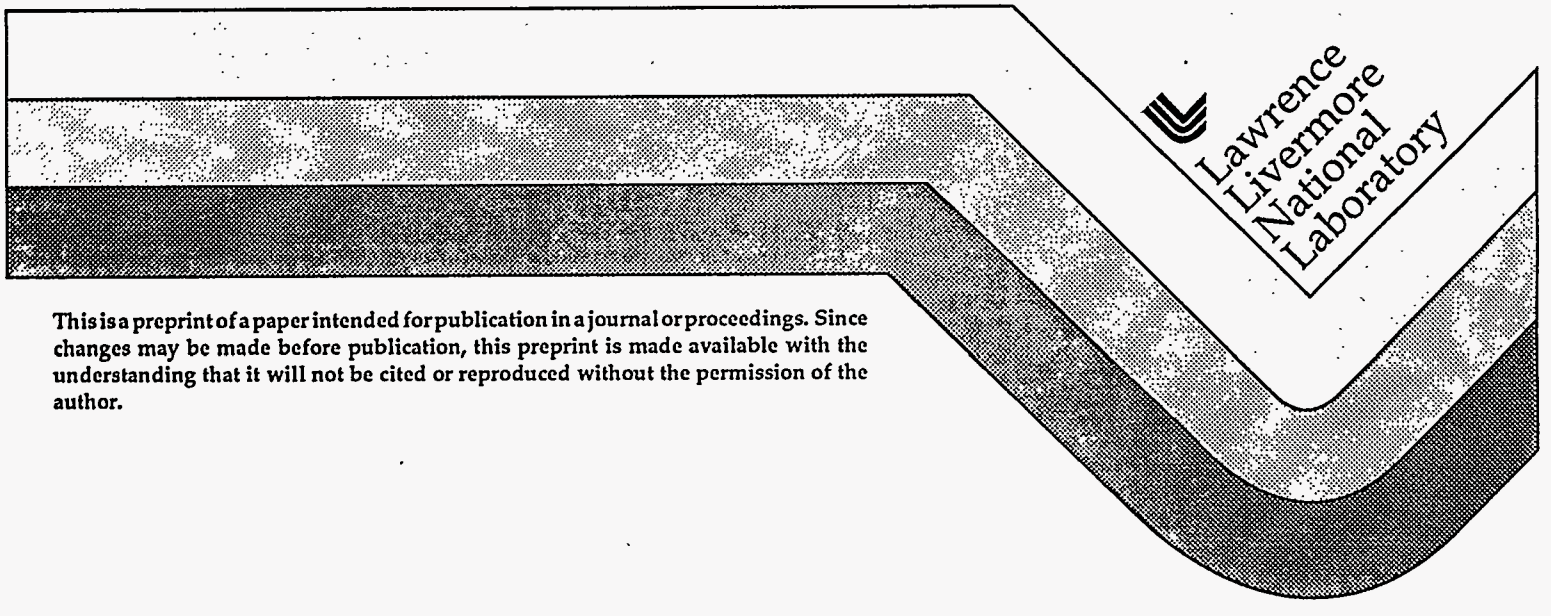

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MULTILAYER THIN FILM THERMOELECTRICS PRODUCED BY SPUTTERING.

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Introduction

Thermoelectric materials are currently used in applications where it is advantageous to have no moving parts which provides an increase in reliability, the elimination of vibration, and ease of miniaturization. While thermoelectrics are used in countless temperature sensing applications, their use for electrical power generation or cooling is limited by their relatively low efficiency. However, they have found a niche generating power in nuclear-fueled power sources for space exploration and in silent power sources for the military. Thermoelectric cooling devices are also currently being used to cool infrared detectors, CCD cameras, and portable food and beverage coolers.

Over the last 30 years increases in the efficiency of thermoelectric materials for power generation or cooling have been small. However the development of the ability to manipulate materials on a nanometer length scale, thus tailoring their electrical properties, has renewed interest in the field. Theoretical predictions by Hicks and Dresselhaus suggest that using 2D quantum confinement of the charge carriers in multilayer thermoelectric materials may lead to an increase in the figure of merit [1-2]. In order to pursue this possible enhancement in the electronic properties of multilayer materials, it is imperative to grow high quality thin film thermoelectric materials. Although thin films of thermoelectric materials have been deposited by various means for over 30 years, a new emphasis must be placed on the film quality to build nanometer length scale multilayers. The growth surface, for example, must be flat at the length scale of multilayer. Also, to reduce interdiffusion in these layered structures, it is important to develop a technique for depositing them at the lowest possible temperatures at which quality growth can be maintained. The high quality films which are essential for building quantum confined layer structures are also of interest for building miniature devices. The ability to build miniature cooling devices which can produce useful temperature differences will depend on the ability to make high quality thermoelectric films.

A number of issues have arisen while trying to grow multilayer thermoelectrics [3,4]. Both the conductivity and the Seebeck coefficient for multilayer films were found to be lower than published values for bulk material of the conduction layer. Thick single layer films were also deposited for comparison with the multilayer films. For the case of Bi and $Bi_{0.86}Sb_{0.14}$ these showed a resistivity consistent with that being observed in the multilayer films, which is about 5 times that of bulk Bi or $Bi_{0.86}Sb_{0.14}$. This is in agreement with results from the literature which suggest that grain boundary and impurity scattering may be the cause of the high resistivity. Volklein et al. have modeled grain boundary scattering and shown it to be consistent with experimental observations [5]. To achieve a high value for the coefficient of performance from thin film materials, it is important that this grain boundary scattering problem be overcome.

In this work we explore the possibility of achieving bulk electrical properties in single layer sputter deposited films grown epitaxially on (111) oriented BaF_2 substrates. There are a number of sputter deposition parameters that can be varied in order to optimize the film quality. It is important to understand the effect of varying the deposition temperature, Ar sputtering gas pressure, and the substrate bias. We will consider only Bi and $Bi_{0.86}Sb_{0.14}$ films in this paper. These materials were chosen since they have the same simple structure, two different band gaps and do not change significantly either in physical or electrical properties with small amounts of cross contamination.

We will also present our work on multilayer thermoelectrics made of Bi and $Bi_{0.86}Sb_{0.14}$ layers. There has been considerable interest in this multilayer structure in the literature. Theoretical calculations of the band structure and interface states of these multilayer structures have been made by Mustafaev and Agassi et al. respectively [6,7]. Experimentally Yoshida et al. have examined similar multilayer structures grown by MBE as well as Bi/Sb multilayer samples in which they report an anomalous thermoelectric power [8].

Experimental procedures

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 for the multilayer samples. Second, it allows multicomponent materials to be deposited from a single source without many of the compositional shifts associated with

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thermal evaporation techniques. Third, by biasing the substrates the argon ions from the sputtering gas can be used to bombard the growing film at low energies. This ion bombardment can change the growth morphology by imparting extra energy to the growth surface. Finally, the technology for large scale deposition has already been developed, and it has been shown that the large quantities of material needed to make multilayer thermoelectrics commercially viable can be produced relatively economically.

The 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 $1x10^{-7}$ torr prior to 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 around 0.01 torr and a power of 15 watts to give deposition rates directly under the gun of about 5 Å/s. To deposit multilayer samples the substrates are 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 the sputtering gun, or sitting the substrate directly below the gun.

Physical characterization

SEM has been used to characterize the morphology of the samples, as well as to measure the thickness of single layer films. X-ray diffraction has been used to determine the crystallographic orientation of the films. A Cu rotating anode Rigaku diffractometer equipped with a single crystal monochrometer has been used for observing the degree of texturing (by observing relative peak intensities as well as the intensity of the diffraction peaks as the sample is rocked in theta). Multilayer side bands on the diffraction peaks give an accurate measure of the bilayer thickness d, which can be determined from the equation:

$$d = \frac{\lambda}{2(\sin\theta_b \pm \sin\theta_{ml})} \quad (1)$$

as well as demonstrating that the structure has not interdiffused during deposition. A Read thin film camera was also used to observe the degree of texturing or epitaxy. It has the advantage of capturing a large portion of reciprocal space on film in a single exposure.

Electrical characterization

The Seebeck coefficient was determined as a function of temperature for these films at temperatures ranging from near the melting point of Bi down to liquid nitrogen temperature [4]. The measurements were made in vacuum by bridging the samples between two stages that 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 do not effect the values measured and to keep the film isolated from the grounded stages. Fine (0.003") K type thermocouples were secured with silver paint to each end of the sample and used for both temperature and voltage measurement. The thermal EMF in each lead is well known with respect to platinum so the graphs shown are plotted as the Seebeck coefficient relative to platinum. The absolute Seebeck coefficient of 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.

The resistivity of the samples has been measured in vacuum over the temperature range from liquid nitrogen temperatures to near the melting temperature of the films. A four point probe technique was used with an alternating current and a pair of lock-in amplifiers [4].

