

Anisotropy in thermoelectric properties of textured ZnSb nanomaterial by high Zn occupancy (132) crystallographic planes

Lalit Deora

Defence Laboratory Jodhpur

Jasa Ram

`jasa.ram.dl@gov.in`

Defence Laboratory Jodhpur

Research Article

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Abstract

The present paper demonstrates preparation of cost effective ZnSb thermoelectric nanomaterial by a cost and energy efficient bottom-up physical synthesis process and anisotropy in its thermoelectric properties for the first time. The formation of single phase ZnSb nanomaterial and texturing has been established by powder X-ray diffraction and high-resolution transmission electron microscopy. The Seebeck coefficient has been found to be higher in the preferential orientation of higher Zn atom occupancy (132) crystallographic planes, which is aligned in perpendicular to pressing direction during spark plasma sintering (SPS). Whereas, electrical conductivity is observed higher in the direction of pressing during SPS, which is preferential orientation of (211) planes. Deficiencies of Zn may be causing increased carrier scattering along higher Zn occupancy (132) crystallographic planes to produce higher Seebeck coefficient and lower electrical conductivity. The SPS consolidation may be enabling texturing in this orthorhombic crystal system due to anisotropy of electrical conductivity. The study may enable decoupling of these two correlated parameters by site specific substitutions within (132) crystallographic plane or outside this plane of ZnSb for obtaining enhanced thermoelectric efficiency.

1. Introduction

Thermoelectric materials offer potential for conversion of thermal energy into electrical energy and vice-versa. This makes these materials attractive for solid-state power generation as well as Peltier cooling. However, these materials have not become part of daily life usages due to three major limitations. The cost of ingredient materials, cost of processing to produce thermoelectric materials and conversion efficiency of thermoelectric material are major challenges for translating them to common applications in real life [1]. In this endeavour, zinc-antimonide has been investigated since last one decade [2–23]. This materials system is attractive due to cost effective ingredients and their earth abundance [3]. Research in zinc-antimonide class of materials started with stoichiometric ZnSb Phase. This phase has relatively higher Seebeck coefficient (α), but lower electrical conductivity (σ). Eventually, research interest shifted to Zn_4Sb_3 phase. This Phase has inherently higher σ and lower thermal conductivity (κ) in comparison to ZnSb Phase, but it is highly unstable to have any practical usages [1, 22–23].

Instead of efforts on improvement in stability of Zn_4Sb_3 phase [1], different approaches of thermoelectric performance improvement in ZnSb phase are presently being pursued by different experimental processing and theoretical techniques [3–21]. Thermoelectric material efficiency is governed by dimensionless figure of merit (zT) [1], defined as $zT = (\alpha^2 \sigma / \kappa) T$. Where, T is the absolute temperature. The ZnSb phase has been experimentally investigated for zT enhancement by substitutions, doping, composite formation and nano-structuring [4–17]. The Zn vacancies created by deviation from stoichiometric composition has been used for zT enhancement in increasing the carrier concentration [4]. The same approach has also been used by substitution of nickel [5], and hetero-valent bismuth in ZnSb for zT increase [6]. The doping of Ag [7], Cu-Ag [8], and Sn [9] in ZnSb has also been reported to enhance zT by carrier concentration increase. In addition, reduction of lattice thermal conductivity has also been reported by partial substitution of Zn by Cd [10], and Cr [11]. Composite

formation of ZnSb with 2D nanosheets of MoS₂ [12], with Zn₄Sb₃ [13], with Ag₃Sb [7], and with SnTe [14] have also been studied for zT enhancement in this cost effective compound. Further, this compound has also been studied in nanostructured form produced by melt-spinning [15], mechanical alloying [16], hydrogen decrepitation [6] and solvo-thermal [2] processes, and thin film deposited by magnetron co-sputtering [17]. These literature reports show potential of improvement in thermoelectric properties in ZnSb. These studies showed requirement of atomic level understanding of this material systems to scientifically plan zT enhancement strategies. Study of anisotropy in thermoelectric properties has potential to offer insight into atomic level influences, but it has not been studied so far to the best of author's knowledge. In addition, economic viability of this cost-effective ingredient based thermoelectric material requires cost and energy efficient process to produce this thermoelectric material.

In the present paper, we report a cost-effective and scalable bottom-up physical process to synthesis ZnSb nanomaterial powder, similar to that reported for Bi_{0.5}Sb_{1.5}Te₃ nanomaterial synthesis [24]. Consolidate as synthesized nanomaterial into cylindrical pellets using spark plasma sintering (SPS) and study anisotropy in transport properties with correlation to preferential textured crystallographic orientations. A comparison of thermoelectric properties with those reported in literature using melt-spinning [15], mechanical alloying [16] and hydrogen decrepitation [6] processes for stoichiometric ZnSb has been done to understand orientation effects in detail.

