After the RISEnergy Transnational Access, Users are required to submit a User Report. This should be done within 4 weeks after the Access is completed unless otherwise agreed. The User Report will be given to the User(s) by the WP2 leader. The report contains sections related to the work performed, the main results and observations that were achieved.

This document should be completed, signed, and sent by e-mail to risenergy@for.kit.edu.

Summary questionnaire for Users who have been granted Transnational Access (TA) under the RISEnergy project Horizon Europe TA scheme. More information on RISEnergy TA can be found in "General Rules" and in "Access Policy" which can be found on the RISEnergy webpage.

Please complete, sign, and send this form, together with the Cost claim by e-mail to risenergy@for.kit.edu *with title: RISEnergy APP141 - reports.*

General information about the project	
Project title (as used in Application)	Optimization of Cascaded Thermoelectric Devices for High-Temperature Applications
Project number (APPXXX) and acronym (max 15 characters)	APP141, CASCAD-THERM
RISEnergy RI(s) accessed	CNR-IMA3: Diathema Lab, CNR-Rome
Keywords (up to five, free text)	Material Characterisation, Thermal Stability, Thermal Interface.
Arrival date (in town where RI is located)	29/06/2025
Departure date (from town where RI is located)	04/07/2025
Starting date of Access (first day at RI)	30/06/2025
Finishing date of Access (last day at RI)	04/07/2025
Number of days not using the RI (during the above period)	0
Reason for not using RI those days (describe)	
Number of days using the RI	5
Number of Users granted Access (group size)	2
Comments	

User

RISEnergy

User group leader or sole applicant (user group member 1)

First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
User travelling to RI?	
Comments	

User group member 2

First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
User travelling to RI?	
Comments	

User group member 3

First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
User travelling to RI?	
Comments	

Access Summary Report - work performed and initial results

Brief description of the objectives of your project (up to 200 words)

Main objectives of the project are listed below:

- Material Characterisation with Raman spectroscopy and Scanning Probe Microscopy
  Investigate the stability of the Tetrahedrite and MgSb material up to 500 °C. With scanning probe microscopy, investigate the local resistance changes across the material cross-section.

- Investigate the possibility of thermal conductivity measurements of Silicon Germanium samples.
  Exploring optothermal thermal conductivity measurement as a method to estimate the thermal conductivity of SiGe

- Evaluation of the Thermal interfacing of conductive coupling for the hot side of the Cascaded Module.

Activities performed (up to 600 words)

[Please summarise the work carried you (steps taken, instrumentation used, techniques employed, data sources consulted etc.)]

Material characterisation and Optothermal Measurements for thermal conductivity with Raman spectroscopy.

Both Tetrahedrite and Magnesium Antimonide samples were evaluated for stability with Raman spectroscopy. The samples were polished and cleaned with 600-grit SiC grinding paper. The sample was placed in the environment chamber, which had a hot stage capable of heating the sample to 600 °C. Raman spectroscopy measurements were executed using a Horiba Scientific LabRam HR Evolution confocal spectrometer equipped with a 100mW green laser source (λ_{exc} = 532 nm) and a computerized XY-table, an electron-multiplier CCD detector, and an Olympus U5RE2 microscope with a 50x long working distance objective (laser spot on the sample surface 1.3 µm). The numerical aperture (NA) of 0.5 was used along with a grating with 300 grooves/mm. A laser power of 10 mW is applied to the sample to heat it. Initially, the Raman spectra of the materials are collected at room temperature to use as a benchmark. Subsequently, the temperature was increased to 500 °C, and at each temperature, the Raman spectra were acquired after holding at that temperature for 3 minutes.

For the measurement of Thermal conductivity with Raman spectroscopy, first, the sample was mounted, and a low-power laser (532 nm) was used to acquire a baseline Raman spectrum, identifying a temperature-sensitive peak. This establishes room temperature spectral characteristics. A calibration curve is obtained by increasing the temperature and capturing the Raman spectra for different temperatures until 600 °C. Next, the power of the laser was increased and focused onto the same spot to induce localized heating. Its power was systematically varied. Raman spectra were then acquired from the heated spot for each heating laser. The temperature change (ΔT) at the laser spot was determined by analysing the shift in the temperature-sensitive Raman peak, using a pre-established calibration curve relating peak shift to temperature. The absorbed laser power (P_{abs}) was calculated from the incident power and the material's optical absorption. Finally, the thermal conductivity (κ) was extracted by fitting the experimental ΔT vs. P_{abs} data to a 2D heat transfer model, accounting for sample geometry. Multiple measurements were taken to ensure statistical significance.