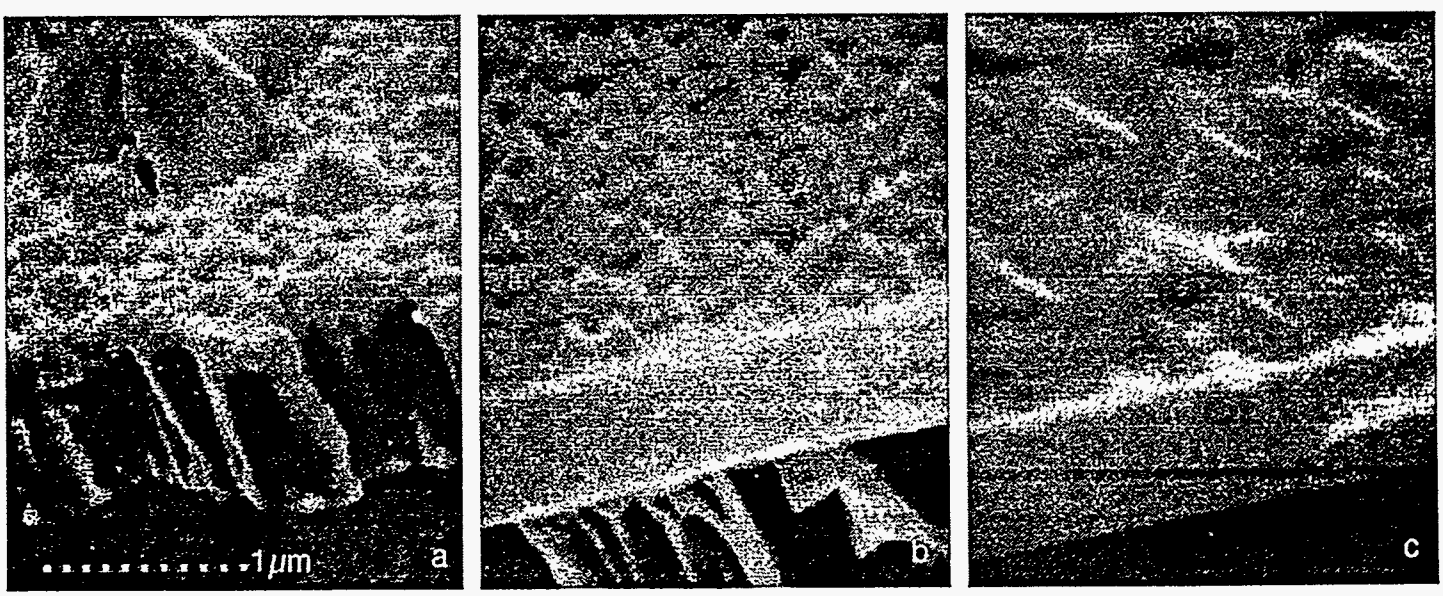

Figure 1: SEM micrographs of a series of Bi films deposited at 8mtorr Ar -10v bias. a) Deposited at 100°C on polished BaF_2. Columnar growth is evident. b) Epitaxial film deposited at 100°C on cleaved BaF_2. c) Epitaxial film deposited at 200°c on cleaved BaF_2.

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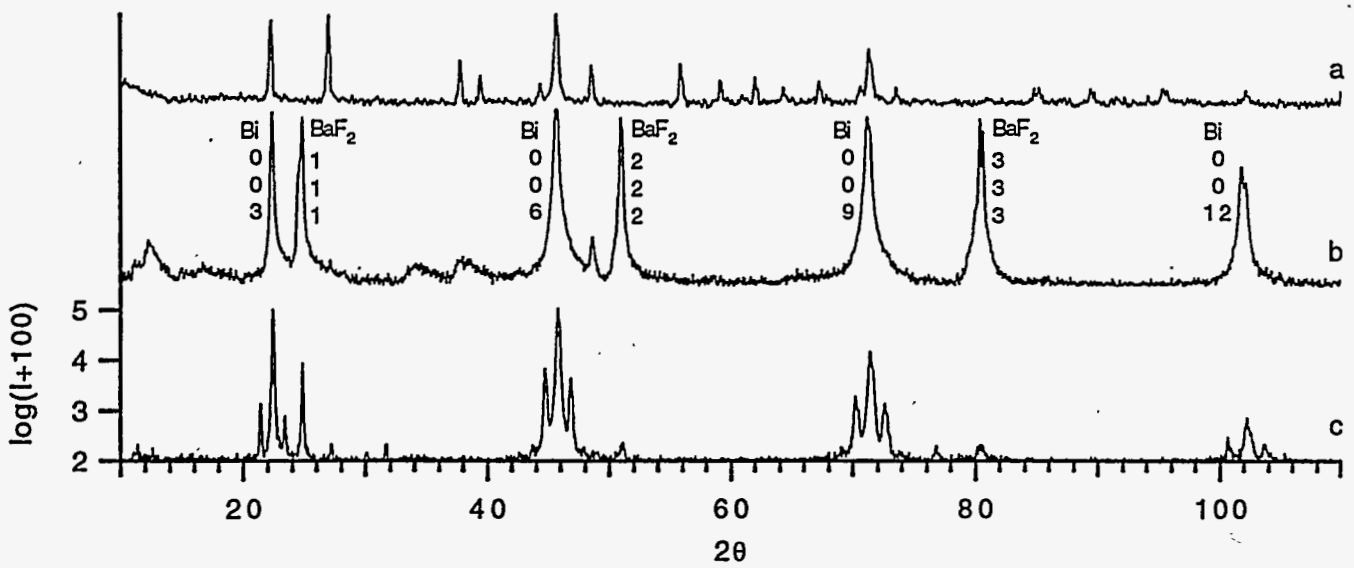

Figure 2: X-ray diffraction scans of a) Bi sample on glass showing texture, b) epitaxial Bi film on cleaved BaF_2, and c) $Bi/Bi_{0.86}Sb_{0.14}$ multilayer film showing satellite peaks.

Results and Discussion

Single layer Bi films

A series of experiments have been performed in order to optimize the quality of single layer Bi and $Bi_{0.86}Sb_{0.14}$ films. The effect of different substrates, deposition temperatures, Ar sputtering gas pressure, and sample bias were investigated and are presented below.

Samples deposited on glass, silicon (with a native oxide) and (1102) sapphire and were all found to have a rough surface (data not shown). This is caused by Bi crystallites nucleating in random orientation on the substrate surface. As the film grows those grains with favorable conditions appear to grow over their neighbors resulting in a non-epitaxial (003) textured film with the average crystallite size increasing with film thickness.

In order to grow films without the rough columnar structure a substrate was chosen on which a Bi film could grow epitaxially. The (111) face of BaF_2 (cubic) is such a substrate having an atomic spacing only 3.6% smaller than the 003 planes of Bi (rhombohedral). $Bi_{1-x}Sb_x$ also has the Bi structure where the addition of Sb reduces the lattice dimensions decreasing the mismatch with BaF_2. Depositing on a polished BaF_2 surface (figure 1a) gave results similar to that found for glass, silicon, and sapphire. Presumably this is a result of surface damage caused by polishing. On freshly cleaved BaF_2, however, epitaxial growth has been achieved (figure 1b, 1c). A difference in the quality of the films can be observed visually. Those on freshly cleaved BaF_2 are shiny while on the other substrates the rough film surface appears dull.

While the non-epitaxial films show numerous faceted crystallites, the epitaxial films show only minimal surface topography in the SEM. The film surface also shows a well aligned triangular surface structure. The length scale of this structure does not change appreciably when the films are grown to a thickness of 3μm. For the non-epitaxial films, the fracture surface is rough with the films breaking along grain boundaries. For the epitaxial films, however, the fracture surface is smooth with no evidence of grain boundaries.

A series of x-ray measurements have been made to examine the crystallographic orientation of the films. For the non-epitaxial films, growth is textured with the (003) axis perpendicular to the substrate. This can be seen from the Read thin film x-ray camera pictures which show the polycrystalline Cu Kα diffraction rings broken into arcs (data not shown). The θ-2θ scan shown in figure 2a also shows evidence of texturing with a much higher relative intensity for the (003) family of peaks than would be expected for a randomly oriented film. θ-2θ scans on films deposited on cleaved BaF_2 show that the degree of orientation is very good. The 003, 006, 009, 0012 peaks from the film dominate the spectra (figure 2b). The BaF_2 111, 222, and 333 peaks can also be seen. The position of

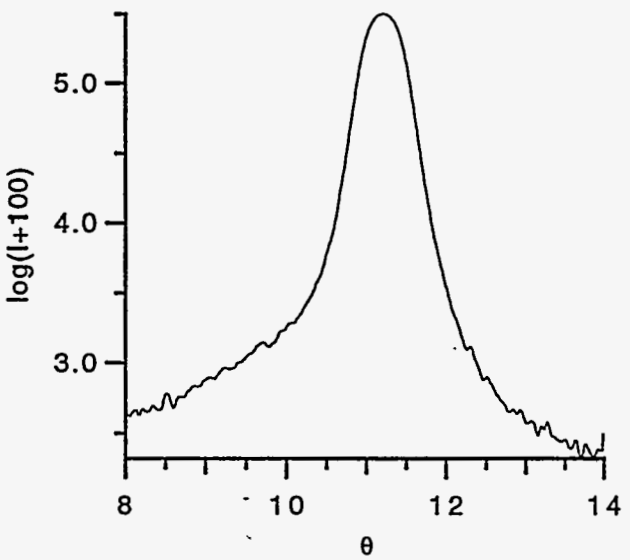

Figure 3 X-ray diffraction scan taken with the detector fixed on the 003 peak at 22.4° of the sample shown in figure 2b and the sample rocked in theta.