2. Methods

In this bottom-up physical synthesis process, Zn (99.99% Alfa Aesar) and Sb (99.5% Alfa Aesar) metal ingredients have been weighed in stoichiometric amount (1:1 molar ratio) and filled in a quartz ampoule. The filled ampoule is evacuated using a diffusion pump and purged multiple time with argon gas and sealed. Then, this ampoule has been heated in rocking tube furnace. The process is shown by a flowchart in figure s1. The melting temperature of 923 K has been used in the present work to facilitate spontaneous reaction for nano-scale grain formation. The ampoule is continuously rocked while heating in this process. After heating of 2 hours, the ampoule has been withdrawn from furnace to quench the material in air at ambient temperature (300 K). The process is schematically shown in figure s1. The SPS consolidation of synthesized material has been carried at optimized 100 MPa pressure and 623 K temperature in a graphite die to obtain cylindrical pellet samples of ZnSb.

The phase formation has been studied using powder X-Ray diffraction (PXRD) technique with Cu K_α source line for powdered form of SPS consolidated pellet as well as pellet observed from both parallel and perpendicular to pressing direction in SPS. Surface morphology and elemental ratio of consolidated pellets have been studied using field emission scanning electron microscopy (FE-SEM) with energy dispersive spectroscopy (EDS) on pellets in parallel and perpendicular to the pressing directions. A piece of pellets has been crushed into fine powder using pestle-mortar to study nanoscale feature of material using high-resolution transmission electron microscopy (HR-TEM) with selected area electron diffraction (SAED) by taking powder dispersed in isopropyl alcohol on a copper grid.

For thermoelectric measurements, cylindrical pellets have been cut into the parallelogram shape and dimensions of about 10 mm × 4 mm × 2 mm and about 10 mm × 10 mm × 2 mm from SPS consolidated pellet of about 12.7 mm (diameter) × 11 mm (thickness) pellet. Round shape pellet of about 12.7 mm (diameter) × 2 mm (thickness) have been directly used in the measurements. Pairs of pellets oriented in parallel and perpendicular to the pressing direction were prepared for the measurements. The Seebeck coefficient and electrical conductivity were simultaneously measured over a temperature range from ambient to 523 K temperatures. Thermal diffusivity was measured using a laser flash apparatus, while specific heat (C_p) was determined using a differential scanning calorimeter (DSC). Reference standard of sapphire has been used in C_p measurements. The solid density of the pellets has been measured using Archimedes' principle with a setup based on a semi-microelectronic balance, was found to be 6.30 g/cm³. These three parameters have been used to calculate thermal conductivity (κ), where $\kappa = DC_p \rho$.

3. Results and discussion

The observed PXRD profile of powder of SPS consolidated pellet of ZnSb has been compared in Fig. 1(a) with calculated profile for orthorhombic crystal system with space group 61 (pbca) in Rietveld refinement using Fullprof software and Bragg positions. The diffraction peaks are sharp, broad and does not show any impurity phase. Good fit (chi square 1.31) has been obtained in Rietveld refinement of powder X-ray diffraction (PXRD) data of powder of ZnSb pellet. This refinement shows that cell parameter values are: $a = 6.2027$, $b = 7.7421$, $c = 8.0963$, and $\alpha = \beta = \gamma = 90.000$. The analysis shows that theoretical density of the material is 6.452 g/cc. Thus, the polycrystalline nanomaterial of ZnSb has reached about 97.6% of theoretically calculated density. The broad peaks in PXRD patterns indicate nanocrystalline origin of the diffraction patterns. The average crystallite size calculated using Scherrer equation for major peaks is 85 nm.

PXRD measurements have also been done on pellets from parallel and perpendicular to pressing directions in SPS to understand preferential orientation, which are shown in Fig. 1(b) and 1(c) respectively. The baseline of both these patterns and most of the peaks, except corresponding to (211) and (132) planes, are mostly similar without much difference in intensity and shape. The intensity corresponding to (132) planes observed from parallel to pressing direction during SPS is higher in comparison to that observed from perpendicular to pressing direction (difference of more than 750 count). At the same time, the PXRD pattern observed from perpendicular to pressing direction has much higher intensity (difference of more than 1k counts) corresponding to (211) planes. Thus, the (132) and (211) planes are preferentially oriented in perpendicular and parallel to the pressing directions respectively. The pictorial representations of atomic positions in these two planes in the inset the Figs. 1(b-c) reveal that (132) plane is dominated by Zn atoms. This characteristic of (132) crystallographic planes of ZnSb to orient in perpendicular to pressing as well as current flow direction in SPS consolidation has been revealed for the first time in literature reports.

FE-SEM images as seen from the directions of pressing and perpendicular to it are shown in figure s2(a-b) and s2(d-e) respectively. Good densification is seen in all the images. Inferences of texturing are also

visible from comparison of these images, as seen from the two perpendicular directions. The quantitative analysis of the EDS spectra as shown in figure s2(c) and s2(f) provides atomic ratios of Zn and Sb as 0.535:0.465 and 0.544:0.456 respectively. The estimated atomic ratios are within 7% of the planned ones. Maintenance of stoichiometry in the process is verified by the EDS results. The analysis also shows that the sample is not Zn deficient and thereafter, expected to be relatively resistive [4].

