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A Study of Development of Thermoelectric Properties for Heat Transfer

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Abstract
The introduction discusses a short summary of the conversion, materials and uses of thermoelectric energy. The few state-of-the-art thermoelectric materials possessing a high ZT (usually >1) are Bi_2Te_3, PbTe, $CoSb_3$ and SiGe. Ternary and quaternary copper chalcogenides have recently been identified as viable candidates for thermoelectric use. These materials are gaining great interest because of the richness of the earth and are mineral compounds. Tetrahedrite (Cu12Sb4S13) is the possible thermoelectric material stated as zT~1 according to the literature. In this work, the tetrahedrite compound is synthesised to evaluate its influence on the thermoelectric characteristics of tetrahedrite using several alternative components. The pure tetrahedrite is a semi-conductor p-type. The substituents with a greater oxidising status are thus replaced at Cu and Sb to lower the carrier concentration to achieve the ideal Fermi level. Furthermore, carrier correction will also minimise the electronic component of heat conductivity. The substitutes may also produce the mass fluctuation of phonons that decreases the thermal conductivity of the grille. Due to its varied features, which provide unlimited opportunity for developing fresh techniques to devise with increased thermoelectric (TE) efficiency, the researchers are interested in the fabrication of bulk crystals in Sb2Te3 compounds.
Keywords: Thermoelectric Properties, Heat Transfer, quaternary copper, tetrahedrite compound, bulk crystals, Sb2Te3 compounds

Introduction

Rapid economic growth of the emerging and developing countries combined with non-implementation of the Kyoto protocol by many nations poses serious concerns about increase in energy consumption and pollution. The fuel deficit and huge power demand have compelled the researchers for a paradigm shift inclined to non-conventional sources. The urge for alternative energy resources leads to an important domain of research dealing with the direct retrieval of waste heat and its conversion into useful electricity. Most of the heat generated from different sources is wasted and thermoelectricity can exploit this unutilized energy. Thus, the field of thermoelectric (TE) is outstanding, as it is associated with power generation and refrigeration, which directly couples thermal and electrical phenomena. The TE device's function either as a pump to transport heat when an electric current is applied or as a generator while it is placed in a temperature gradient. They facilitate more efficient use of energy and have the option of utilizing unused heat from industrial process and domestic appliances. Environmental friendliness due to the absence of fluorinated cooling agents, compactness, quietness, reliability as well as scalability are the advantages of solid-state TE conversion and it enables the substitution of compression-based refrigeration with Peltier coolers. Automobile industries are exploring the possibilities of electrical power generation using heat from the radiator and exhaust (Bang et al. 2016). Well-established radioactive thermoelectric generators (RTG) are used in space technologies (Bathula et al. 2015).

TE cooling is used in infrared detectors, optoelectronic devices and computers (Naim et al. 2015). As per the estimation of World Health Organization, about 20% of global population is deprived of electricity and the economic progress has been inhibited due to denial of this vital resource. Obviously, solar and wind power technologies are choices to provide electricity to rural and isolated areas. Though, photovoltaic panels have become accessible at an affordable cost, poor weather conditions and lack of sunlight adversely affect their usage. Furthermore, high cost, complexity, and intermittent supply have restricted the application of wind turbines in rural remote areas for electrification. Nearly 40% of world population depends on the traditional cook

stove along with biomass or wood, as fuel. Specially designed TE generator integrated with a stove can utilize the heat from the flames as input for production of electricity, which can be used for lighting and powering some supplementary gadgets (Shaughnessy et al. 2012). Thus, the field of thermoelectric is garnered as a prospective, rapidly growing refrigeration and power generation technology, for the existence of economically viable, environmentally benign system.