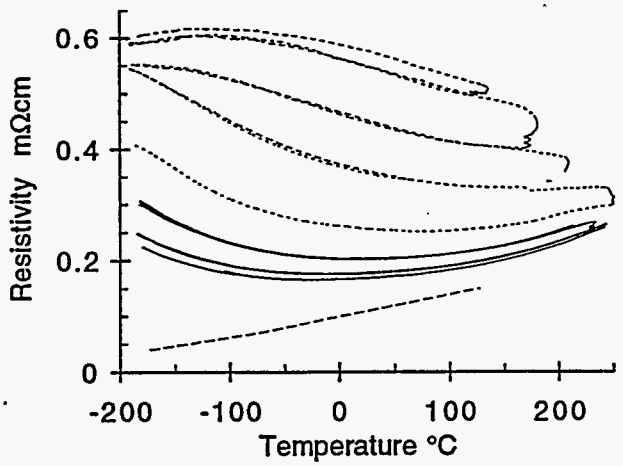

Figure 4: Resistivity of Bi. Top curve (small dash) shows a sample deposited at 100°C, -25v bias, and 16 mtorr Ar. The sample was temperature cycled from liquid nitrogen temperature to progressively higher temperatures. Annealing that occurs during each temperature cycle lowers the resistivity. Center curve (solid line) shows a sample deposited at 200°C, -10v bias, and 8mtorr Ar. Bottom curve (long dashes) is literature value for bulk Bi.

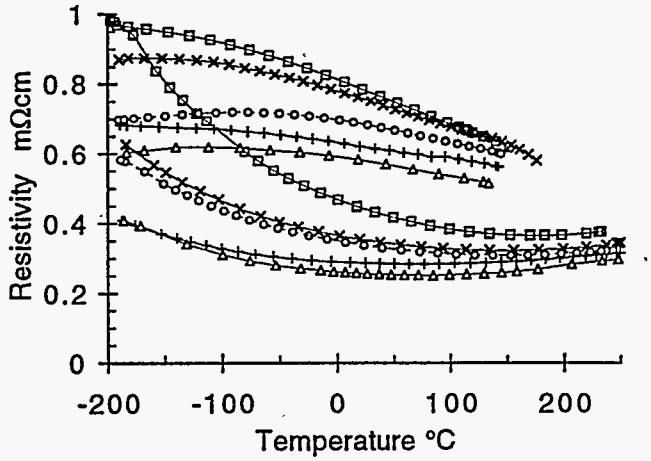

Figure 5: Resistivity of Bi films deposited at 100°C under different conditions. Both the as deposited resistivity is shown (convex) as well as resistivity after a 250°C anneal. In all cases the resistance has decreased with annealing. A film deposited at 8mtorr Ar and 10v bias on polished BaF_2 (□), or on cleaved BaF_2 (X). A film deposited on cleaved BaF_2 at -25v bias, and 8mtorr Ar (O), or on cleaved BaF_2 at -10v bias and 16mtorr Ar (+), or on cleaved BaF_2 at -25v bias and 16mtorr Ar (Δ). Small uncertainties in sample dimensions made it necessary to adjust the vertical scale for individual samples based on the annealed high temperature behavior to make trends more clear.

these peaks can be calibrated with the BaF_2 111 lines from the substrate. The angles for the film peaks are lower in angle than one would expect showing that the unit cell is elongated out of the plane of the film as a result of compressive stress in the plane of the film. With the detector positioned on the (003) peak, the sample can be rocked in theta to show the degree of orientation of the crystallites (figure 3). This shows that the orientation of the crystallites with the substrate is good to within about 1/2°.

Increasing the deposition temperature gives the depositing atoms increased mobility on the growth surface and allows the growth of higher quality films. Increased temperature may also help reduce the incorporation of foreign atoms, such as Ar in the growing film, since they are more likely to be desorbed at higher temperature. Depositing Bi films at 200°C gives a larger surface structure as show in figure 1c. The resistivity of this film is also quite low and above room temperature there is metallic behavior (increasing resistance with increasing temperature) as is found in the bulk. Further annealing to nearly the melting point reduces the resistivity slightly further. This behavior can be compared with the resistivity of films deposited at 100°C. In these films the reisitivity is considerably higher. With annealing (in the measurement chamber) the resistivity of these films also drops but the films never become as metallic as the films deposited at higher temperature (figure 4).

For multilayer growth it is important to be able to deposit films at the lowest possible temperature in order to avoid interdiffusion. Surface mobility can be increased during sputtering by providing energy by low energy ion bombardment. This can be achieved by negatively biasing the sample holder and substrates.

Increasing the Ar pressure or increasing the bias of the substrate holder both have an effect on the sample resistivity as can be seen in figure 5. In both cases the resistance is reduced in the as deposited film. There is also a decrease in the slope of the resistance curve. Both high bias and Ar pressure lead to the deposition of the lowest resistance films at 100°C.

In summary it has been shown that single layer epitaxial films can be grown on freshly cleaved BaF_2 substrates. Films grown at higher temperatures have a lower resistivity than those deposited at lower temperature. Deposition with a substrate bias and a higher Ar pressure improved the conductivity of the films deposited at low temperature. However in all cases the resistivity is still higher than that of bulk material.

<u>$Bi/Bi_{0.86}Sb_{0.14}$ multilayer sample</u>

Multilayer samples composed of Bi and $Bi_{0.86}Sb_{0.14}$ layers have been deposited on cleaved BaF_2 both at room temperature and 100°C. The existence of a multilayer structure has been verified by x-ray diffraction (figure 2c). The satellite peaks clearly show that a layered structure has been achieved with a layer pair repeat distance of 91Å.

The Seebeck coefficients of the Bi and $Bi/Bi_{0.86}Sb_{0.14}$ multilayer films are shown in figure 6. The Seebeck coefficient for both films is found to be approximately the same.

Our work agrees the work of Yoshida et. al. in which they found that samples grown on BaF_2 had no evidence of an anomalously high Seebeck coefficient. This is in contrast to their reports of an anomalous thermoelectric power in Bi/Sb multilayer samples deposited on Si.

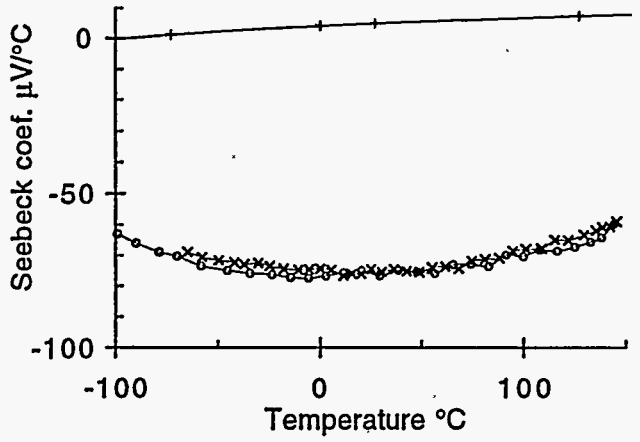

Figure 6: Seebeck coefficient of a Bi film (o) and a $Bi/Bi_{0.86}Sb_{0.14}$ multilayer film (x) relative to platinum. The top curve (+) is the absolute Seebeck coefficient relative to platinum and forms the zero for the absolute Seebeck coefficient scale.

The resistivity of these films can be compared with that of Bi and $Bi_{0.86}Sb_{0.14}$ single layer films. The resistivity of the $Bi_{0.86}Sb_{0.14}$ film is higher than that of the Bi film, as is found for bulk material (figure 7). The resistivity for the multilayer is higher than either the Bi or $Bi_{0.86}Sb_{0.14}$ single layer films. The multilayer films show no evidence of enhancement in the electrical conductivity from confinement of the conduction electrons in the multilayer structure. It is likely that the observed small increase in resistance is from scattering at the multilayer interfaces.

In conclusion we have demonstrated that multilayers of Bi and $Bi_{0.86}Sb_{0.14}$ can be deposited at high enough temperature to achieve epitaxy, and yet not get complete interdiffusion of the layers. This is the first time that high quality epitaxial multilayers of these materials have been produced at a length scale where quantum confinement effects may become apparent. The small modulation in the bandgap in this multilayer structure, however, is found not enhance the electrical conductivity.

Directions

We have demonstrated that it is possible to deposit multilayer films at high enough temperature to achieve epitaxial growth in the Bi, $Bi_{0.86}Sb_{0.14}$ system, and presented many of the important parameters for sputter deposition. This work is now being extended to the $Bi_{2-x}Sb_xTe_{3-y}Se_y$ compound thermoelectric materials. This family of materials includes some of the best thermoelectric materials known, as well as allowing a wide range of possible bandgaps. The similarity of crystal structure of these materials to Bi and $Bi_{0.86}Sb_{0.14}$ suggests that many of the parameters defined above can be applied to these more complicated materials. These high quality multilayers will allow us to explore the possibility of theoretically predicted inhancements in thermoelectric materials caused by quantum confignment in layered structures.

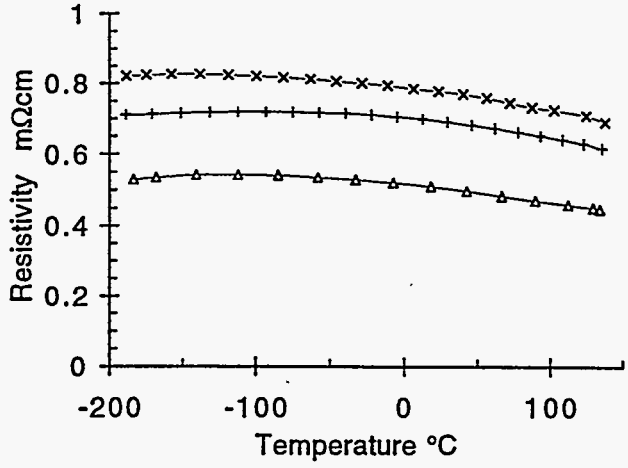

Figure 7: Resistivity for films of: Bi (Δ), $Bi_{0.86}Sb_{0.14}$ (+), and $Bi/Bi_{0.86}Sb_{0.14}$ multilayer (x).