HR-TEM of one typical grain of the ZnSb is shown in Fig. 2(a) and its inset shows SAED pattern. The ring patterns in the SAED image shows that a grain of few 100 nm contains many randomly oriented crystallites in it. More lower resolution HR-TEM images of some grains are shown in figure s3. Figures 2(b-c) shows HR-TEM images upon increasing the resolution. Atomic level resolution in Fig. 2(d) revealed few nm size crystallites, which are randomly oriented. The crystallite sizes resemble to those reported using chemical process [2]. The interatomic spacing measurement in two typical crystallites within this image are amplified in Fig. 2(e-f) for illustration purposes. This amplified image also shows good crystalline quality of the material synthesized by this process.

Thermoelectric measurements revealed large anisotropy in Seebeck coefficient and electrical conductivity of the ZnSb pellets, as shown in Figs. 3(a) and 3(b) respectively. This may be first report on anisotropy in ZnSb. The Seebeck coefficient has been found to have higher value along perpendicular to pressing direction in SPS, which is preferential orientation of (132) planes. Whereas electrical conductivity is higher along the direction of pressing, which is preferential orientation of (211) crystallographic planes. Further, a comparison of these parameters with respect to those reported in literature for ZnSb nanomaterials synthesized by different processes [6, 15–16] has been made in these figures. Two repeated measurements of the present work are also shown in figure s4. The samples of the present study are relatively resistive [6, 16], but shows higher Seebeck coefficient. This outcome may be caused by lack of Zn deficiency [4], as revealed by EDS measurement. The carrier concentration in the present study may be relatively lower [4]. The lower electrical conductivity and increased Seebeck coefficient may be associated with lower carrier concentration.

Thermoelectric properties in both parallel and perpendicular to the pressing direction in spark plasma sintering consolidation of ZnSb synthesized by bottom-up physical process and their comparison with those reported in literature for ZnSb nanomaterials produced using hydrogen decrepitation [5], melt-spinning [15], and mechanical alloying [16] processes. **(a)** Seebeck coefficient shows large anisotropy with higher values along preferential orientation of (132) crystallographic planes, and **(b)** electrical conductivity also shows large anisotropy with higher value along the preferential orientation of (211) crystallographic planes. Large electrical conductivity increase in case of melt-spinning process may be related to increased Zn deficiency [4]. **(c)** Power-factor values of about 1 mW/m.K^2 has been obtained in the direction of pressing and it shows large anisotropy. **(d)** Thermal conductivity does not show anisotropy despite crystallographic texturing. **(e)** Figure of merit values are equivalent to those reported by other processes of nanomaterial synthesis and proves potential of this cost and energy efficient process in producing quality functional properties.

It is also noted that electrical conductivity is much higher in melt-spinning process [15], in comparison to other reports [6, 16] and the present study. EDS study in this paper revealed that the samples of that study were relatively Zn deficient. This Zn deficient caused increased carrier concentration [4], and thereby, increased electrical conductivity. In the present work, Zn deficiencies may be causing increased carrier scattering along higher Zn occupancy (132) crystallographic planes. This results in increase of Seebeck coefficient and decrease of electrical conductivity along this orientation. Further, ZnSb material system is theoretically reported to be n-type [19], whereas polycrystalline form of this compound is p-type [3–17]. This indicate that antisite defects may be causing change in type of carriers in electrical transport properties in polycrystalline ZnSb [24]. Increased electrical conductivity upon nano-scale crystallite size formation also supports this understanding [6, 15–16]. In addition to Zn vacancy point defects, anti-site defects may also be causing increased carrier scattering in (132) crystallographic planes to produce anisotropy in electrical transport properties.

Thus, the deficiency of Zn is capable of causing increase in electrical conductivity as well as increase in Seebeck coefficient, but in two perpendicular directions. Therefore, it is essential to make directional measurement for identification of optimum power-factor direction in ZnSb synthesized by powder processing techniques. In the present work, higher electrical conductivity in the direction of pressing results into higher power-factor in this direction, as shown in Fig. 3(c), reaching value of more than 1.0 mW/m.K^2 throughout the temperature range of measurement. This power-factor value is higher in comparison to those reported by hydrogen decrepitation as well as mechanical alloying processes [6, 16]. A closer resemblance of power-factor values of the present study in perpendicular to pressing direction with these reports gives an indication that electrical measurements may have been made in this direction in these two studies [6, 16], without worrying about maintaining the directionality of measurements.

The Seebeck coefficient and electrical conductivity have also been measured while heating as well as cooling, as shown in Fig. 4. It is observed that repeatable profile is obtained above 425 K temperature. Below this temperature, electrical conductivity increases and Seebeck coefficient decreases during the cooling cycle. This hysteresis may be caused by decreased carrier scattering in cooling cycle. The power-factor plotted in inset of this figure does not become repeatable in the cycles, which further confirms the role of carrier scattering mechanism in causing hysteresis in electrical transport during heating-cooling cycle.

