PREPARATION AND THERMOELECTRIC PROPERTIES OF SOME ARSENOPYRITE PHASES BASED ON $RuSb_{2-2x}Te_{2x}$ AND $RuSb_{2-2x}Se_{2x}$ SOLID SOLUTIONS

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Abstract

The synthesis and some properties of several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions is reported. The existence of solid solutions with the arsenopyrite structure in the system $RuSb_{2-2x}Te_{2x}$ for $0.25<x<0.65$ was confirmed. in the $RuSb_{2-2x}Se_{2x}$ system, no series of solid solutions was found and only the RuSbSe phase was prepared in single phase form. in an effort to assess the potential of these materials for thermoelectric applications, electrical resistivity, Seebeck coefficient, and thermal conductivity were measured between 300 and 1000K. Low thermal conductivity values were achieved for the ternary and quaternary compositions investigated, about five times lower than those obtained for binary compounds such as $IrSb_2$ and $CoSb_2$ at room temperature. **in** these materials, scattering of" the phonons by electrons transferred between mixed valence ions accounts to the low thermal conductivity observed. Despite these low values, the samples are characterized by low carrier mobility and high electrical resistivit y values, resulting in thermoelectric figures of merit lower than state-of-the-a~ t thermoelectric materials.

Keywords: A. intermetallic compounds, A. semiconductors, 1). transport properties.

1. introduction

Thermoelectric devices are reliable, can operate unattended in hostile environments and are also environmentally friendly but new more efficient materials are needed to expand their range of applications. The identification and development of novel more efficient thermoelectric materials has been the focus of a renewed interest over the past few years [1,2]. As part of a broad search for advanced thermoelectric materials, we have investigated the thermoelectric properties of several materials with the arsenopyrite crystallographic structure and recently reported on the preparation and characterization of the thermoelectric properties of $Ir_xCo_{1-x}Sb_2$ alloys [3], Many binary and ternary compounds with the pyrite, marcasite and arsenopyrite type of crystal structure are known [4]. These three types of crystal structure are closely related and the arsenopyrite

can be considered as a monoclinic distortion of the marcasite lattice. Many of these compounds are semiconductors [4] but their usefulness for thermoelectric applications has been little investigated. Some Seebeck coefficient and energy band gap data can be found in [5] for several ternary arsenopyrite compounds. 1 lowever, more work is needed to fully assess the potential of these materials and, in particular, thermal conductivity data would be of interest. Based on our investigation of binary compounds [3], their thermal conductivity is too large to be useful thermoelectrics and efforts should focus on thermal conductivity reduction. Alloys between binary compounds as well as ternary compounds are expected to have lower thermal conductivity in particular because of the possible phonon scattering point defect scattering,

As part of our investigation of arsenopyrite materials as thermoelectric materials, we have investigated the thermoelectric properties of several phase based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions, 11 was found that a complete series of solid solutions exists in the system $RuSb_{2-2x}Te_{2x}$ [6]. The alloys have the marcasite structure for $x>0.65$, the arsenopyrite structure for $0.25\leq x\leq 0.65$, and the loellingite structure for $x<0.25$. A band gap of 0.5 CV was determined from electrical resistivity measurements for the composition RuSbTe [5]. The infrared reflection spectra was also studied for this same composition [7], The investigation of the existence of solid solutions between $RuSb_2$ and $RuSe_2$ has not been completely investigated and only the existence of the solid solution RuSbSe was reported. Based on thermoelectric power and electrical resistivity measurements, this compound was found to be a semiconductor with an estimated band gap of about 0.35 CV [5]. We report in this paper on the preparation and characterization of the thermoelectric properties of several arsenopyrite phases based on based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions.

2. Experimental

Single phase, polycrystalline samples were prepared by heating stoichiometric mixtures of the elements with intermediate crushing and subsequent hot-pressing. Ruthenium (99.95%), cobalt (99.99%), antimony (99.9999%), tellurium (99.999%), and selenium (99.999%) powders were used to synthesize the samples. The mixtures of the elements were placed in a plastic vial before being loaded in a steel die where they were compressed into dense cylindrical pellets. The pellets were sealed under vacuum in quartz ampoules which were heated for 5 days at temperatures between 873 and 1073K, depending on the composition. The products were then removed from the ampoules, crushed, ground in an agate mortar, sealed again in quartz ampoules and heated for 5 days at temperatures between 873 and 1073K. Products of the annealing were then removed from the ampoules and analyzed by x-ray diffractometry (XRD). When single phase, the powders were hot-pressed in graphite dies into dense samples about 10 mm long and 6.35 mm in diameter. The hot-pressing was conducted at a pressure of about 20,000 psi and at a temperature between 1023 and 1 123K for about 2 hours under argon atmosphere. The exact temperatures at which the hot-pressing was conducted are listed in '1'able 1 for all the samples prepared.