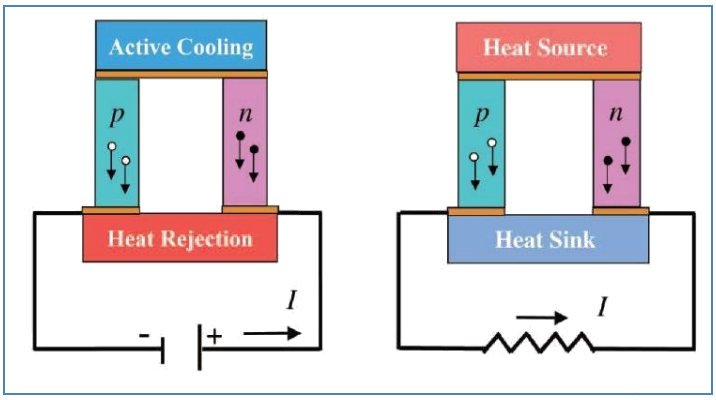
Impact Of Thermoelectricity In Science And Technology

TE materials have gained heightened attention globally on account of their unique applications. Tremendous progress has taken place in this field after Ioffe (1957) proposed the investigations of semiconductors (SC). Since 1900, there has been a resurgence of interest in producing new TE materials having properties optimized over wide range of temperature with higher performance due to low efficiency of the existing ones. Advances in modern technological era have been accomplished with an increase in the density of electronic devices, which requires adequate temperature control and efficient removal of heat for their proper operation. Industrial and military applications create increased activity in SC field, which demands highly efficient TE materials than those presently in use. As the hunt for promising bulk TE substances escalates, some of the material systems disclose high potential for attaining good The TE effects are utilized in a circuit to produce useful heating, cooling or power generation and an ideal TE circuit is shown in Fig. 1.

figure of merit possessing low thermal conductivity and electrical resistivity. Among the wide group of SC, synthesis of bulk crystals of Sb_2Te_3 and related compounds arouse the interest of researchers due to their superior attributes suitable for room temperature TE applications. They encompass a set of inherent physical and electronic properties suitable for TE refrigeration and provide great flexibility for structural design and band gap engineering. Therefore, the following section outlines a brief account of the solid-state properties, principles and parameters which contribute to establish high performance in TE compounds.

Performance Parameters Of Thermoelectric

The rapid developments in thermoelectric technology employing semiconductors suggest that, devices could be constructed with much higher efficiency than using metals. This realization has brought about considerable exploration on the properties of semiconducting materials. Thus, a critical understanding of basic TE principles and development strategies is vital for the optimization of energy conversion. The TE modules can convert directly heat into electricity by the Seebeck effect and use current to produce thermal energy from Peltier effect. According to the former, for a small temperature difference (ΔT) between the junctions, the generated electromotive force (ΔE), is found to be proportional to the difference in temperature and characteristic of the materials.



(a) (b)

Fig. 1: TE Module For (a) Refrigeration and (b) Power Generation

Under steady state conditions, heat supplied will produce a temperature difference, which results in power generation and conversely the refrigeration. The coefficients of thermal and electrical conduction of a material can be coupled to constitute a single parameter, called dimensionless figure of merit,

The term ($S^2\sigma$) is called power factor (*PF*) and an increase in the figure of merit can be achieved either by enhancing *PF* or reducing thermal conductivity. All the three factors (S, σ and κ), are functions of carrier concentration n. In metals, n is very high; but in insulators, it is very small whereas, in semiconductors, n is moderate.

Therefore, to obtain large electrical conductivity, it is necessary for a material to have a small band gap (E_g). As such, it can be concluded that, narrow band gap semiconductors are the most suitable materials for thermoelectric cooling. Fig. 2 qualitatively describes the relationship of important material parameters, S, σ, κ and n in accordance with the existing transport theory (Goncalves and Godart 2014).

