



## Analysis of heat capacity and Mössbauer data for $\text{LuZnSn}_2$ compound

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**Abstract.** New analysis of heat capacity data is presented for  $\text{LuZnSn}_2$  compound that takes into account anharmonic effects together with the existence of Einstein modes.  $^{119\text{m}}\text{Sn}$  Mössbauer spectroscopy was used to monitor the hyperfine parameters at the two crystallographically inequivalent Sn sites in the studied compound. The problem of non-unique mathematical resonance spectrum description and the problem how to choose physically meaningful set of hyperfine parameters will be thoroughly discussed. Measured quadrupole interaction constants by  $^{119\text{m}}\text{Sn}$  Mössbauer spectroscopy give estimations for  $V_{zz}$  component of electric field gradient tensor at both Sn sites in  $\text{LuZnSn}_2$ .

**Key words:** heat capacity • Mössbauer studies • rare earth ternary compounds

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### Introduction

Recently, new ternary intermetallic compounds  $\text{RZnSn}_2$  ( $\text{R} = \text{Y, Gd, Tb, Dy, Ho, Er, Tm, Lu}$ ) were synthesized and it was shown that they crystallize in the tetragonal  $\text{HfCuSi}_2$  structure type [1]. These compounds belong to the rich family of  $\text{R(A)-T-X}$  ( $\text{R}$  – rare earth or  $\text{A}$  – actinide element,  $\text{T}$  –  $d$  metal,  $\text{X}$  –  $p$  element) ternary intermetallics [2–21], which present a variety of crystal and magnetic structures and a plethora of interesting physical properties, including complex magnetism, unconventional superconductivity, and intermediate valence. Magnetic susceptibility and heat capacity results show that  $\text{LuZnSn}_2$  compound does not undergo any magnetic phase transition down to 1.9 K [1, 22], and therefore this compound is an ideal reference material for the estimation of magnetic contributions to heat capacity of magnetically ordered isostructural compounds.

The aim of this work is to present and discuss a new analysis of heat capacity data [22] obtained for  $\text{LuZnSn}_2$  with the help of an extended Debye model that fits experimental data in whole range of temperatures in contrast to the results obtained earlier [22] where simple Debye model was used. Additionally, the results of preliminary Mössbauer investigation made with  $^{119}\text{Sn}$  source will be reported and discussed for the compound under study.

## Experimental

The polycrystalline LuZnSn<sub>2</sub> sample has been synthesized by reacting the elements at ~1000°C followed by annealing at 400°C, as described previously, and its crystal structure was determined from powder diffractogram using the FullProf program [23] for Rietveld refinements [1].

The bulk magnetic and specific heat measurements were performed in the temperature range of 1.9–300 K by means of the vibrating sample magnetometer (VSM) and the heat capacity (HC) options of the Quantum Design physical property measurement system (PPMS). The specific heat studies reported here were made in zero external magnetic field. Special care was taken to correct in a proper way the raw HC results taking into account the specific heat contribution originating from the adhesive addenda (*Apiezon N*) that is used to couple a given sample to the PPMS HC platform.

The <sup>119</sup>Sn Mössbauer spectrum has been measured at room temperature utilizing <sup>119m</sup>Sn source in CaSnO<sub>3</sub> matrix. A Mössbauer spectrometer of an electromechanical type was used in the constant-acceleration mode. The 23.875 keV  $\gamma$ -rays were detected with a proportional counter. The velocity scale was calibrated at room temperature with a <sup>57</sup>Co(Rh) source and a metallic iron foil. The obtained resonance absorption spectrum of <sup>119m</sup>Sn was analyzed by means of least-squares fitting procedures using a full hyperfine interaction Hamiltonian within the transmission integral.

## Results and discussion

### X ray diffraction

X-ray diffraction analysis showed that the LuZnSn<sub>2</sub> compound crystallizes in the tetragonal HfCuSi<sub>2</sub> structure type (*P4/nmm* space group). Two symmetry inequivalent Sn sites *2a* (*4m2*) and *2c* (*4mm*), exist in the unit cell and a 1:1 nominal occupational ratio was observed [1].