Localised Resistance Measurements with C-AFM

Conductive Atomic Force Microscopy (C-AFM) is a powerful SPM technique that simultaneously maps surface topography and local electrical current. Both Magnesium Antimonide and Tetrahedrite samples are mounted within the AFM, and measurements are done on surfaces. For conductive AFM measurements, first cantilever tip is aligned to the region of interest using the optical microscope, then the sample is approached. The AFM tip in contact mode is engaged on the surface of the material to ensure continuous electrical contact.

Parameters like the force of the tip are also optimized to maintain stable contact and good electrical connection without damaging the tip or sample. In the next step, scanner parameters like scan size and rate are fixed. Then, a precisely constant voltage bias of 1 V is applied to the sample surface. Finally, the scan commences, and a highly sensitive current amplifier measures the current flowing through the tip-sample junction, revealing variations in electrical properties across the surface. Both samples used in the measurement were previously heat-treated by isothermally holding at 260 °C.

Evaluation of Thermal interfaces at the hot side of the Cascaded Module

For evaluating the thermal interfacing at the high-temperature region of the cascaded TEG, the TEG was placed in the VTEC. VTEC (Vacuum & Temperature Electronic Characterization) is a vacuum chamber that allows the electrical characterization of materials in conditions of high vacuum (with pressures < 10-7 mbar) as a function of temperature: currently, the chamber is equipped with 3 stages which allow for characterization at low (up to 600 ° C), medium (up to 1000 °C) and high (up to 1800°C) temperature. In the current study, the medium-temperature stage is used, featuring a Boron nitride plate that interfaces with a graphite heater. The TEG setup comprises a stainless steel plate located in the high-temperature region, enabling conductive heat transfer to the TEG. The TEG setup, along with the stainless steel plate, is placed on top of the BN plate directly, and also with a Mo plate between the BN and stainless steel plates. In both cases, temperatures at the steel plate and the hot side of the TEG are measured to understand the effects of both kinds of thermal interfacing. The schematic of the cascaded TEG and the experimental setup is given in Figure **1**. Thermocouples are then attached to the steel plate to monitor the temperature change.