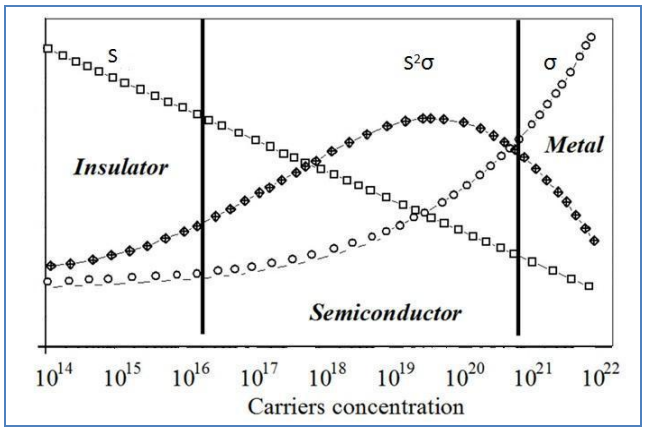


Fig.2: Dependence of *S*, *PF* and *σ*, on The Concentration of Carriers

From the plot, semiconductors are seen to have the best combination of S and σ, as evidenced by the position of the maximum for *PF*, which occurs at carrier concentrations in the range 10^{18} to 10^{21} cm^{-3}. According to this model, good thermoelectric materials are either heavily doped or degenerate semiconductors with small E_g. Attenuation of the phonon contribution to κ, without the reduction of σ is a criterion for the optimization of *ZT*. Therefore, it is inevitable to explore the novelty of potential candidates like Sb_2Te_3 based materials and adopt strategies to produce good quality crystals for achieving efficiency. In this regard, realization about the theories of nucleation and fundamentals of thermodynamics, which drives the crystallization process, empower one to do a systematic study of the diverse approaches towards crystal growth procedures.

Crystal Engineering And Design

Good quality crystals free from flaws have great demand for application in thermoelectric (TE), microelectronics, optoelectronics, and various other fields, which cannot be met by natural resources. The development of science and technology for the production of tailor-made superior semiconducting (SC) crystals constitutes the root of materials research. The building blocks are geometrically arranged orderly in the space lattice of single crystals and they possess good thermal stability with high mechanical strength. In the growth process, a controlled phase transformation takes place from a disordered fluid to solid state where the atomic constituents can be mixed perfectly by thermal convection. The important steps in the synthesis are attaining the required degree of supersaturation/supercooling, formation of defect free nuclei and control of various parameters. Appropriate growth technology is to be adopted for achieving the desired characteristics of the semiconductor crystals. The principal methods applied for the production of bulk single samples are classified mainly into three categories based on the transition of phases as described below.

1 Growth From Melt

Single crystals are directly produced from melt of a material, when it does not dissociate largely during melting and has no destructive phase transformation leading to polycrystallinity. This is one of the most attractive methods for industrial purposes where, fusion and re-solidification of substance take place to form large size bulk crystals.

The crystallization of a polycrystalline ingot into single crystal happens with the elimination of grain boundaries, by adopting proper thermodynamical conditions. To take control over the orientation, thermal anisotropy and growth rate, often seed crystals are employed. The most prominent techniques are Czochralski, Bridgman-Stockbarger, flame fusion and float zone methods (Svechnikova et al. 2000). In flame fusion, powders of the compound are passed through an oxy-hydrogen flame, which is vertically aligned. It is usually desirable to prepare alloys and compound semiconductors for optoelectronic applications by Czochralski technique. Large crystals of metal alloys, organic and inorganic compounds, etc. are obtained from Bridgman method (Scheel et al.2011).

2 Growth From Solution

Solution growth is well suited to those materials which suffer from decomposition in the melt or solid at high temperatures and undergo structural transformations while cooling. In this process, a saturated solution of the material is prepared using a suitable solvent and crystallization takes place under critical supersaturation. Solution growth techniques offer an advantage of producing large volumes with the possibility of comfortable scaling-up options. Depending on the requirement, low temperature aqueous solutions or high temperature flux routes are chosen. One of the simplest and least expensive methods for production of pharmaceutical and optical crystals is low temperature solution growth, whereas flux methods are employed for the incongruently melting substances. Nonlinear optical crystals and solid-state laser materials are generally obtained from high temperature preparations.