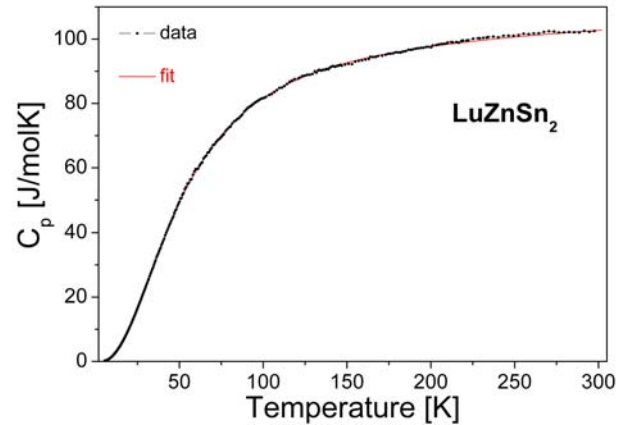
### Heat capacity

Figure 1 presents the temperature dependence of the specific heat. It can be seen that the total specific heat data ( $C_p$ ) can be fitted very well in the whole temperature range with the formula that accounts

for the phonon and electronic contributions ( $C_p = C_{\text{lattice}} = C_{\text{ph}} + C_{\text{el}}$ ;  $C_{\text{el}} = \gamma T$ ):

$$(1) \quad C_p = C_{\text{ph+el}} = 9R \frac{1}{1-\alpha T} \left( \frac{T}{\theta_D} \right)^3 \int_0^{\frac{\theta_D}{T}} \frac{x^4 e^x}{(e^x - 1)^2} dx \\ + R \frac{1}{1-\alpha T} \sum_i m_i \frac{\left( \frac{\theta_{E_i}}{T} \right)^2 e^{\theta_{E_i}/T}}{(e^{\theta_{E_i}/T} - 1)^2} + \gamma T$$

where  $\theta_D$  is the Debye temperature,  $\theta_{E_i}$  ( $i = 1, 2$  or  $3$ ) are the Einstein temperatures,  $\alpha$  is the anharmonic coefficient,  $R$  is the gas constant and  $\gamma$  stands for an electronic specific heat coefficient. All parameters derived from the fit are gathered in Table 1 together with the applied multiplicities  $m_i$  for each individual Einstein branch  $E_i$ . The last column of Table 1 contains the parameters obtained previously using the standard Debye formula for the description of the pure phonon contribution [22]. It is clearly seen that these last parameters, in spite of simplifications that were used (see Ref. [22]) are in good agreement with those obtained in this work, pointing to a conclusion that the simple Debye formula gives rather good estimation for the Debye temperature  $\theta_D$ , at least as a first approximation. It has to be also underlined, that the obtained value of  $\gamma$  ( $\gamma = (5.28 \pm 0.05) \text{ mJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-2}$ ) is typical for nonmagnetic metallic ternary compounds.



**Fig. 1.** Temperature dependence of the specific heat ( $C_p$ ) for the LuZnSn<sub>2</sub>. The continuous line presents the fit to LuZnSn<sub>2</sub> data using the modified Debye expression (see Eq. (1)), as described in the text. The derived fitting parameters are gathered in Table 1.

**Table 1.** The results for the derived fitting parameters from heat capacity data using Eq. (1)

Parameter	Value	Multiplicity ( $m_i$ )	Parameter values previously obtained in Ref. [22] using simplified assumptions
$\theta_D$	$(141.78 \pm 0.34) \text{ K}$		$(148.0 \pm 3.0) \text{ K}$
$\theta_{E1}$	$(56.38 \pm 0.11) \text{ K}$	$\times 1$	
$\theta_{E2}$	$(156.50 \pm 0.47) \text{ K}$	$\times 5$	
$\theta_{E3}$	$(247.4 \pm 2.1) \text{ K}$	$\times 3$	
$\alpha$	$(13.08 \pm 0.77) \times 10^{-5} \text{ K}^{-1}$		
$\gamma$	$(5.28 \pm 0.05) \times 10^{-3} \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-2}$		$(5.75 \pm 0.20) \times 10^{-3} \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-2}$

<sup>119</sup>Sn Mössbauer spectroscopy

The point symmetries of Sn environments i.e.  $\bar{4}m2$  (2a site) and  $4mm$  (2c site), ensure non-zero, axially symmetric (with asymmetry parameter  $\eta = 0$ ) electric field gradient tensor EFG at these sites, and hence a non-zero electric quadrupole hyperfine interactions at the Sn positions. In accord with the two non-cubic environments of Sn atom in the crystal structure (i.e. 2a and 2c sites) the obtained room temperature spectrum can be fitted with two quadrupole split components including single line impurity in the form of metallic tin (with isomer shift  $\delta_{is}$  kept constant and equal to 2.552(9) mm/s [24]), which is also well established from X-ray analysis and originating from the synthesis procedure as well as the known decomposition process of the probe with time. The problem is that the applied fitting procedures are not unique, which has been illustrated in Fig. 2 for only two extreme examples.