Thermal diffusivity plotted in figure s5(a) and therefore, thermal conductivity plotted in Fig. 3(e) does not show anisotropy in ZnSb. This indicates that anisotropy in Seebeck coefficient and electrical conductivity may not be attributed to any band asymmetry and may be essentially related to carrier dynamics. Thus, directional measurements may be not be required for this orthorhombic crystal system like those required in trigonal (rhombohedral) crystal systems [24]. However, it is essential to identify direction of higher power-factor, which is highly anisotropic. The values of thermal conductivity are relatively higher than those reported by hydrogen decrepitation and mechanical alloying processes [6, 16], but low than

those reported by melt-spinning processes [15]. In order to understand these differences, Lorentz number (L) has been estimated using simplified Eq. (1) in the units of $10^{-8} \text{ W}\Omega/\text{K}^2$ from literature [25].

$$L = 1.5 + \exp \left[-\frac{|\alpha|}{116} \right]$$

1

Where, α is in the units of $\mu\text{V}/\text{K}$. The carrier contribution to thermal conductivity (κ_e) has been calculated as $\kappa_e = L\sigma T$. Lattice plus bipolar contribution (κ_{l+b}) to thermal conductivity has been calculated by subtracting calculated values of κ_e from measured total thermal conductivity (κ). The values of κ_e and κ_{l+b} are plotted in Fig. 5 for the present work as well as for the comparative process studies [6, 15–16]. This figure shows that lattice thermal conductivity essentially determines the total thermal conductivity in all these studies, which corroborate with detailed investigation of thermal transport in ZnSb [20]. The electronic contribution to thermal conductivity is much higher in melt-spinning process in comparison to all other processes [6, 15–16], possibly due to high carrier concentration associated with higher Zn vacancies [4]. It is also noted that electronic thermal conductivity increases in the present study as well as in mechanical alloying process as temperature is increased, which indicates thermal broadening and signifies presence of energy dependent scattering of charge carriers. This reaffirm that increase in Seebeck coefficient should be associated with increased carrier scattering in the present study. The lattice contribution to thermal conductivity is relatively higher in the present study in comparison to those reported by mechanical alloying [16], and hydrogen decrepitation processes [6], which may be within the error associated with measurement of specific heat. The higher values of lattice thermal conductivity in melt-spinning process [15] may be associated with amount of contribution of anharmonic behaviour of the Zn atoms having higher vacancies of Zn [20].

The values of the figure of merit in Fig. 3(e) shows that it is higher in parallel to pressing direction and it is equivalent to those reported by other processes for stoichiometric compound. This equivalency in values of thermoelectric figure of merit has been obtained in the present study by enhanced power-factor despite relatively higher thermal conductivity.

4. Conclusions

Bottom-up physical process has been successful in producing functional single phase ZnSb nanomaterials. Few nm sizes of crystallites have been revealed by HR-TEM technique. Such smaller sizes of nano-crystallite of ZnSb have been recently reported using chemical process [2]. The present process does not allow impurity addition, unlike chemical process. Thus, cost factor of processing ZnSb nanomaterial has been successfully addressed.

Directional PXRD and thermoelectric measurements have been made for the first time in ZnSb to reveal crystallographic texturing and high anisotropy in electrical transport properties. Further, the analysis

shows that increased carrier scattering by Zn deficiencies along (132) crystallographic planes cause higher Seebeck coefficient and lower electrical conductivity, due to higher occupancy of Zn atoms in it. This atomic level revelation is very useful in thermoelectric efficiency enhancement strategies. The carrier dynamics substantially influences values of Seebeck coefficient and electrical conductivity along (132) crystallographic planes. Therefore, it is essential to make directional electrical transport measurements to select higher power-factor value direction. Increased Zn deficiencies [1]. The doping of Ag [7], Ag-Cu [8], and Sn [9] at Zn sites may cause electrical conductivity enhancement along the direction of pressing, but increase in Seebeck coefficient may also be higher in perpendicular pressing direction in this material systems. Similarly, substitutions of Cd [10], and Cr [11] at Zn sites may cause better thermal conductivity reduction in perpendicular to the pressing direction. Thus, the revelation of anisotropy in electrical transport properties and its strong relevance to highly Zn atom populated (132) crystallographic planes may enable well-thought-out decision of site-specific substitutions for enhancement of constituent thermoelectric properties. The flexibility and novelty of the present process also allow incorporation of suitable doping and composite formation. Functional quality of the process has been shown by equivalent thermoelectric performance for stoichiometric compound.

Declarations

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Declaration of competing interest

Authors declare that there is no competing interest of work presented in this article.

Data availability statement

Authors declare that all the data are available with corresponding author upon a written request.