XRD analysis was performed at room temperature on a Siemens D-500 diffractometer using Cu-K_α radiation, Small additions of Si powders were made to the samples as an internal standard, Selected samples cut from the hot-pressed bats were polished using standard metallographic techniques. Microprobe analysis (MPA) was performed on these samples to determine their atomic composition using a JEOL JXA-733 electron superprobe operating at 20x103 V of accelerating potential and 25x10-g A of probe current. Pure elements were used as standards and X-ray intensity measurements of peak and background were conducted by wavelength dispersive spectrometry. The density of the samples was calculated from the weight and dimensions of the samples. The decomposition temperature of selected samples was also determined using differential thermal analysis (1)TA). A Dupont 1600°C DTA apparatus was used for DTA measurements. The samples were scaled under 1 O"s Torr vacuum in quartz ampoules 5 mm in diameter and 15 mm long. Argon was used as the purge gas and the decomposition temperatures were determined from the heating curves with an heating rate of 2°C rei"]. The accuracy was estimated at ± 1 0°C.

Samples in the form of disks (typically a 1 mm thick, 6.35 mm diameter slice) were cut from **the** bars using a diamond saw for electrical and thermal transport property measurements. All samples were characterized at room temperature by Seebeck coefficient, 1 lall effect and electrical resistivity measurements. I ligh temperature resistivity, I Ian effect, Seebeck coefficient, thermal diffusivity, and heat capacity measurements were also conducted on selected samples between mom temperature and about 1000K. The electrical resistivity (p) was measured using the van der Pauw technique with a current of 100 mA using a special high temperature apparatus [8]. The 1 lall coefficient (R_H) was measured in the same apparatus with a constant magnetic field value of ~ 10,400 Gauss. The carrier density was calculated from the I fall coefficient, assuming a scattering factor of 1 in a single carrier scheme, by $p/n = 1/R_H e$ where p and n are the densities of holes and electrons, respectively, and c is the electron charge. The Hall mobility (μ_H) was calculated from the Hall coefficient and the resistivity values by $\mu_H = R_H/p$. The errors were estimated to be ± 0.S% and ± 2% for the resistivity and 1 lall coefficient data, respectively. The Seebeck coefficient of the samples was measured on the same samples used for resistivity and f lall coefficient measurements using a high temperature light pulse technique [9]. The error of the Seebeck coefficient measurement was estimated to be less than ± 3%. The heat capacity and thermal diffusivity were measured using a hash diffusivity technique [1 0]. The thermal conductivity was calculated from the experimental density, heat capacity, and thermal diffusivity values. The overall error in the thermal conductivity measurements was estimated to be about ± 1 0%.

3. Results and discussion

3.1. XRD, MPA, density, and DTA measurements results

The results of x-ray, microprobe, density, and differential thermal analysis measurements are summarized in '1'able 1. The x-ray results obtained for $RuSb_{2-2x}Te_{2x}$ alloys with

$0.3 \leq x \leq 0.6$ confirmed the existence of a continuous series of solid solutions with the arsenopyrite structure for $0.25 \leq x \leq 0.65$ established by Lutz et al. [6]. Similar findings were reported for the systems $FeSb_2$-$FeTe_2$ and $CoSb_2$-$CoTe_2$ [11]. Two single phase alloys between $RuSb_{2-2x}Te_{2x}$ compositions and the arsenopyrite compound $CoSb_2$ were also prepared. The composition of these samples is listed in Table 1. The density of the hot-pressed $RuSb_{2-2x}Te_{2x}$-based samples is listed in Table 1 and was found to be between 94 and 97% of the theoretical density. Microprobe analysis confirmed that the samples were single phase and the compositions determined by MPA are listed in Table 1 and were found to be close to the nominal compositions. We also successfully prepared samples of $RuSb_{2-2x}Se_{2x}$ for $x = 0.5$ but x-ray analysis of samples with $x = 0.35$ and 0.6 revealed a multiphase mixture, mostly composed of the compounds $RuSb_2$ and $RuSe_2$. This result suggests that, unlike to the $RuSb_{2-2x}Te_{2x}$ system, $RuSb_2$ and $RuSe_2$ do not form a complete series of solid solutions. We also prepared single phase samples of alloys between RuSbTe and RuSbSe and RuSbSe and $CoSb_2$. All single phase RuSbSe-based samples were hot-pressed and the density of the hot-pressed samples is listed in Table 1 as well as their composition, determined by MPA. We found an average decomposition temperature of 966°C and 849°C for the solid solutions RuSbTe and RuSbSe, respectively.