3 Growth From Vapor

The growth of single crystals from vapor phase is superior, owing to the novel properties acquired and hence, this technique is used for the preparation of bulk, thin film and nanocrystals. The binding energy of the atoms should be high for successful deposition without significant dissociation of the vaporized material. The low growth rate improves the crystalline quality and reduces the density of dislocations and point defects, though the yield is less compared to that of melt methods. Good quality samples can be obtained from vapor deposition, if suitable preparative environment is provided to attain supersaturation. Though, several technical approaches are available in this category, they can be consolidated mainly into physical vapor deposition (PVD) and chemical vapor deposition (CVD). In PVD, powders of the substance are evaporated at the hot end and then crystallized at the cooler region of the ampoule. Sublimation is a simple PVD process in which solid source is placed in a horizontal ampoule such that, it sublimes and transports to the deposition zone. Under circumstances where the vapor pressure of the material is less for obtaining considerable evaporation rate, the addition of a chemical agent such as chlorine, bromine, iodine, etc. accelerates the dissociation of source substance and subsequently, transportation of the vapors to growth region takes place. Though, the yield in CVD is quite satisfactory, the impurity contamination affects the quality of crystals. Physical vapor deposition is a versatile process which involves the evaporation and subsequent deposition of materials on a substrate. In PVD, the source material can be in solid or molten liquid phase and on heating, they are transformed into vapors either by sublimation or vaporization. Among all growth processes, PVD is the most efficient one to yield crystals of well-defined planes, devoid of extraneous phases and is generally employed for materials having high vapor pressure. High pure crystals with uniformity of composition and structural perfection can be grown from vapor phase (Faktor and Garret 1974). Preceded by nucleation, crystallization occurs either spontaneously or supported by adsorption

of atoms. If nucleation sets rapidly, multiple sites give rise to polycrystals.

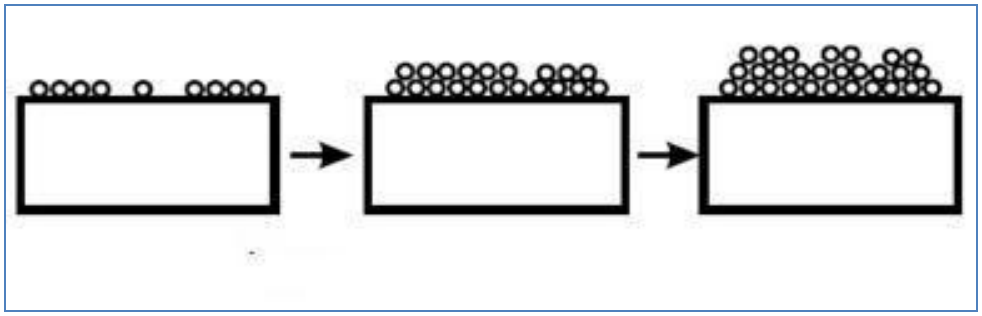


Fig. 3: Schematic Diagram of Frank-Van Der Merwe Mode of Growth

As per Frank-Van der Merwe approach, the good compatibility between the adatoms and substrate causes tangential deposition of layers, hindering clusters (Sheel and Fukuda 2003). Stacking of this process continues for further deposition and growth proceeds in a layered structure as depicted in Fig. 3.

Literature Survey On Substitution Studies Of Tetrahedrite

Cu12Sb4S13 is a potential thermoelectric material that has achieved a practical limit of zT~1. This is due to reasonably high PF (~1.6 W/m-K^2) and low (~1 W/m-K). From the various reports it was observed that the substitution and/or alloying lead to achieve high zT. The pristine tetrahedrite exhibits metallic behaviour. Most of the reports demonstrate the substitution of transitional elements at Cu site. The general formula of the tetrahedrite is given by Cu10Tr2(Sb, As, Bi)4S13 (Tr = Zn, Fe, Mn, Co, Ni, Cd).

The pristine Cu12Sb4S13 shows electrical resistivity of around 0.01 m-m and Seebeck coefficient ~120 at 600 K. The temperature dependant behaviour of these properties exhibits a metallic like behaviour. Substitution of monovalent $Cu^{+1}$with higher oxidation state transition elements (Zn^{+2}, Fe^{+3}, Ni^{+2}, Mn^{+2}, Co^{+2}, Ni^{+2}) effectively bring down the carrier concentration. This help in attaining the optimum value of thermoelectric properties to gain high zT and is reported to be ~1 in most of the cases. The enhancement in zT is observed due to increased PF in some cases and the reduced thermal conductivity is observed in all the cases. The suppressed carrier concentration significantly decreases the electronic part of thermal conductivity. Firstly, Suekuni et al. have reported the low temperature thermoelectric properties of Mn, Fe, Co, Zn, Ni substituted tetrahedrite up to room temperature (RT). The value of was reported to be ~1 W/m-k at RT. The L of tetrahedrite is as low as 0.4 W/m-K which approaches the amorphous limit. He has obtained the maximum zT of 0.13 for pure tetrahedrite and 0.15 for Cu10Ni2Sb4S13 at 340 K. Lu et al has reported thermoelectric properties of synthetic as well as the natural mineral of tetrahedrite. He has demonstrated the direct utilization of minerals for thermoelectric application by simple processing. The samples are processed by mixing synthetic sample with natural mineral at equal ratio to explore its thermoelectric properties. The synthesis process was by ball milling the mixture to obtain desired stoichiometry. The zT values ~1 for the Zn substitution is obtained due to significant reduction. This is a result of compensating holes by substitution of higher oxidation state Zn^{+2} on sites which tend to decrease the electronic part of thermal conductivity. However, there was no improvement in the power factor.