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Figures

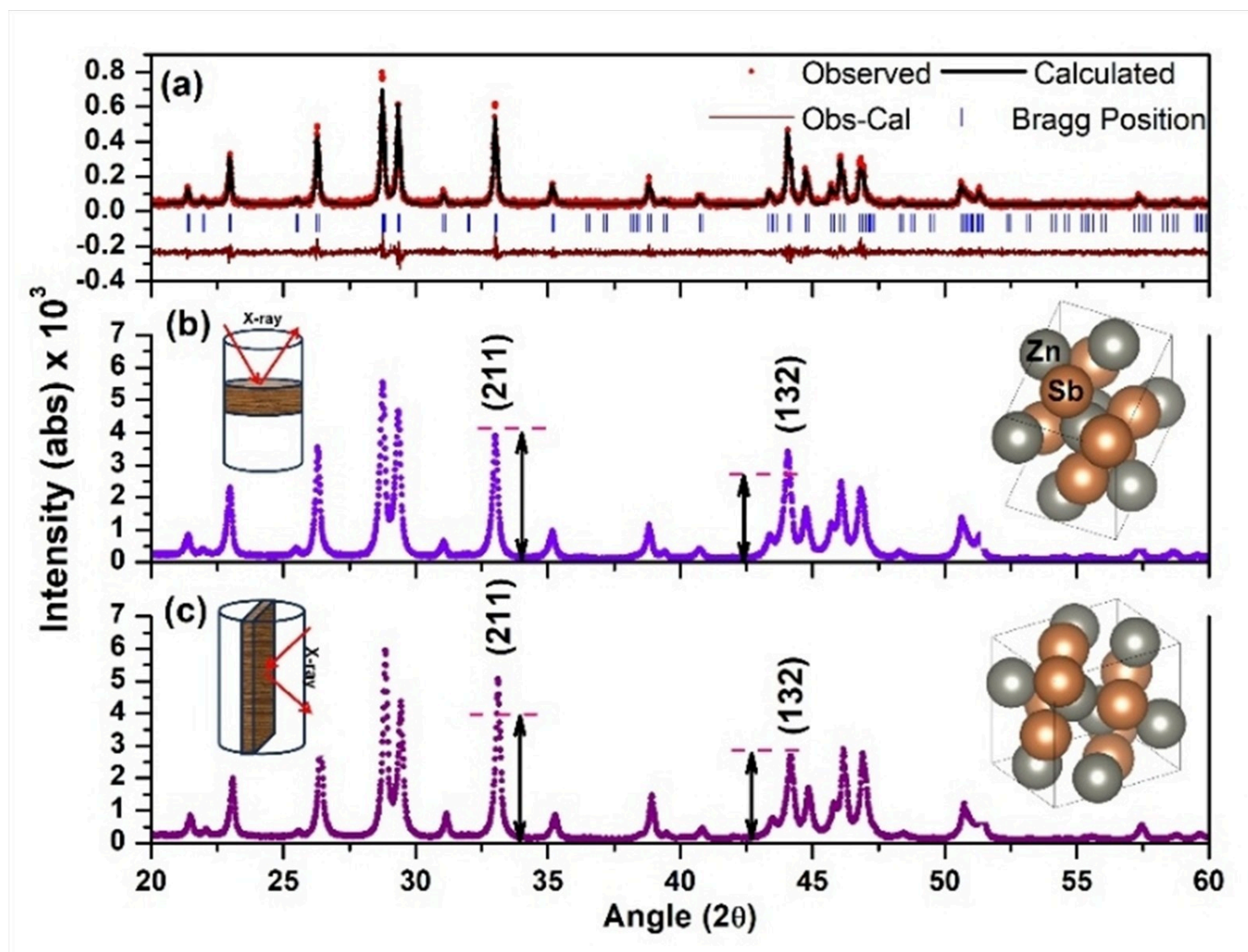


Figure 1

PXRD patterns of ZnSb for (a) powdered pellet as observed, calculated by Rietveld refinement, Bragg positions for space group 61 (pbca) and difference of observed and calculated values, and pellets

observed from **(b)** perpendicular and **(c)** parallel to pressing direction are shown to make out the differences in relative intensity to show texturing. Inset of (b-c) shows atomic arrangement in (132) and (211) crystallographic planes. Intensity corresponding to (132) crystallographic planes is relatively higher in observation from the direction of pressing and it is highly populated by Zn atoms. The directional PXRD characterization revealed texturing in this orthorhombic crystal system ZnSb nanomaterial.