3.2. Thermoelectric properties

The room temperature properties of several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions are listed in Table II. For all samples, the values for the Seebeck coefficient, Hall carrier concentration, Hall mobility, and electrical resistivity are indicative of a semiconducting behavior with mixed conduction by both electrons and holes at room temperature. The carrier mobility values are low, resulting in high electrical resistivity values. For $RuSb_{2-2x}Te_{2x}$ solid solutions with $x = 0.5$ and $x = 0.3$, the conductivity is of p-type at room temperature while for $x = 0.6$ the conductivity is of n-type. In a recent investigation of the thermoelectric properties of $Ir_xCo_{1-x}Sb_2$ alloys [3], it was found that single crystals of the arsenopyrite compound $CoSb_2$ had relatively high carrier mobility and low electrical resistivity values. In an attempt to reduce the electrical resistivity of $RuSb_{2-2x}Te_{2x}$ solid solutions, we prepared $(RuSb_{2-2x}Te_{2x})_{1-y}$-$(CoSb_2)_y$ alloys. The room temperature properties of these alloys are also listed in Table II. Although these alloys have a lower electrical resistivity than Te-rich $RuSb_{2-2x}Te_{2x}$ solid solutions, the lowest resistivity was achieved for the Ir-poor sample (2 ARST3) with a room temperature value of 59 mΩcm. The addition of Co did not result in an increase of the carrier mobility. The electrical resistivity of the RuSbSe sample is also high and even higher for the $(RuSbSe)_{0.5}$-$(RuSbTe)_{0.5}$ alloy. The $(RuSbSe)_{0.9}$-$(CoSb_2)_{0.1}$ alloy has a significantly lower electrical resistivity value of 37.9 mΩcm at room temperature.

The high-temperature electrical resistivity and Seebeck coefficient values for the samples listed in Table II are shown in Figs. 1 and 2, respectively. For all samples, the electrical resistivity decreases with increasing temperature. A band gap value of 0.61 and 0.59 eV was estimated for the solid solutions RuSbTe and RuSbSe, respectively from the quasi-linear variations of the electrical resistivity at high temperatures. This is in good

agreement with a previous estimate of 0.5 eV for RuSbTe [5] but slightly higher than the estimate of 0.35 CV for RuSbSe [5]. The lowest electrical resistivity value was achieved for the $(RuSbSe)_{0.9}$-$(CoSb_2)_{0.1}$ alloy with a value of 8 mΩcm at a temperature of 1000K. This value is still relatively high considering that the optimal value for achieving maximum thermoelectric efficiency for a classical semiconductor is about 1 mΩcm. The Seebeck coefficient values arc shown in Fig. 2. All Sb-rich compositions have p-type Seebeck coefficient. The lowest electrical resistivity corresponds to the highest Seebeck coefficient. For example, the sample with the highest room temperature Seebeck coefficient (1ARSS3) has also the lowest electrical resistivity. This is also an indication that both electrons and holes participate to the conduction which reduce the magnitude of the Seebeck coefficient especially around room temperature.