Substitution of Fe on the Cu site tends to increase the electrical resistivity due to carrier compensation similar to Zn. The magnitude of the change is about twice when compared to Zn substitution. This was attributed to the higher oxidation of Fe, which is +3. The zT value of ~0.8 is reported for Fe substituted sample. Suekuni et al., have reported that for Ni substituted tetrahedrite maximum zT of 0.7 is obtained at 665 K. The enhancement in zT was attributed to reduced thermal conductivity which arise due to hole compensation. In addition, Suekuni has explored the presence of low energy vibrational Cu (2) atom results in the scattering of phonons. This was also

explained by Raju et al. where the large atomic displacement of Cu (2) atom was reported to create dynamic and static off-plane disorder. He has obtained high zT for the parent compound which is more than the reported value. There was no improvement observed in zT value for Mn substituted tetrahedrite. However, the reduction in thermal conductivity is reported which is similar to the other reports for different substituents. It was clearly witnessed that the sample is showing metallic nature in pure form and also at low substitution concentration. Raju et al. have also obtained the maximum zT~1 for Co substituted tetrahedrite. It was attributed to the combined improvement in PF and reduced. The enhanced PF was attributed to the optimized carrier concentration and useful modification in the band structure. He has also reported the mechanical properties which are significant during the device fabrication to have an idea of mechanical stability. Few reports discussed the substitution of heavy element such as Te, Sn and Ge at both antimony and copper site. The zT of 0.92 at 723 K is reported for optimum substitution value of Te at Sb site. The total thermal conductivity was reduced as a result of reduced electronic part and in addition due to substitution effect of Te. Further, the different substitution method was reported with enhanced figure of merit. In this stage the thermoelectric properties are optimized by co-substitution Ni and Zn at copper site zT above unity is achieved for the co-substituted tetrahedrite. This was attributed to the tuning of Fermi energy to obtain high PF and also reduction of lattice and electronic thermal conductivities. Adding Ni into the lattice facilitated the additional valence band which arises from spin-split Ni states. In addition to this, co-substitution helped in achieving the theoretically minimal thermal conductivity.

Selection Of Substituents To Improve Thermoelectric Properties Of Tetrahedrite

In the present work the compound is prepared with different substitution elements namely Bi, Cd, and Cr to study their effect on thermoelectric properties of tetrahedrite. The pure tetrahedrite is a p-type degenerate semiconducting nature. Hence the substituents with higher oxidation state are substituted in Cu and Sb site to reduce the carrier concentration in order to obtain the optimum Fermi level. In addition, the compensation of carriers will also reduce the electronic part of thermal conductivity. The substituents can also create the mass fluctuation scattering of phonons which reduces the lattice thermal conductivity.

The thermoelectric properties of Bi substituted tetrahedrite at Sb site are discussed in the chapter 3. Bi bearing tetrahedrite called Annivite is available as a natural mineral which can be used for thermoelectric application with simple process steps to obtain desired stoichiometry. In the Rednizy locality of south western Poland, Bi-rich Ag-bearing tetrahedrite minerals were discovered with a Bi content reaching up to 15.86 wt.%, or 1.36 atoms per formula unit (apfu). These varieties were found in the form of inclusions and intergrowths with other sulphides. Bi-rich tetrahedrite series containing 18.23 wt. % Bi or 1.60 atoms per formula unit have also been found in Russia. These occurrences suggest that there is a wide range of Bi for Sb substitution in naturally occurring tetrahedrite minerals. It therefore follows that we need to explore the thermoelectric properties of Bi-containing tetrahedrites, as they are readily available as natural minerals with a wide range of compositions. Bi belongs to 5A group in the periodic table which is same as Sb. This indicates the ease of substitution of Bi at the Sb site. When it comes to thermoelectric properties, the substitution of heavier Bi can produce mass fluctuation scattering and reduce lattice thermal conductivity.