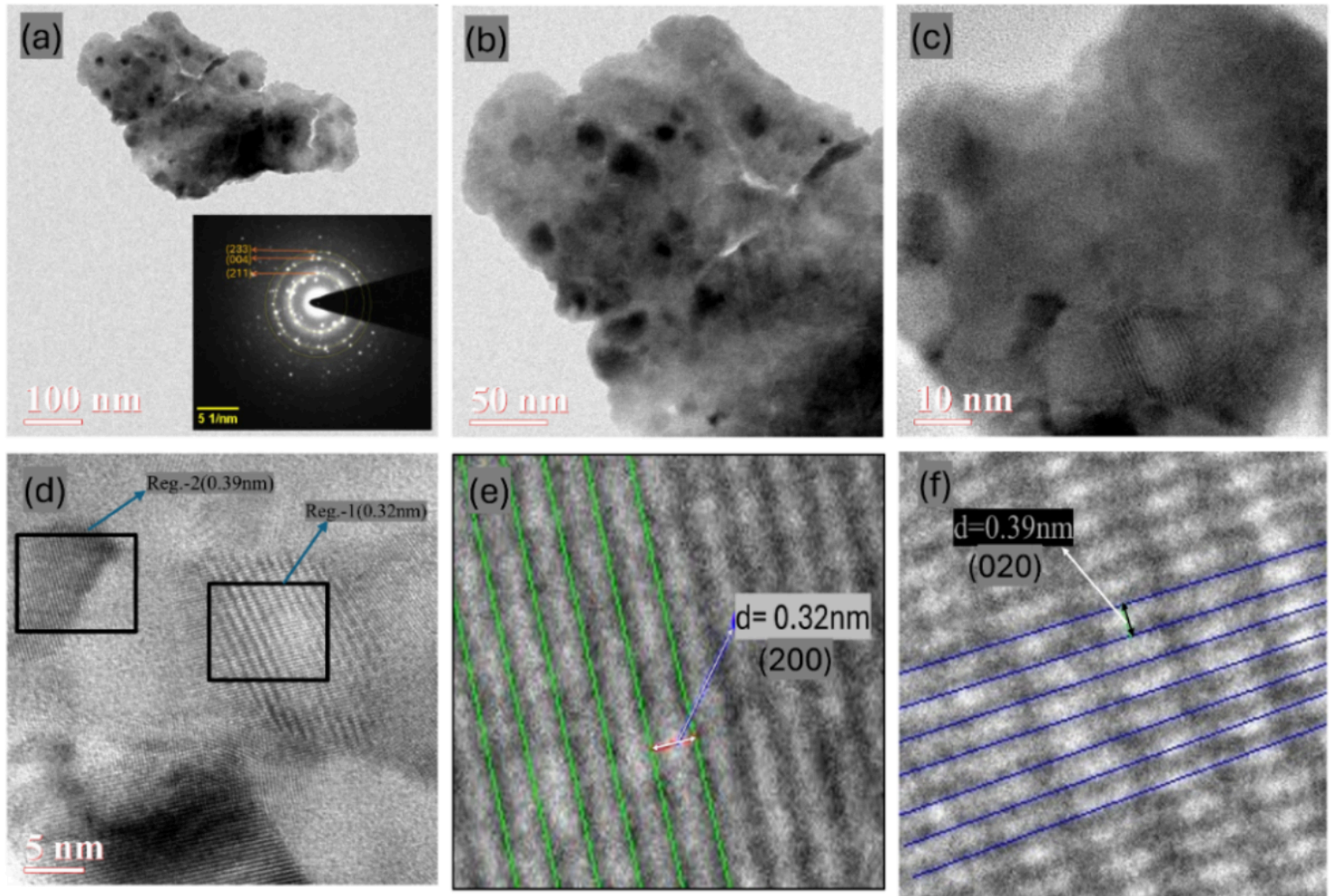


Figure 2

HR-TEM images of a ZnSb grain **(a-c)** showing multiple crystallites within a grain of few 100 nm size. SAED pattern of image (a) in its inset showing random orientations of crystallites within this single grain. **(d)** High resolution image showing randomly oriented crystallite planes having few nm dimensions. **(e-f)** Amplified view of two typical regions of (d), showing good crystalline quality of the material produced by the process.