Wc present in Fig. 3 the thermal conductivity of l<tlSbz.j,rl'cz,-based alloys. The values arc also compared to those obtained for the binary compounds It Sb_2 and $CoSb_2$ [3]. The room temperature values are ranging between 25 and 49 $mWcm^{-1}$ K-'. This is a significant decrease compared to the values in the order of 110 mWcm '1K"' for $CoSb_2$ and $IrSb_2$ [3]. The thermal conductivity is almost temperature independent for all samples except for alloys with $CoSb_2$ which show an increase in the thermal conductivity at the highest temperatures. This can bc attributed to a phase transition as it was observed in $CoSb_2$ [3]. For $RuSb_{2-2x}Te_{2x}$ solid solutions, the minimum in the thermal conductivity was expected to occur for x= **0.5 where** maximum mass and size fluctuations occur on the anion site However, wc found that the composition RuSbTe has in fact the highest thermal conductivity of all $RuSb_{2-2x}Te_{2x}$ arsenopyr itc solid solutions measured (see '1'able II). in addition to point defect scattering, other phonon scattering mechanisms must bc taken into account to explain this unusual finding. It is also possible that the phonons arc scattered by electrons transferred between Ru ions in these samples as it was recently suggested to explain the low lattice thermal conductivity of the skutterudite compound $Ru_{0.5}Pd_{0.5}Sb_3$ [J 2]. The Ru atoms arc expected to change their valence state from 4+ in $RuSb_2$ to 2+ in $RuTe_2$ in the $RuSb_{2-2x}Te_{2x}$ series. Therefore, depending on the x value, the ratio between the different Ru ions varies, resulting in different phonon scattering rates, Our experimental results seem to indicate that the highest scattering rates occur near x= 0.3. These results arc supported by the data on $(RuSb_{2-2x}Te_{2x})_{1-y}(CoSb_2)_y$ alloys. A decrease in thermal conductivity is observed for the $(RuSbTe)_{0.9}(CoSb_2)_{0.1}$ alloy compared to RuSbTe. This is due to the increase mass and size fluctuations introduced by the Co atoms in the lattice. However, the thermal conductivity is even lower for the $(RuSb_{1.3}Te_{(1.7)})_{0.9}(CoSb_2)_{0.1}$ alloy which also suggests that a phonon scattering by electron transfer between mixed valence Ru ions occurs.

The thermal conductivity values for **RuSbSc-based** alloys are shown in Fig. **4.** The room temperature values arc ranging between 23 and 49 $mWcm^{-1}K^{-1}$ and the thermal conductivity is almost temperature independent. The thermal conductivity of RuSbSe is lower than for RuSbTe because of the higher mass fluctuation produced by the Sc atoms compared to the Te atoms, The thermal conductivity reaches 25 $mWcm^{-1}$ K-l for the $(RuSbTe)_{0.5}(RuSbSe)_{0.5}$ due to phonon scattering by point defects, However, it seems that point defect phonon scattering dots not produce scattering rates as high electron transfer

phonon scattering between mixed valence ions, as observed for RuSbrl'c-based alloys. This scattering mechanism could not be observed in RuSbSc-based alloys because single phase samples with compositions shifted from the RuSbSe stoichiometry could not be prepared. For the $(RuSbSe)_{0.9}(CoSb_2)_{0.1}$ alloy, a significant decrease in thermal conductivity was observed compared to RuSbSe due to the addition of Co atom into the lattice structure. For this composition, the thermal conductivity is nearly temperature independent with a value of about 22 $mWcm^{-1}K^{-1}$.

Low thermal conductivity values were achieved for several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions, comparable to those obtained for state-of-the-at(thermoelectric materials. f lowever, the power factor values (α^2/ρ) arc also low mainly due to the iow carrier mobility of the samples, resulting in high electrical resistivity values. A maximum power factor value of only 5 $\mu Wcm^{-1}K^{-1}$ was obtained for sample 1ARSS3 at a temperature of 900K, The calculated maximum thermoelectric figure of merit ZT ($ZT = \alpha^2/\rho\lambda$) was 0.2 at a temperature of 900K for sample 1ARSS3. Because of the low carrier mobility in these materials, it is unlikely that ZT values higher than the maximum ZT value of 1 common to all state-of-the-art thermoelectric materials can be achieved.

4, **Conclusion**

W c have studied the existence anti thermoelectric properties of several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions. We have found that all binary and ternary compositions investigated have a semiconducting behavior with low thermal conductivity values. In addition to mass and strain fluctuations electron transfer between mixed valence ions was considered as a possible phonon scattering mechanism to explain the low thermal conductivity values. Although the samples exhibit relatively large Seebeck coefficient and low thermal conductivity values, the carrier mobility values in the samples are low and a maximum thermoelectric figure of merit ZT of only 0.2 was obtained at a temperature of 900K. This is significantly lower than the upper limit of 1 common to the best thermoelectric materials known to date. Although no efforts was done to optimize the properties of these samples, it is unlikely that these materials can outperform state-of-the-art thermoelectric materials.