Acknowledgments

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Improve thermoelectric efficiency using multilayer materials:

Hicks - Dresselhaus theory:

- Quantum well confinement
  - Taylor band structure
  - Increase conductivity

Experimental multilayer sputter deposition issues:

- Physical issues:
  - Flat abrupt interfaces - growth morphology
  - Large grain size - grain boundary scattering
  - Interdiffusion - low melting point metals
- Electronic issues:
  - Quantum confinement - barrier height
  - Doping - semiconductors

Outline:

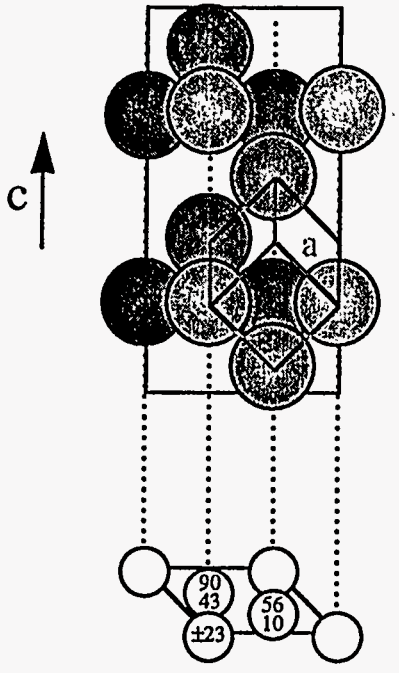
1) Growth and characterization of high quality Bi single layers
   - Suitable substrate
   - Optimize sputter conditions

2) Growth of epitaxial $Bi/Bi_{0.86}Sb_{0.14}$ multilayers
   - Electronic properties

3) Future direction

Crystal structure of Bi and Sb:

Rhombohedral structure.



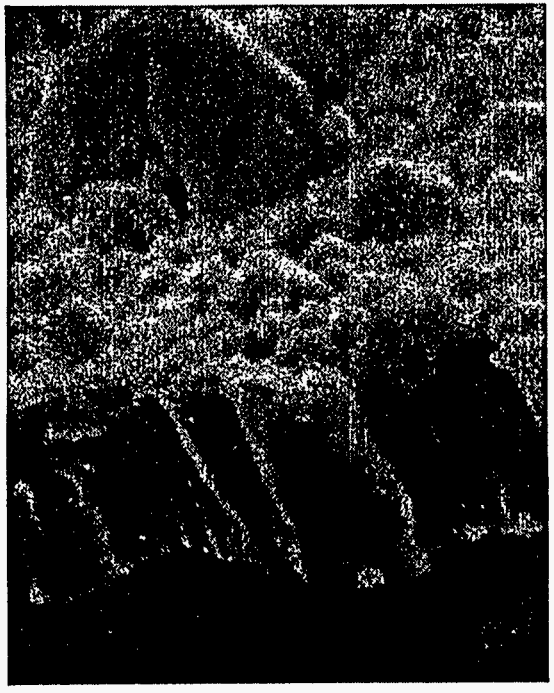
Very close to simple cubic,
but with a slight symmetry-breaking
distortion along the cubic 111 direction.

Atom density in the pseudo-cubic 111 direction is $1/a^2\sqrt{3}$,
while the faces are more dense with density $1/a^2$.

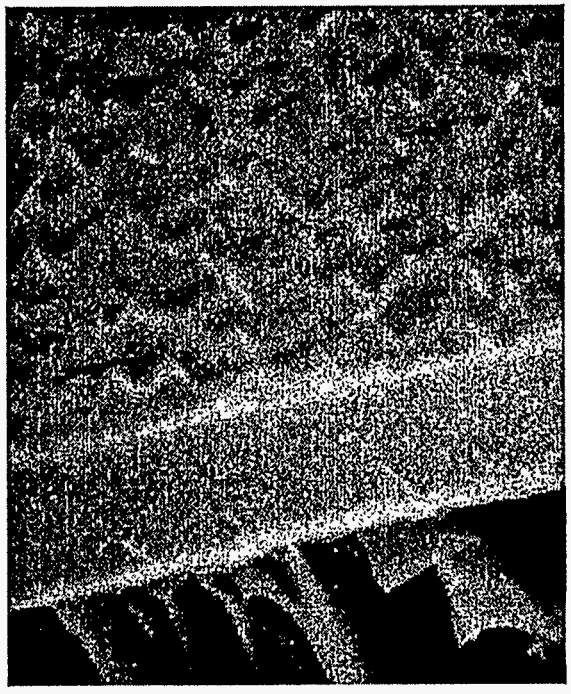
Substrate: cubic BaF_2, (111) face 3.6% smaller than Bi

SEM micrographs:

1μm thick Bi films



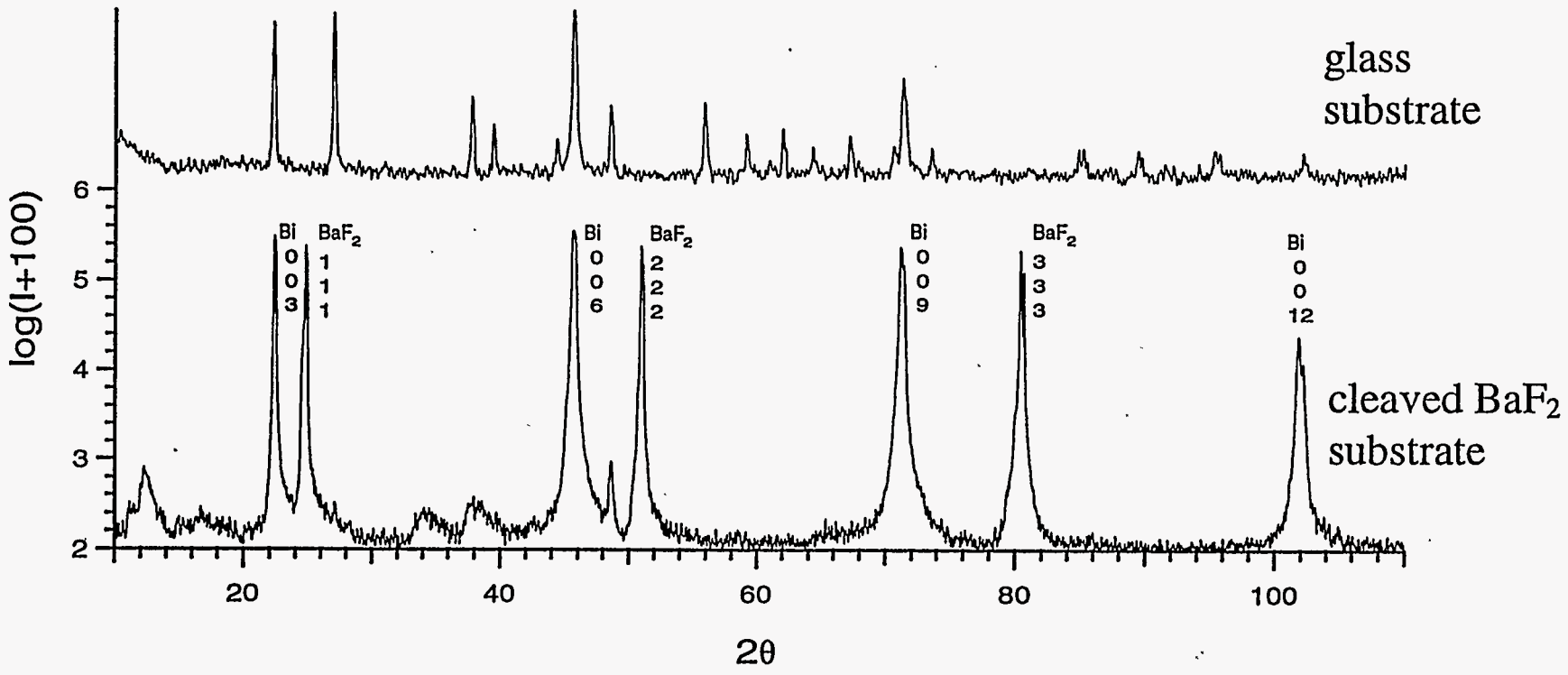
Polished BaF_2 100°C



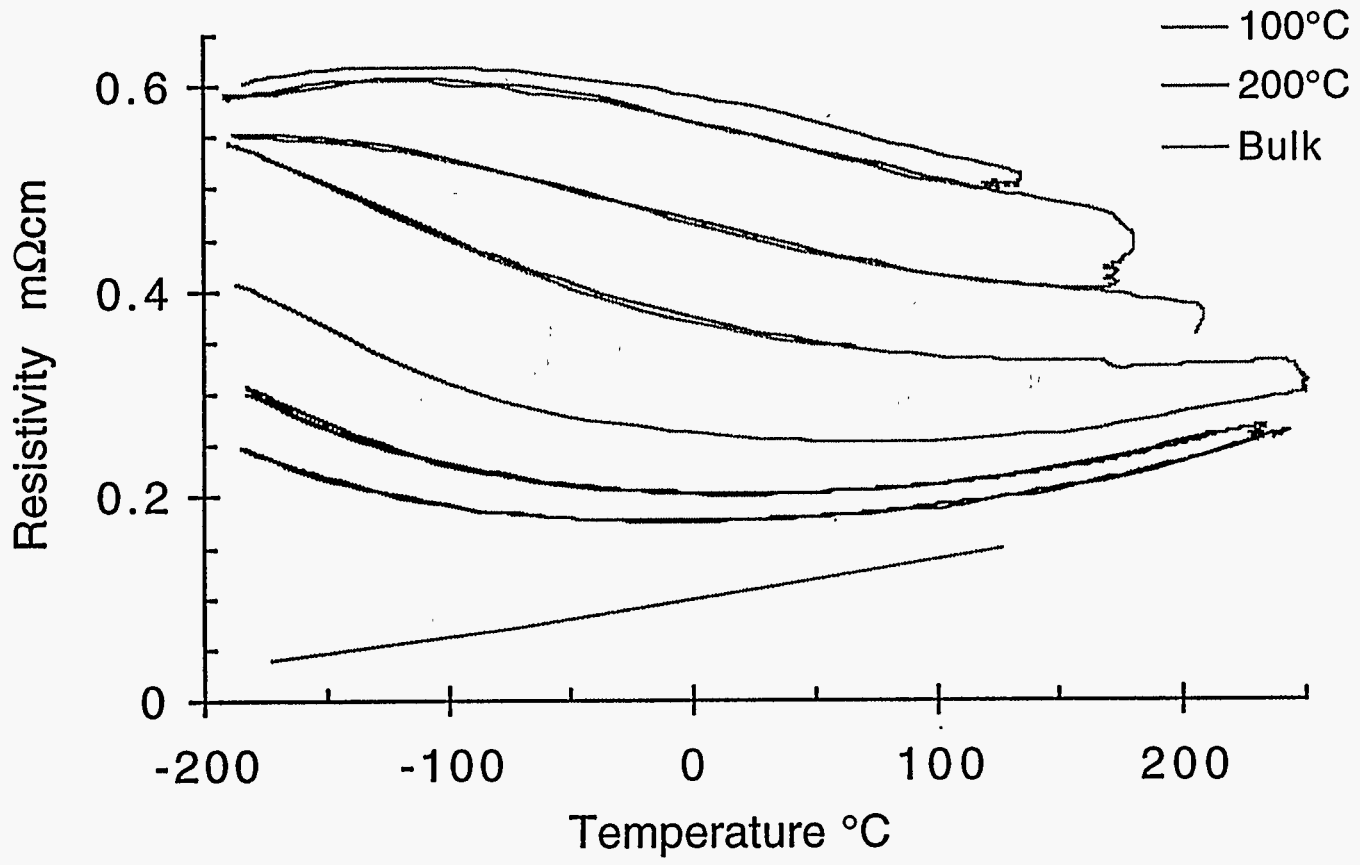
Cleaved BaF_2 100°C
Epitaxial growth

Cleaved BaF_2 200°C

X-ray diffraction of single layer Bi films:



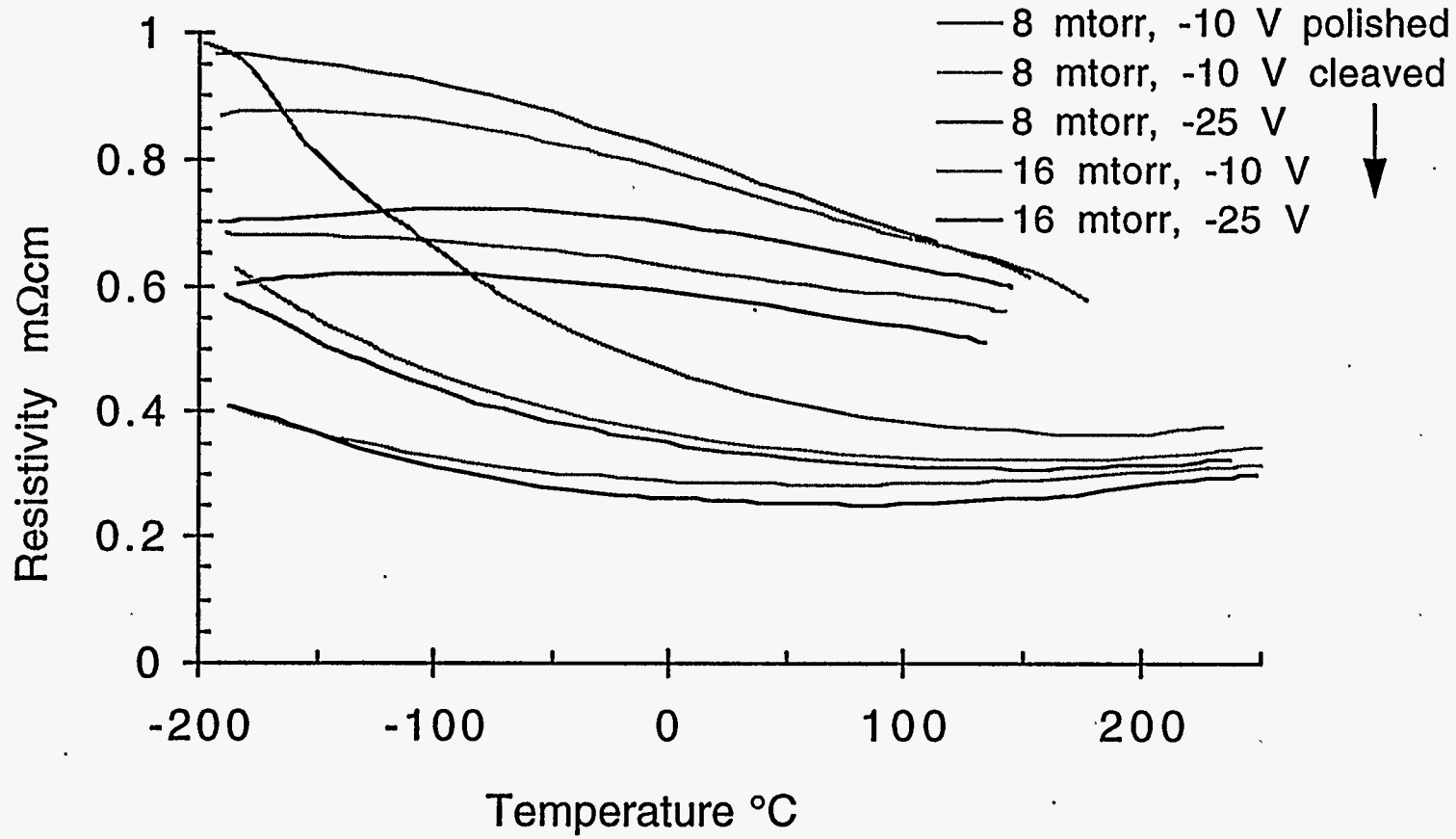
Resistivity: As a function of deposition temperature and annealing.
Single layer Bi films.



Higher temperature decreases the film resistivity.

Resistivity: Dependence on gas pressure and sample bias.

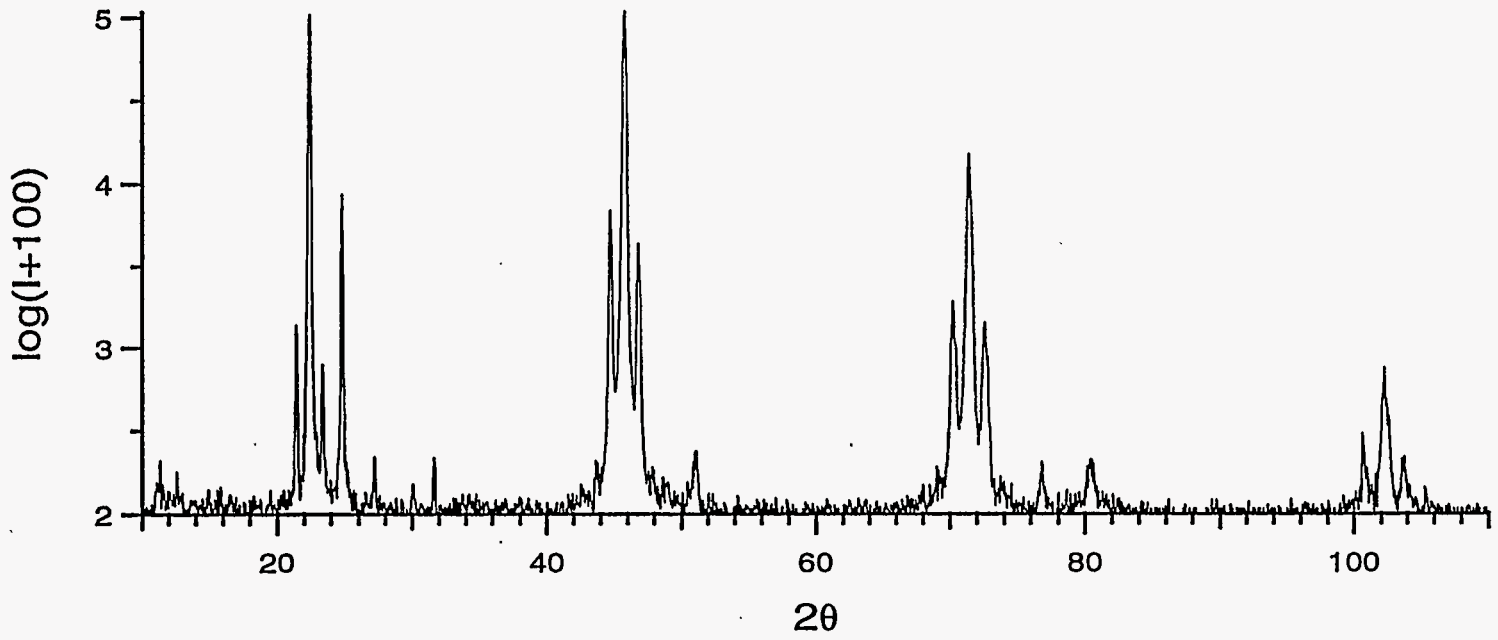
Single layer Bi films.