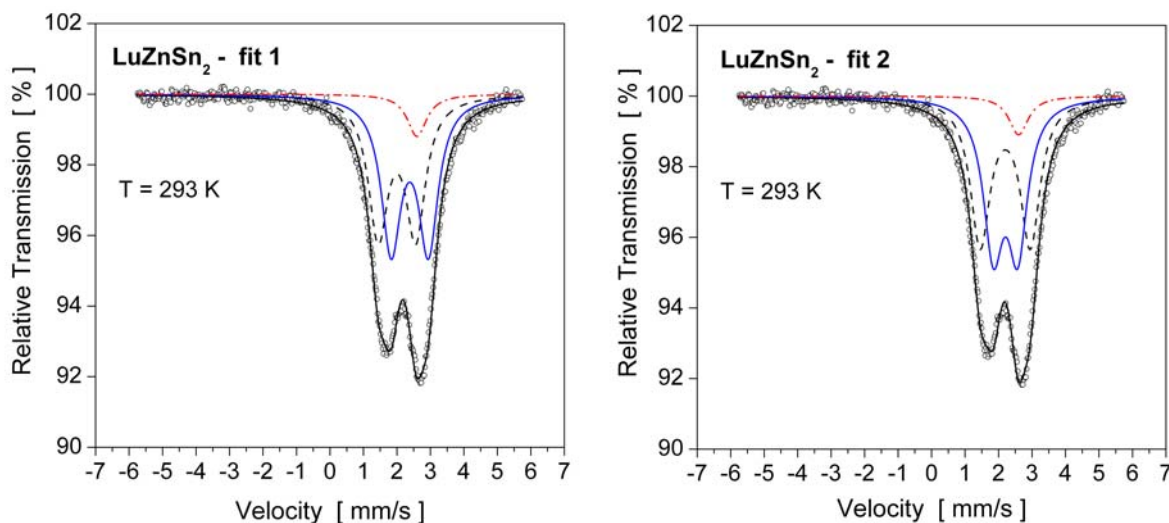
Generally, to get reliable results, from a given fitting procedure, some parameters, like half widths for source  $\Gamma_s$  and absorber  $\Gamma_a$ , have to be constrained and kept constant, being equal to those derived from earlier experiments with reference to BaSnO<sub>3</sub> absorber. In this case these values are as follows:  $\Gamma_s = 0.37$  mm/s and  $\Gamma_a = 0.33$  mm/s. Additionally, only fits with comparable relative intensities for both quadrupole components were accepted to reflect the fact that the occupation of above-mentioned Sn sites are equal. It must be stressed, that fits with constrained amplitudes of main Sn contributions

with their 1:1 ratio, do not change remarkably the goodness,  $\chi^2$ , of such a fit. Small differences between relative intensities can be always explained by possible differences in Debye–Waller  $f$  factors for both symmetry sites of tin. This is the main reason why in Table 2 the derived Mössbauer results are presented for fits with unconstrained main Sn contributions. The subtraction of the sum of the derived intensities for both quadrupole components from 100% gives the contribution of the Sn impurity being of about 6% for different fit tries (see Table 2). Closer inspection of the obtained results (Table 2) clearly shows that both fits have almost the same quality factors  $\chi^2$ , in spite of the fact they lead to different interpretations. In the first case (fit number 1, left side of Fig. 2) one can see that two Sn locations have much different isomer shifts but almost comparable quadrupole splittings, in frame of the observed error limits, while in the second case (fit number 2, right side of Fig. 2) the opposite situation takes place, i.e. isomer shifts are in turn almost the same but quadrupole splittings are quite different. It seems that this second situation describes in a much better way the physical reality, since for two different symmetry positions the observed quadrupole splittings should be rather different. As a matter of fact, such situation is observed in the closely related defected GdT<sub>x</sub>Sn<sub>2</sub> compounds [21], where Sn atoms also occupy two different symmetry positions and where Mössbauer spectra can be fitted in a proper way only with two different quadrupole splittings, but with very similar isomer shifts for both Sn sites. On the other hand