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Table captions

Table I. X-ray, microprobe, differential thermal analysis, and density results for several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions

Table II. Hall carrier concentration (n/p), Hall mobility (p), electrical resistivity (p), Seebeck coefficient (α), thermal conductivity (λ), and energy band gap (ΔE) values for several hot-pressed arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions

Figure captions

Figure 1. Electrical resistivity versus inverse temperature for several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions

Figure 2. Seebeck coefficient versus temperature for several arsenopyrite phases based on $RuSb_{2-2x}Te_{2x}$ and $RuSb_{2-2x}Se_{2x}$ solid solutions

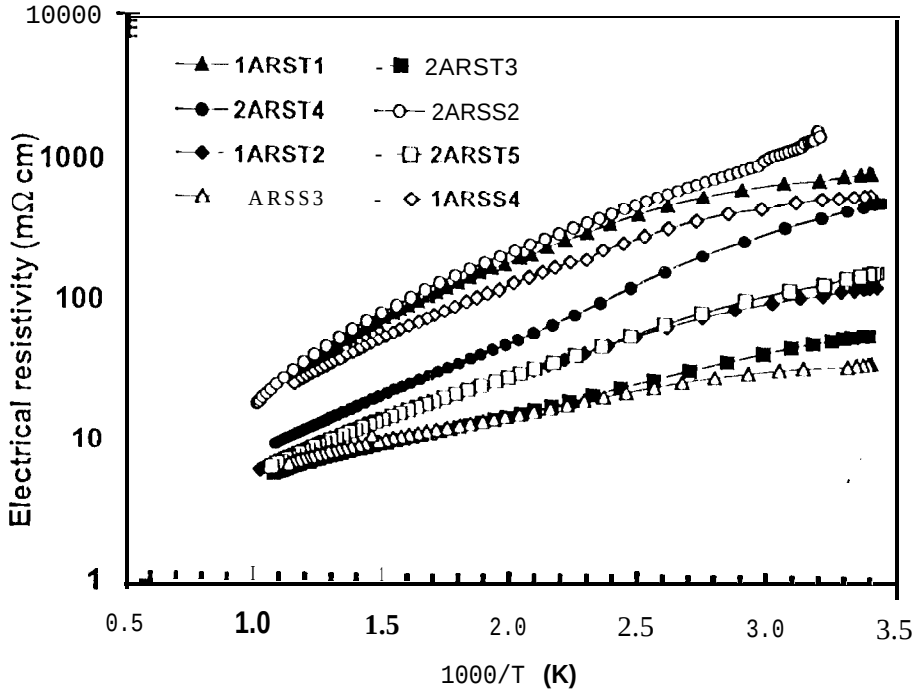
Figure 3. Thermal conductivity versus temperature for $RuSb_{2-2x}Te_{2x}$ -based alloys. Values for the binary compounds $IrSb_2$ and $CoSb_2$ are also shown for comparison [3].

Figure 4. Thermal conductivity versus temperature for RuSbSe- based alloys. Values for the binary compounds $IrSb_2$ and $CoSb_2$ are also shown for comparison [3].

sample	Nominal composition	X-ray results	Hot-pressing temperature (“c)	Geometrical density (g cm”)	Decomposition temperature (°C) Ru	Microprobe results (at%) Sb	Te	Se	Co
1ARS11	RuSbTe	single phase	850	8.73	966	31.3 34	34,7	-	-
?ARS13	$RuSb_{1.4}Te_{0.6}$	single phase	850	8.88		31,3 48	20.6	-	-
?ARS14	$RuSb_{0.8}Te_{1.2}$	single phase	850	7.47	-	31.2 77	4?,2	-	-
1ARS12	$(RuSbTe)_{0.9}(CoSb_2)_{0.1}$	single phase	800	8.56		27.6 39	29.9	-	3.5
?AF{S15	$(RuSb_{1.3}Te_{0.7})_{0.9}(CoSb_2)_{0.1}$	single phase	850	8.62	-	**29.3 49**	**19,9**	-	**2,2**
1ARSS4	RuSbSe	single phase	750	8.01	849	3?.1 34	33.9	-	-
2ARSS2	$(RuSbSe)_{0.}$,(RuSbTe),,	single phase	750	8.36		31.6 34	17.4	17	-
1ARSS3	$(RuSbSe)_{0.9}(CoSb_2)_{0.1}$	single phase	800	8.19		29 38	30.7	-	2.6
1ARSS5	$RuSb_{1.3}Se_{0.7}$	mutliphase	-			-	-	-	-
1ARSS6	$RuSb_{0.8}Se_{1.2}$	mutliphase	-			-	-	-	-