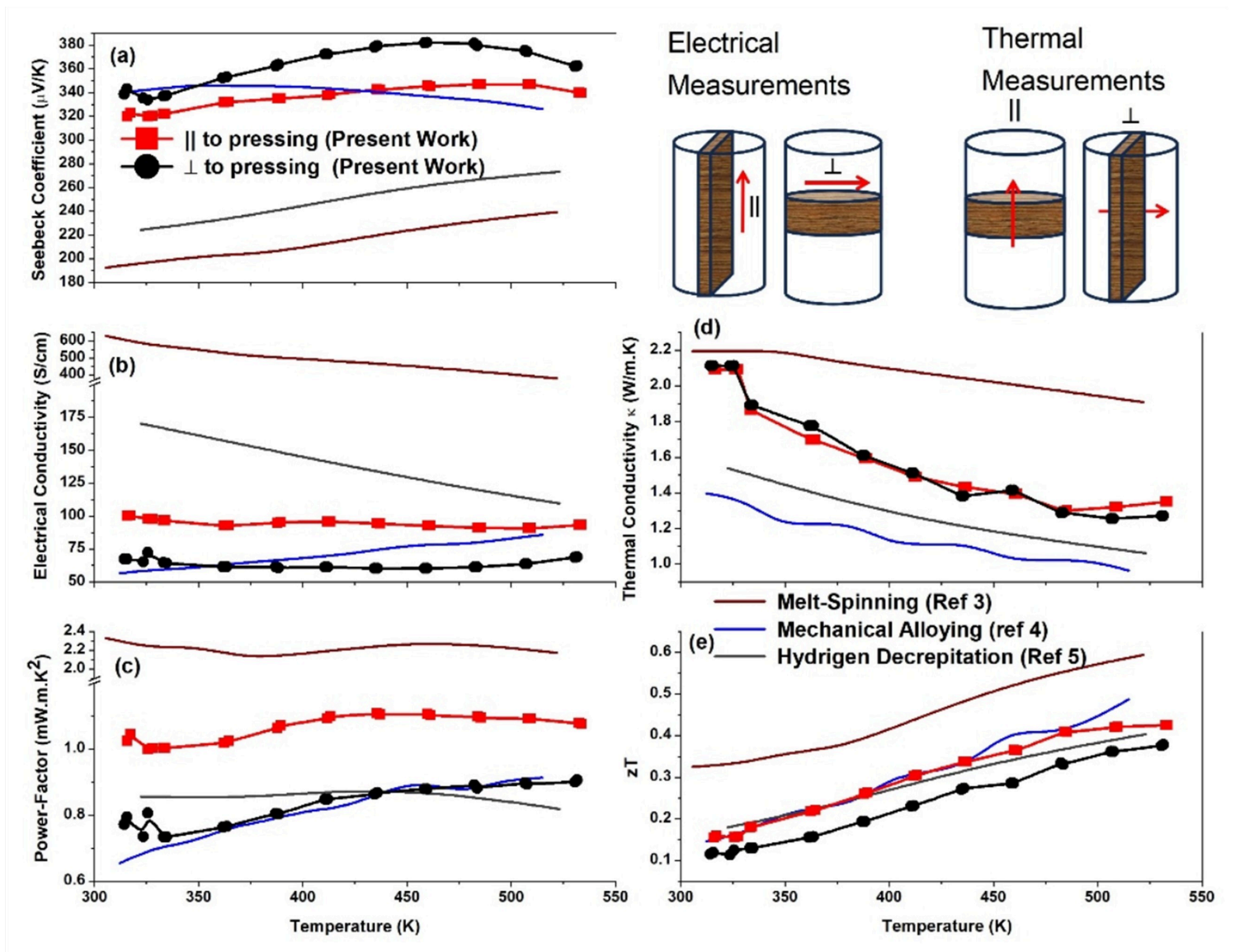


Figure 3

Thermoelectric properties in both parallel and perpendicular to the pressing direction in SPS consolidation and their comparison with those reported in literature using melt-spinning [3], mechanical alloying [4] and hydrogen decrepitation [5] processes.

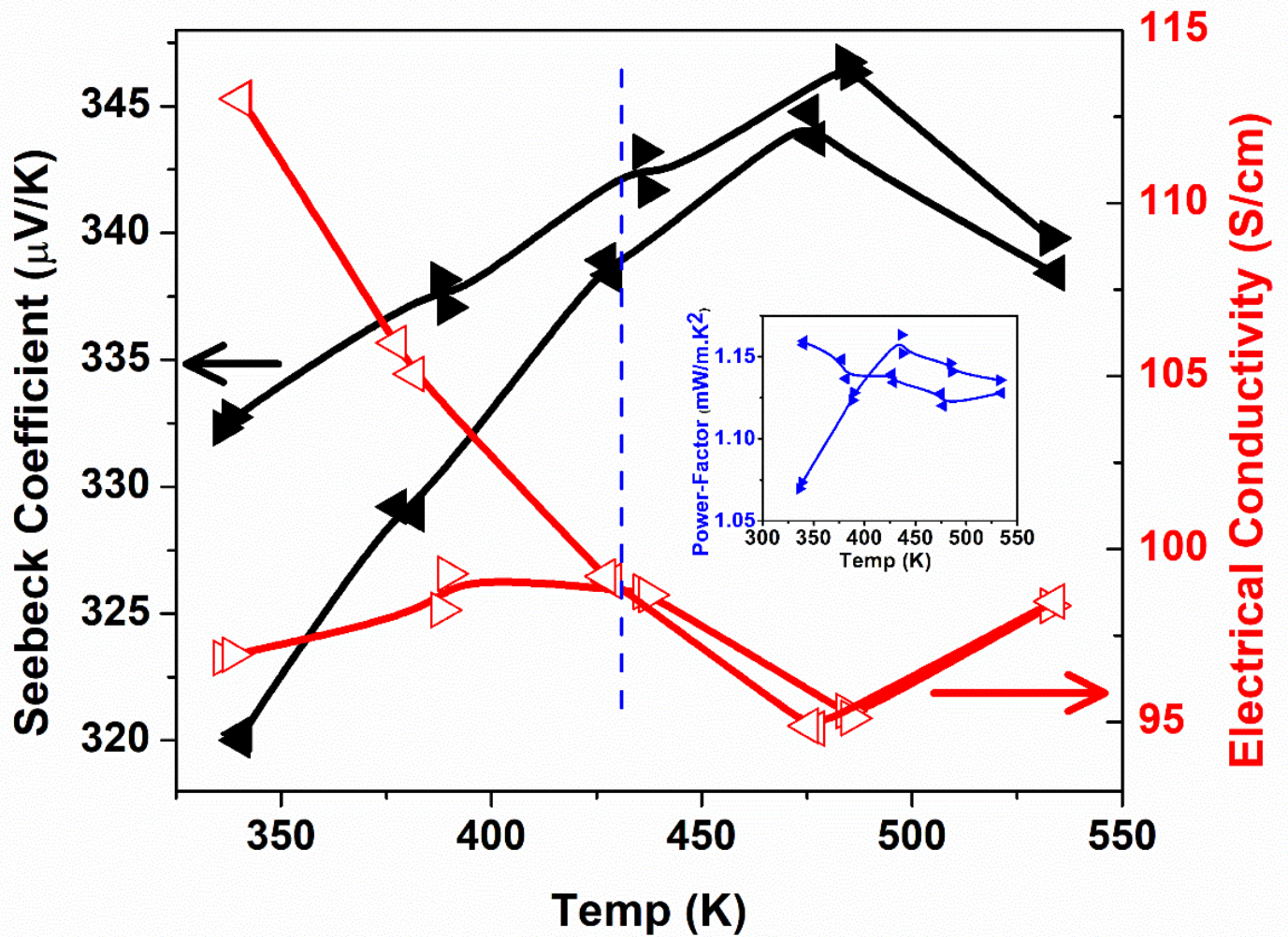


Figure 4

Seebeck coefficient and electrical conductivity of ZnSb nanomaterial are measured while heating (shown by right side arrow symbols) as well as cooling (shown by left side arrow symbols) cycles. Inset shows power-factor. Hysteresis has been observed below 425 K temperature in both Seebeck coefficient, electrical conductivity as well as power-factor, which indicate role of carrier scattering in electrical transport properties.