**Table 2.** Hyperfine interaction parameters inferred from the <sup>119</sup>Sn resonance spectrum obtained for the LuZnSn<sub>2</sub> intermetallic compound at 293 K using two different fitting procedures

Fit number	Component	$\delta_{is}$ [mm/s]	$ \Delta E_Q ^*$ [mm/s]	Relative area [%]	$\chi^2$
1	I	2.000(7)	2.236(12)	44.5(1.4)	0.9670
	II	2.383(6)	2.224(15)	49.0(1.3)	
2	I	2.200(3)	3.027(23)	45.1(1.1)	0.9671
	II	2.207(4)	1.495(20)	48.9(1.2)	

\* $\Delta E_Q = eQV_{zz} = 2\Delta$  where  $\Delta$  is overall quadrupole splitting for a given component in the recorded spectrum (see Fig. 2).



**Fig. 2.** <sup>119</sup>Sn Mössbauer spectrum of LuZnSn<sub>2</sub> at room temperature fitted with two extreme fitting philosophies, using two quadrupole contributions and one strictly settled single line metallic tin impurity component (see text for explanation).

an assumption that isomer shifts should be much different at Sn sites seems to be also unreasonable, since for Sn atoms their change requires a rather big change in valence electron  $sp$  configuration of own tin atom [25], and this is not expected here, as in the  $2a$  position Sn atoms interact directly with other four Sn atoms and four Lu atoms, while at  $2c$  site interact with four Zn (instead of Sn) and four Lu atoms but valence  $s$  configurations of Sn and Zn atoms stay roughly the same. The observed systematics of isomer shifts of Sn in most intermetallic systems (including ternary systems) [25] is in line, with the previous statement, showing that the effective number of  $5s$  tin electrons is merely the same and not much different than one (see Table 9a in Ref. [25]). In this context, one can state that a support from theory in predicting Mössbauer parameters would be of great importance. In particular, the theoretical calculations could enable the right association of the obtained sets of experimental results for hyperfine parameters to a given site in the crystallographic structure and also allowing for right interpretation of the derived isomer shifts and quadrupole splittings, giving a deeper insight into the electronic structure of the studied material. In this aspect one can only speculate, for example, that a larger quadrupole splitting could be associated to lower symmetry site of Sn. Nowadays, the use of density functional theory (DFT), is quite successful in this matter, but up to our best knowledge such calculations were not made for Sn hyperfine parameters in the system under study.

The values of the electric field gradient  $V_{zz}$  at the tin nuclei can be obtained using the quadrupole interaction constants  $\Delta E_Q = eQV_{zz}$  (where nuclear quadrupole moment  $Q$  for the first excited state of  $^{119m}\text{Sn}$  is equal to  $Q = -0.094$  b [26]) given in Table 2 by the simple relation:  $V_{zz} = -8.47128 \times 10^{21} \Delta E_Q$  [mm/s] V/m<sup>2</sup> and for the preferred fit number 2 they are  $|V_{zz1}| = 25.646 \times 10^{21}$  V/m<sup>2</sup> and  $|V_{zz2}| = 12.664 \times 10^{21}$  V/m<sup>2</sup>, respectively.

## Conclusions

It was shown here that the experimental heat capacity data,  $C_p(T)$ , for  $\text{LuZnSn}_2$  can be successfully fitted in the whole temperature range taking into account anharmonic effects occurring at elevated temperatures, as well as by inclusion of respectively chosen Einstein modes. In this way, the observed above 36 K fit deviations from the  $C_p(T)$  experimental results when these data are analyzed in term of simplified Debye model [22] were canceled.

The resonance spectrum recorded at room temperature can be fitted with different pairs of main quadrupole split subsets of hyperfine parameters for two crystallographically inequivalent Sn sites in  $\text{LuZnSn}_2$ . However, from a physical point of view, only fit with two different quadrupole interaction constants and comparable isomer shifts for both Sn sites is acceptable. Those obtained by  $^{119m}\text{Sn}$  Mössbauer spectroscopy quadrupole interaction constants allow for the determination of  $V_{zz}$  components of electric field gradient (EFG) tensor at both Sn sites

in  $\text{LuZnSn}_2$ . The derived isomer shifts of about 2.20 mm/s are characteristic for tin in metallic systems.

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