Sample	Nominal composition	Conductivity type	n/p (cm^{-3})	μ ($cm^2 V^{-1} s^{-1}$)	ρ ($10^{-3}\ \Omega$ cm)	(1. ($\mu V K^{-1}$)	λ at 370K ($mW cm^{-1}K^{-1}$)	ΔE (ev)
1ARST1	RuSbTe	p	2.06E+20	0.03	861.0	66.0	49	0.61
2ARST3	$Rush,._4Te_{.6}$	p	2.46E+20	0.43	59.9	13?	?1	
2ARST4	$RuSb_{0.8}Te_{12}$	n	4.05E+20	0.03	5?7. ?	33	36	
1ARST2	$(RuSbTe)_{0.9}(CoSb_2)_0$,	p	1.16E+20	0.41	131.7	134	37	
2ARST5	$(RuSb_{1.3}Te_{0.7})_{0.9}(CoSb_2)_0$,	p	1.29E+20	0.?9	166.3	1?5	25	
1ARSS4	RuSbSe	p	6.69E+20	0.01	597.1	37	34	0.6
2ARSS2	$(RuSbSe)_{0.5}(RuSbTe)_{0.5}$	p	2.62E+20	0.01	1542.0	87	25	
1ARSS3	$(RuSbSe)_{0.9}(CoSb_2)_0$,	p	4.45E+20	0.38	37.9	154	23	

Table II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100
10
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Electrical resistivity (mΩ cm)
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2ARST3
2ARST4
2ARSS2
1ARST2
2ARST5
ARSS3
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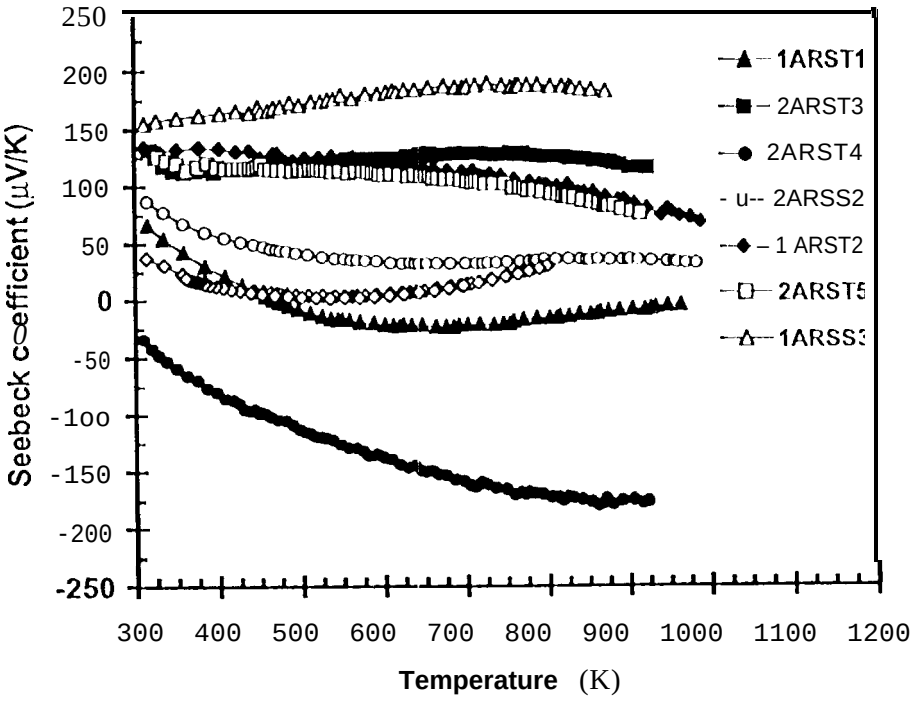
1.5
2.0
2.5
3.0
3.5
1000/T (K)

Fig (1)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150
100
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-50
-100
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-200
-250
Seebeck coefficient (µV/K)
300
400
500
600
700
800
900
1000
1100
1200
Temperature (K)
1ARST1
2ARST3
2ARST4
2ARSS2
1 ARST2
2ARST5
1ARSS3

Fig (2)