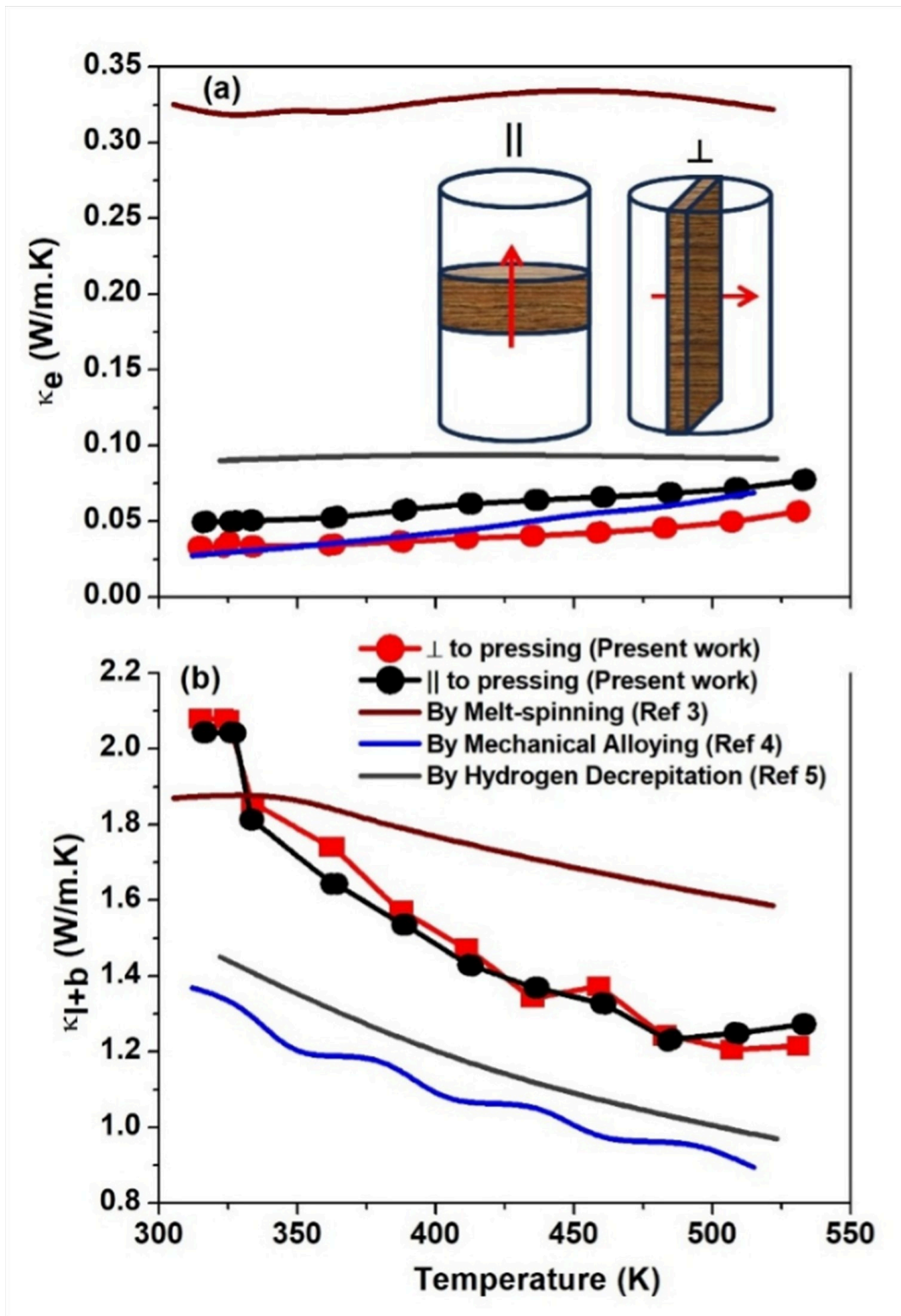


Figure 5

(a) Carrier contribution (κ_e) and (b) lattice with bipolar contributions (κ_{l+b}) to total thermal conductivity shows that carrier contribution is very low and total thermal conductivity is primarily governed by lattice thermal conductivity. κ_e shows isotropy, whereas, κ_{l+b} is isotropic. The values of κ_e resembles well to those reported by mechanical alloying [16] and lower than reported values for hydrogen decrepitation [6]. Values of κ_e are much higher by melt-spin processing [15], similar to higher electrical conductivity. The

values of κ_{I+b} around room temperature by this process is closer to the present process, but its value is higher at higher temperatures. This indicates that Zn atom vacancies may be causing increase in both κ_e and κ_{I+b} .

Supplementary Files

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