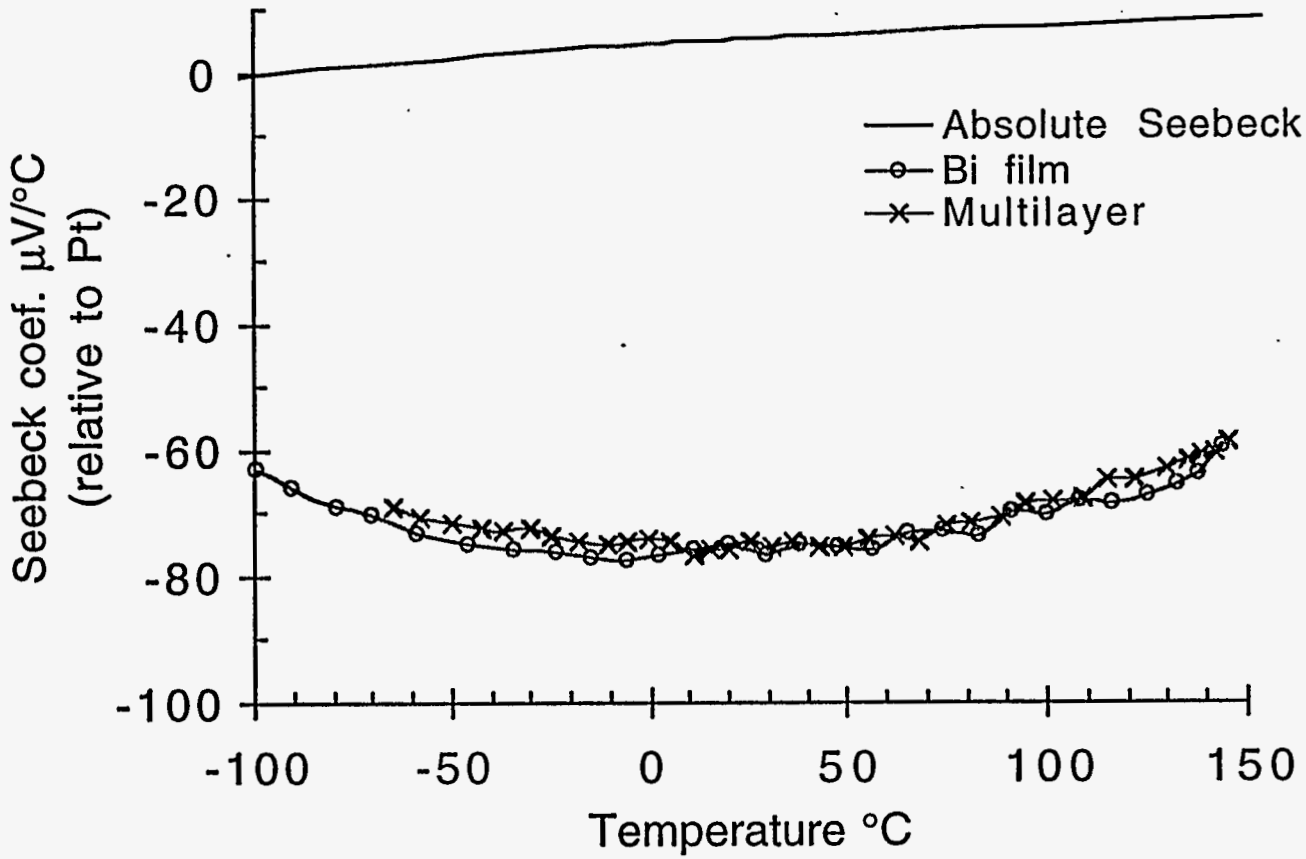
Increasing gas pressure and increasing sample bias decrease film resistivity

X-ray diffraction of $Bi/Bi_{0.86}Sb_{0.14}$ multilayer:

$$d = \frac{\lambda}{2(\sin\theta_b \pm \sin\theta_{ml})} = 91\text{Å}$$

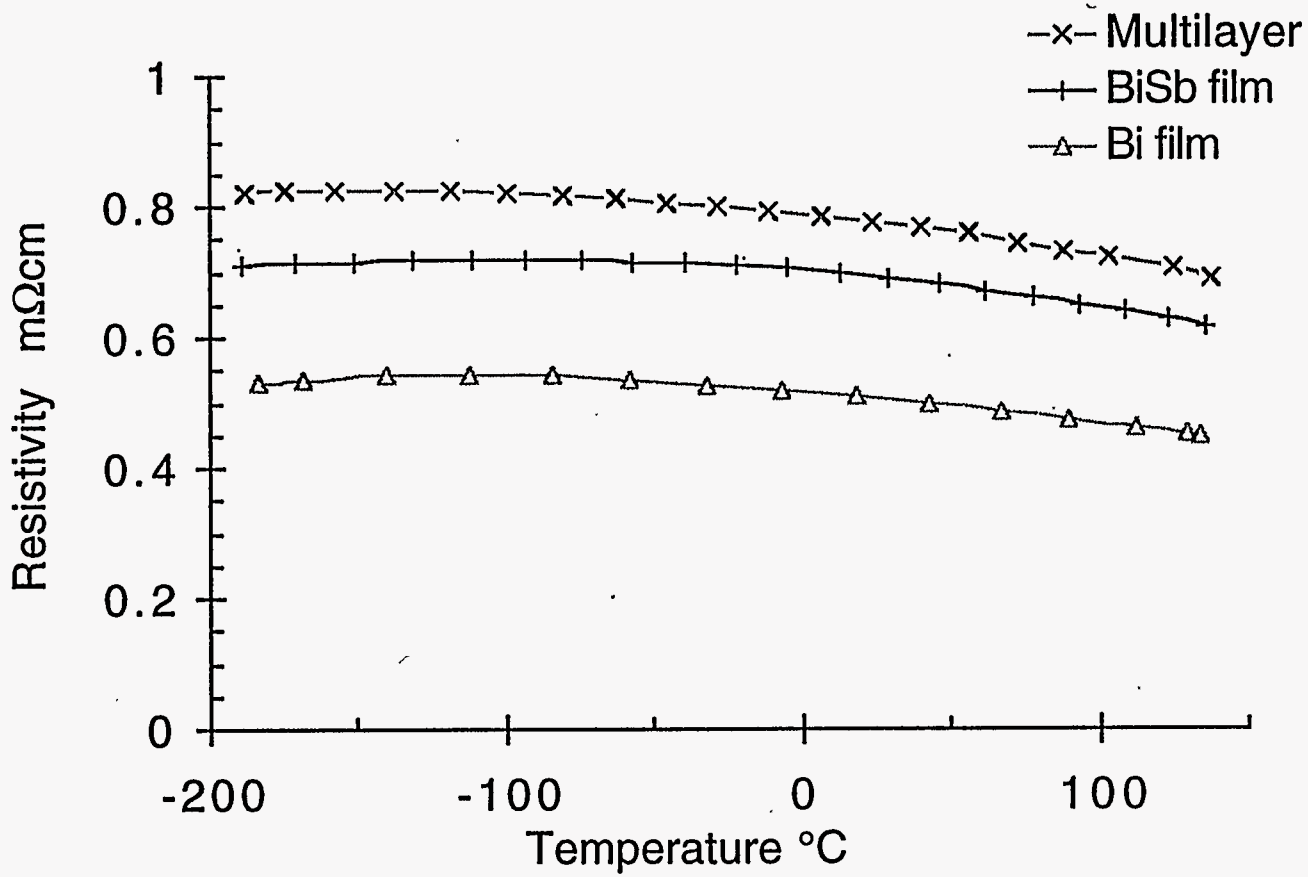


Seebeck coefficient:



Seebeck coefficient is unchanged in the multilayer structure.

Multilayer resistivity:



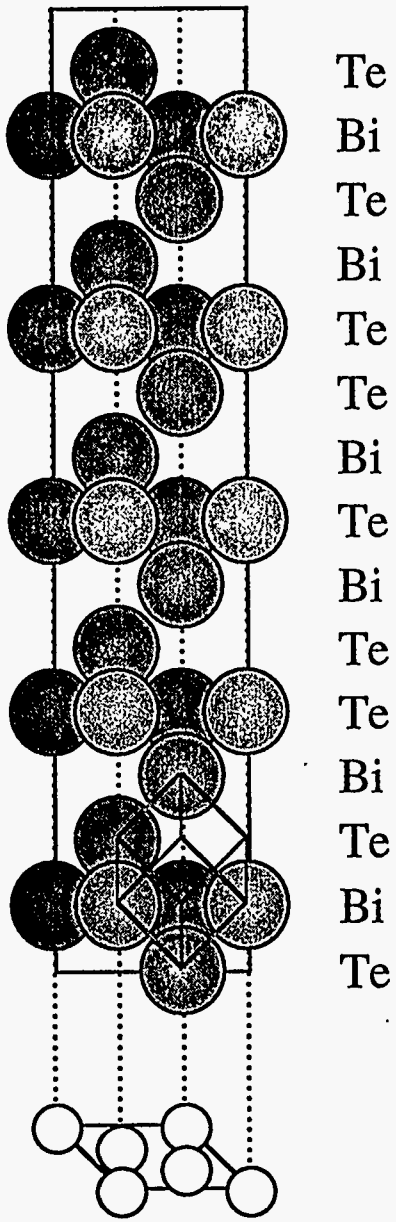
Resistance is higher in the multilayer.
Scattering from the layers.