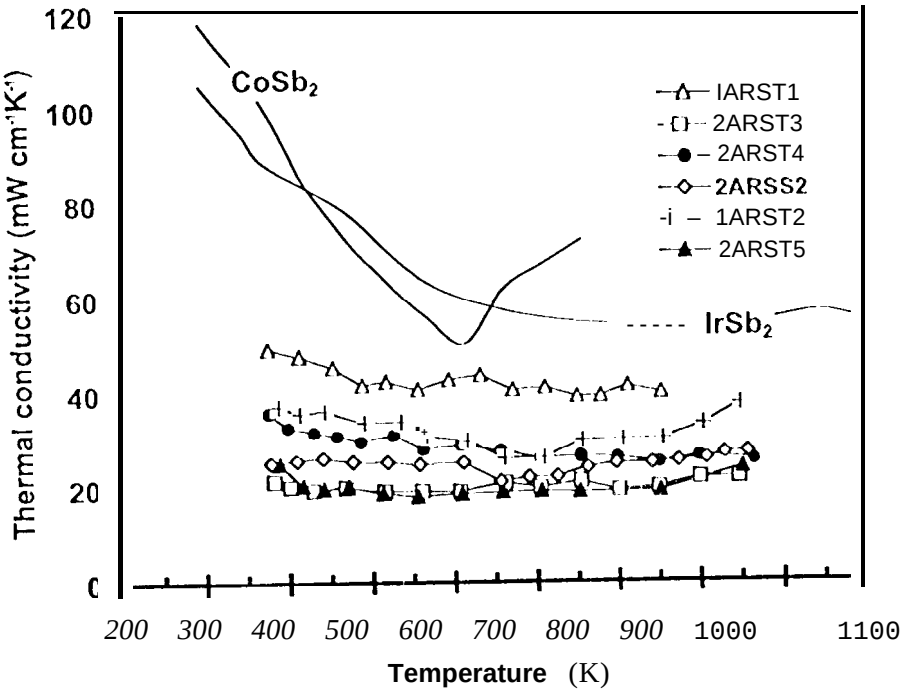
120
100
80
60
40
20
0
Thermal conductivity (mW cm⁻¹K⁻¹)
CoSb2
IrSb2
IARST1
2ARST3
2ARST4
2ARSS2
1ARST2
2ARST5
200
300
400
500
600
700
800
900
1000
1100
Temperature (K)

Fig.3

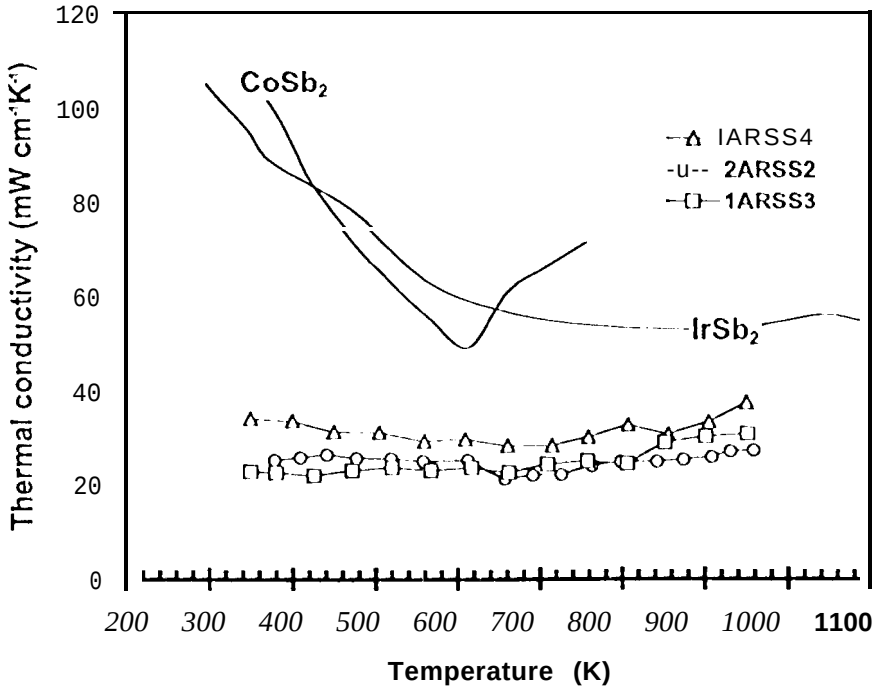
120
100
80
60
40
20
0
Thermal conductivity (mW cm⁻¹K⁻¹)
CoSb₂
IrSb₂
IARSS4
2ARSS2
1ARSS3
200
300
400
500
600
700
800
900
1000
1100
Temperature (K)

Fig 4