The Further studies have been carried out by substituting transition metal elements in the Cu site. Johnson et al. have reported the possibility of Cd substitution in Cu12Sb4S13. He has given the general chemical formula of tetrahedrite (M1)6(M2)6(X)4(Z)13 where M1=Cu, Ag; M2=Cu, Zn, Fe, Mn, Cd, Hg; X= Sb, As, Bi, Te and Z= S, Se. Among all transition elements at M2 site, the Cd is heavier metal element. Hence substitution of Cd in tetrahedrite is more advantageous to improve its thermoelectric properties. In chapter 4 the effect of Cd substitution at Cu^{+1} site is discussed. Cd has higher oxidation state (+2) when compared to Cu (+1) which help in the reduction of carrier to

optimum value for the improvement of power factor. In addition, the inclusion of heavier atom can result in reducing lattice thermal conductivity. The transition element Cd is substituted at Cu site due to its higher oxidation state. The objective is to optimize the Fermi level of the p-type degenerate tetrahedrite compound to obtain high zT. The band structure studies of pure Cu12Sb4S13 shows that the Cu and S atoms have main contribution towards the VB. The substitution of Cd^{+2} at Cu^{+1} site fills the holes in the valence band and push the Fermi level into the band gap. The substitution is made with different Cd concentration in order to obtain the optimum Fermi level. Cd strictly follows the +2-oxidation state due its electronic configuration of [Kr] $4d^{10}$ $5s^2$ with filled orbitals. Hence it can effectively fill the states above the Fermi level in the VB of tetrahedrite without creating an additional impurity level. The reduction of carrier concentration enhances the Seebeck coefficient with optimum values of electrical resistivity to improve the power factor. In addition, this also supress the carrier part of thermal conductivity. The substitution of heavy Cd at copper site creates mass fluctuation in the system which scatters the phonons and reduces lattice thermal conductivity. These aspects result in the enhancement of zT.

In the next work the thermoelectric properties of Cr substituted tetrahedrite is studied. Cr has multiple oxidation states (+2, +3) however it is more stable at +3. Substitution of higher oxidation state element is more effective to compensate holes and optimize the carrier concentration of p-type material. The substitution limit of Cr can be high due to its ionic radius (Cr^{+3}: 0.615 Å) which is comparable to Cu (Cu^{+1}: 0.6 Å, Cu^{+2}: 0.57 Å). The electronic configuration of Cr is [Ar] 3d5 4s1, indicating partially filled electronic states (with 6 electrons) in the outer shell. Due to partially filled states, Cr can act as a magnetic impurity in the tetrahedrite system. The substitution of similar magnetic impurities such as Ni and Co has improved the power factor of tetrahedrite due to the favourable changes in the band structure.

Conclusion

In this study the thermoelectric characteristics of Cu12Sb4S13 were examined by the replacement of Bi at Sb and Cd, Cr at Cu site. The chemicals were synthesised by a process of melt synthesis, which were afterwards rinsed and pressed hot to generate dense pellets for future study. The investigation of the crystal structure was performed by XRD and Rietveld refinement. The micrographs were taken using SEM and EDS/WDS was used to carry out compositional analyses. These findings reveal that the main phase with evenly distributed minor secondary phases such as Cu3SbS4, CuSbS2 and Cu3SbS3 in all the Cu12Sb4S13 samples is. In the temperature range between 300 K and 700 K, thermoelectrical parameters such as Seebeck coefficient, electrical resistivity, and thermal conductivity were measured. These characteristics have been used to establish the thermoelectric value of value. As described in chapter 3, Bi was replaced on Sb sites with a different Cu12Sb4-xBox S13 (x=0, 0.2, 0.4, 0.6, 0.8) concentration. The result of XRD is the main stage with a little quantity of secondary phase of the Cu12Sb4-xBox S13. Rietveld refinement showed that the grid parameter is raised practically linearly with the Bi content rise. This indicates the unit cell's extension to Bi substitution. Substitution of Bi at low concentration is beneficial.

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