Conclusions:

- Growth of high quality epitaxial Bi and $Bi_{0.86}Sb_{0.14}$ films by sputtering
- Epitaxial $Bi/Bi_{0.86}Sb_{0.14}$ multilayer
  - Resistance not improved
  - Not a great enough band gap difference between layers
- Sputtering techniques developed can be applied to other systems

Directions:

- Extend to $Bi_{2-x}Sb_xTe_{3-y}Se_y$ compound thermoelectrics
  - Good thermoelectrics
  - Large range of bandgap
  - Small amounts of interdiffusion is allowable
    - Interdiffusion caused low Seebeck coefficient in BiSb/PbTeSe



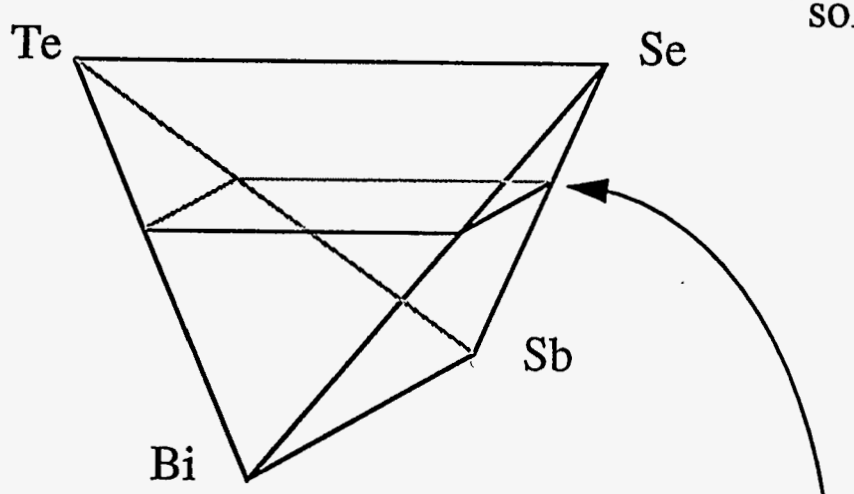
Bi_2Te_3
and solid solutions with Sb and Se

Sb replaces Bi
Se replaces Te

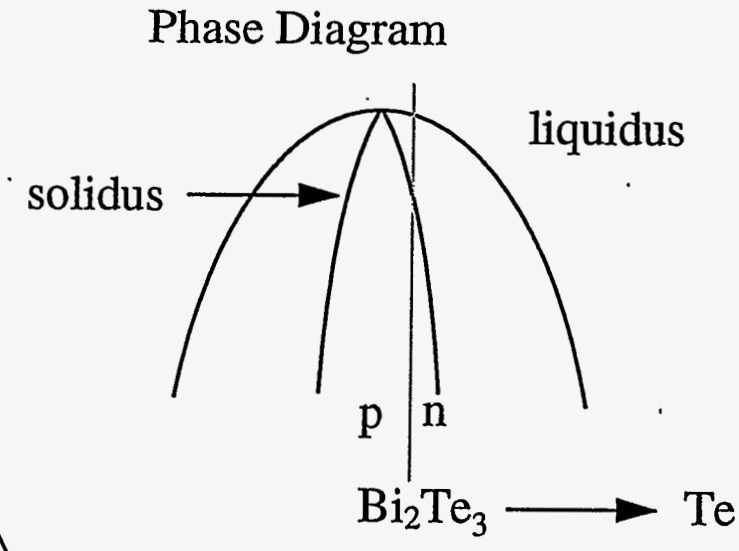
Rhombohedral

Layered compound

Bi,Sb,Te,Se system:



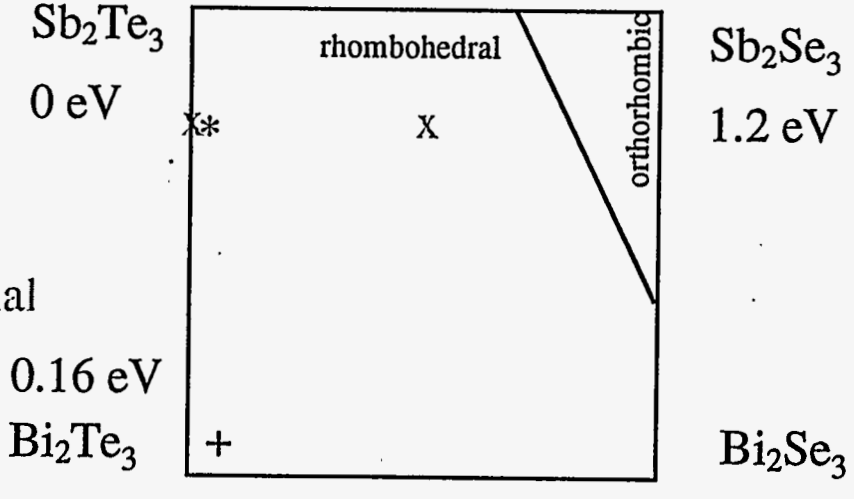
Phase Diagram



* best p type

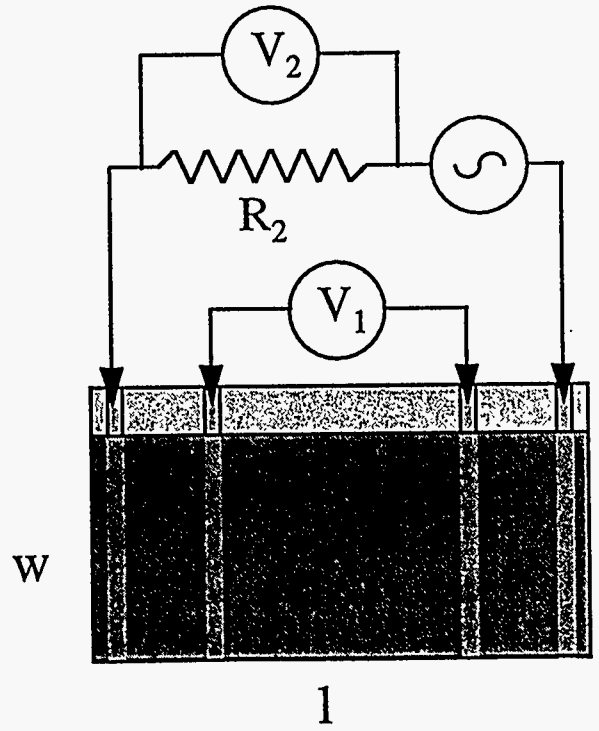
\+ best n type (SbI_3 doped)

x proposed multilayer material



Resistance:

Vacuum for a large temperature range
4 point probe technique
Alternating current
Eliminate induced temperature gradients
Lock-in amplifier
small voltages
Computerized data logging



$$\frac{1}{R} = \frac{V_2}{V_1 R_2} - \cancel{\frac{1}{R_s}}^{\,0}$$

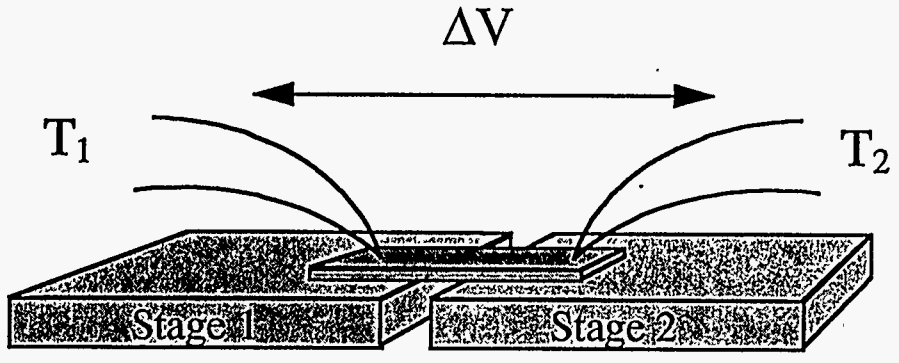
Measure thickness to find resistivity.

$$\rho = \frac{Rwd}{l} \approx Rd$$

Seebeck measurement:

$$S = \frac{\Delta V}{\Delta T}$$

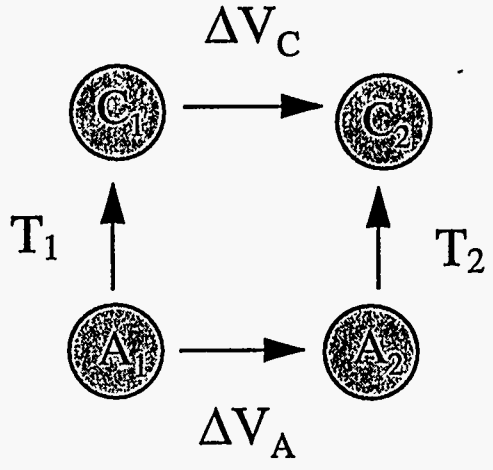
Vacuum for large temperature range
Computerized data logging
Calculate and display Seebeck coefficient
K Type thermocouple
Measure ΔV across chromel and alumel leads
Correct for ΔV induced in the thermocouple leads



Contact with silver paint
Electrical
Thermal

Substrate correction:

$$V_F = V_m + \frac{R_F}{R_S}\left(V_m - V_S\right)^{0}$$



$$S = \frac{V}{\Delta T}$$

Two ways to find ΔT:

$$T_2 - T_1 = \Delta T$$

$$\Delta V_C - T_2 - \Delta V_A + T_1 = 0$$

$$\Delta V_C - \Delta V_A = T_2 - T_1 = \Delta T$$

The voltages need to be corrected for the EMF generated in the leads between the sample and room temperature.

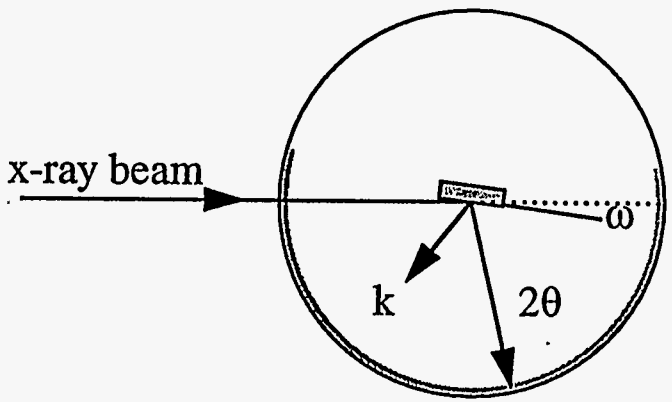
$$V_{A(T_1,T_2)} = \Delta V_A - \left(\int_{T_0}^{T_1} S_A(T)dT - \int_{T_0}^{T_2} S_A(T)dT \right)$$

6 ways to find the Seebeck coefficient.

Chromel:	$\frac{V_{C(T_1,T_2)}}{T_2 - T_1}$	$\frac{V_{C(T_1,\Delta T)}}{\Delta T}$	$\frac{V_{C(T_2,\Delta T)}}{\Delta T}$	Average
Alumel:	$\frac{V_{A(T_1,T_2)}}{T_2 - T_1}$	$\frac{V_{A(T_1,\Delta T)}}{\Delta T}$	$\frac{V_{A(T_2,\Delta T)}}{\Delta T}$	Standard deviation

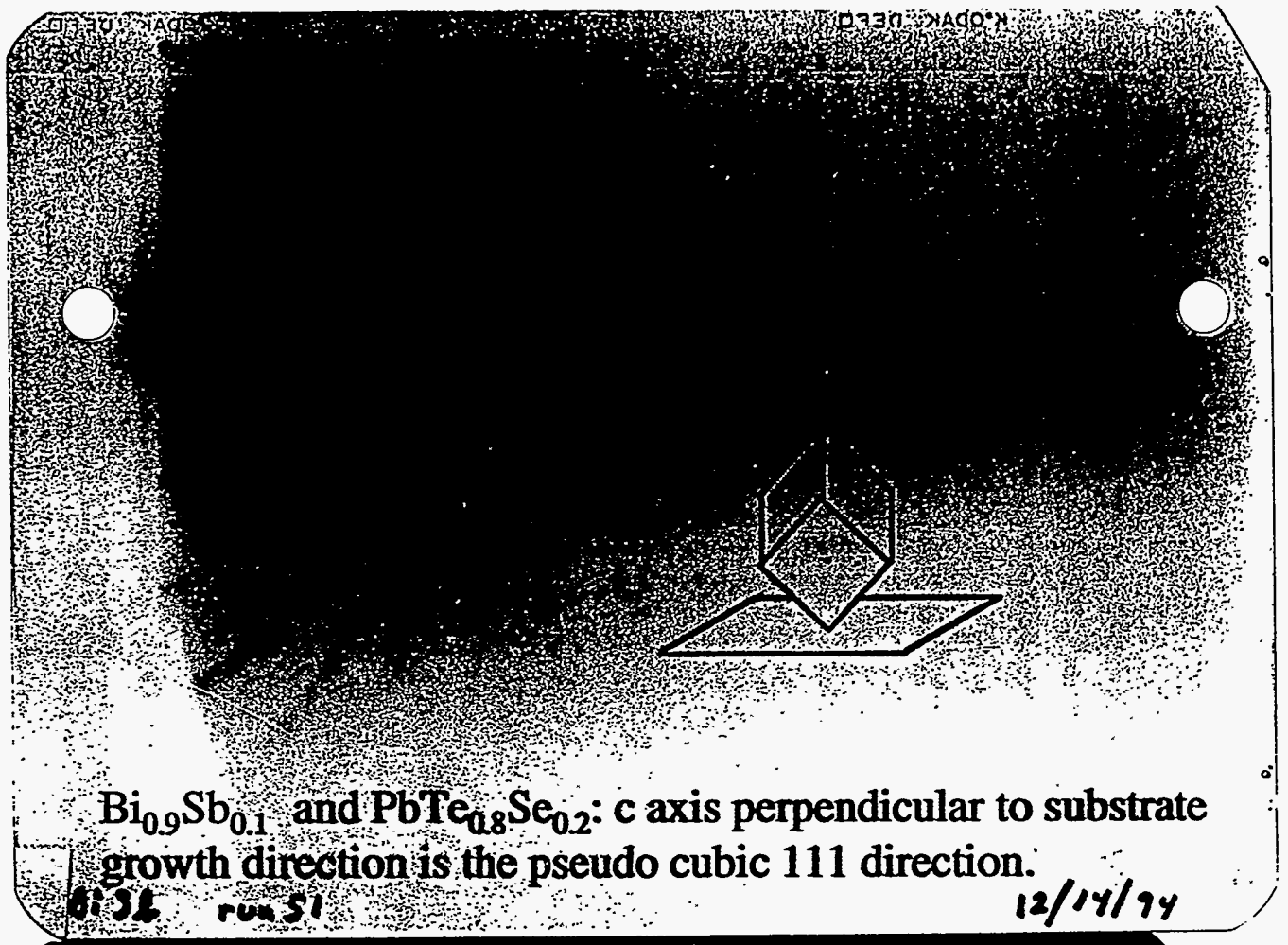
Structural characterization:

Read Thin Film Camera	Cu kα radiation monochromatic	bremsstrahlung white
Single crystal (epitaxial film)	none	spots
Textured film	spots	curves
Polycrystalline (random orientation)	rings	background



$$n\lambda = 2d\sin(\theta)$$

X-ray diffraction pattern pictures

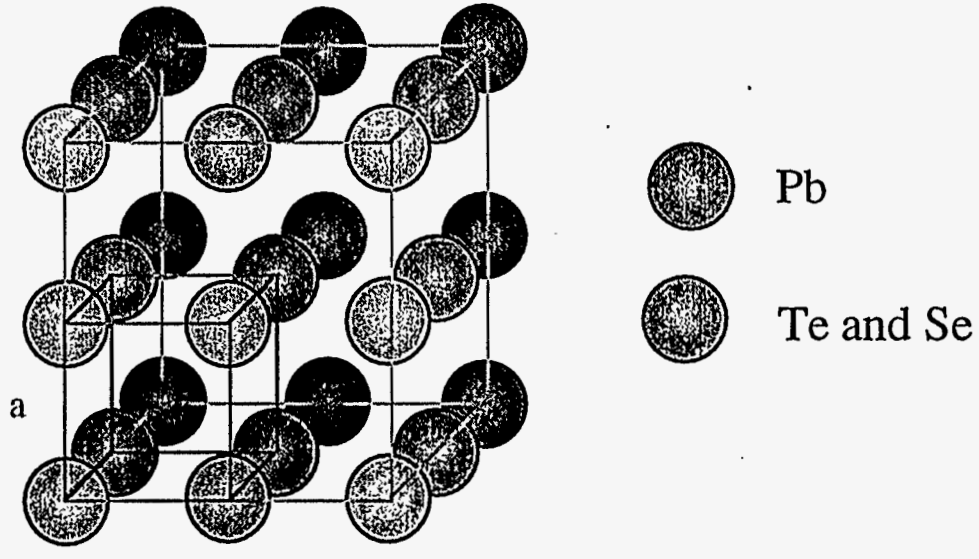


$Bi_{0.9}Sb_{0.1}$ and $PbTe_{0.8}Se_{0.2}$: c axis perpendicular to substrate growth direction is the pseudo cubic 111 direction.

6:38 run 51 12/14/94

Crystal structure $PbTe_{0.8}Se_{0.2}$:

NaCl Structure



Doping:

matrix	n	p
Bi_2Te_3 (and family)	Te SbI_3	Bi Pb
PbTe	Pb PbI_2, Ge_2Te_3	Te Na_2Te
Bi, $Bi_{1-x}Sb_x$	undoped	Sn,Pb

Te reduces the Seebeck in Bi.

Multilayers:

$Bi_{0.9}Sb_{0.1}/PbTe_{0.8}Se_{0.2}$

$Bi_{0.9}Sb_{0.1}/Bi_2Te_3$

$Bi/Bi_{0.9}Sb_{0.1}$

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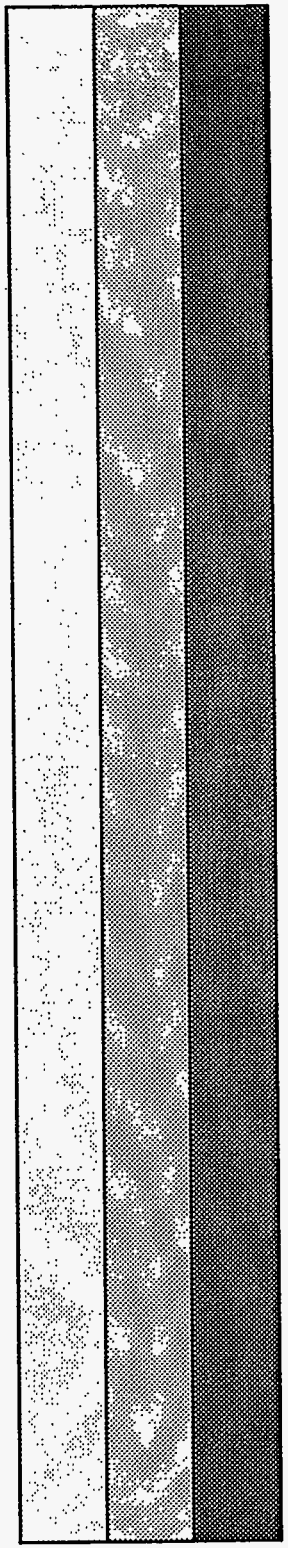

Recycled
Recyclable