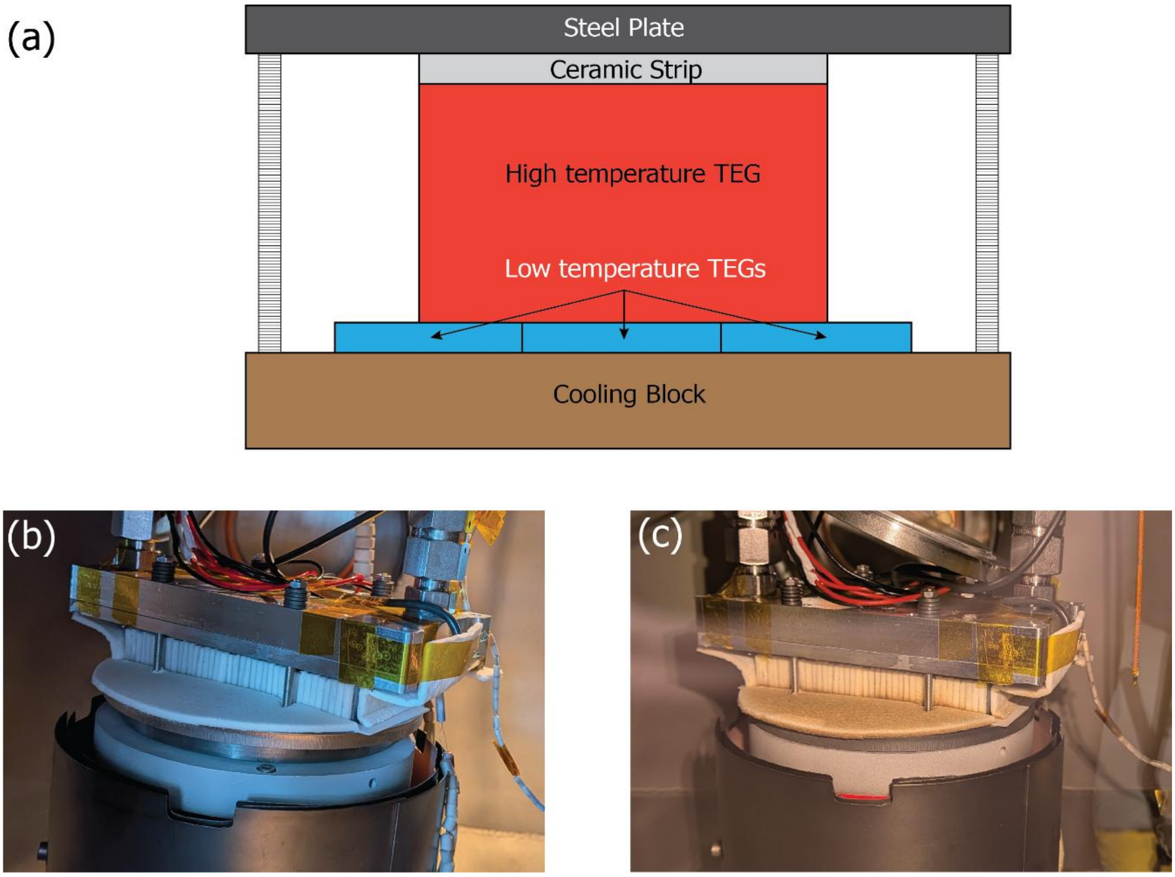


Figure 1(a) Schematic of the cascaded TEG and the experimental setup used to observe the temperatures in the stainless steel plate with both types of thermal interface with the heater (b) with a molybdenum plate and (c) without a molybdenum plate.

Scientific results (up to 800 words)

[Summarise the (initial) outcomes of your study at the RI(s).]

1.1 Evaluation of Tetrahedrite and Magnesium Antimonide (Mg_3Sb_2) at high temperatures (up to 500 °C)

In the case of the Mg_3Sb_2 samples, no Raman spectra could be observed at room temperature or at elevated temperatures. Even after carefully polishing off the surface oxidation layer and repeating the measurements, the results remained unchanged.

However, in the case of Tetrahedrite samples, at room temperature, Raman spectra can be observed with two distinct peaks at ~90 cm^{-1} and 360 cm^{-1}, as presented in Figure 2. When the temperature is increased, the peaks can be observed to have reduced intensity. This can be related to the reduction in Stokes Raman intensity caused by the decrease in population of the ground state. However, no other changes are observed in the material spectra at higher temperatures. This can be indicative of the material stability at measurement temperatures. To validate these results, the Raman spectra were compared with TGA results of the material done at 350 °C, in which no change in the material was observed for the time corresponding to Raman measurements.

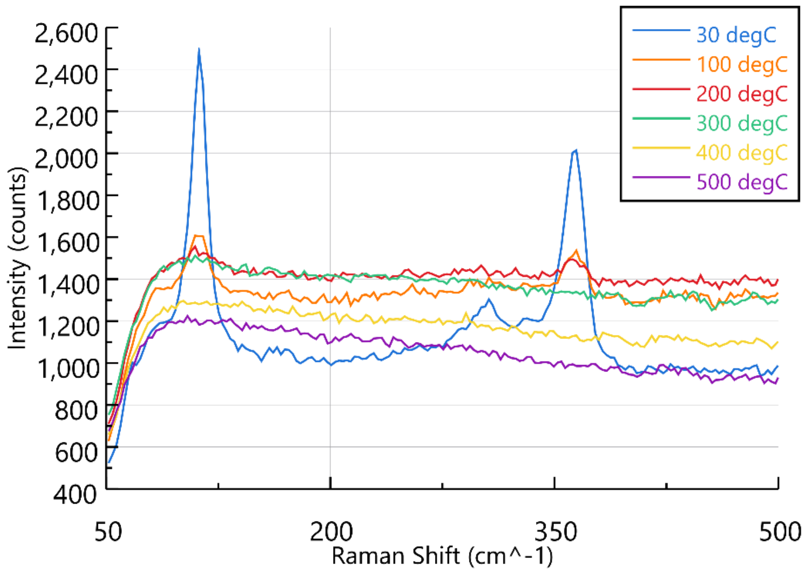


Figure 2: Raman spectra of Tetrahedrite obtained at different temperatures, indicating the stability of the material

1.2 Optothermal measurement of thermal conductivity based on Raman Spectroscopy of SiGe wafer material

The Raman spectra obtained at different temperatures on a 10 mm × 10 mm wafer of 0.5 mm thickness are given in Figure **3**. The Raman spectra do not indicate any structural changes or formation of new phases.

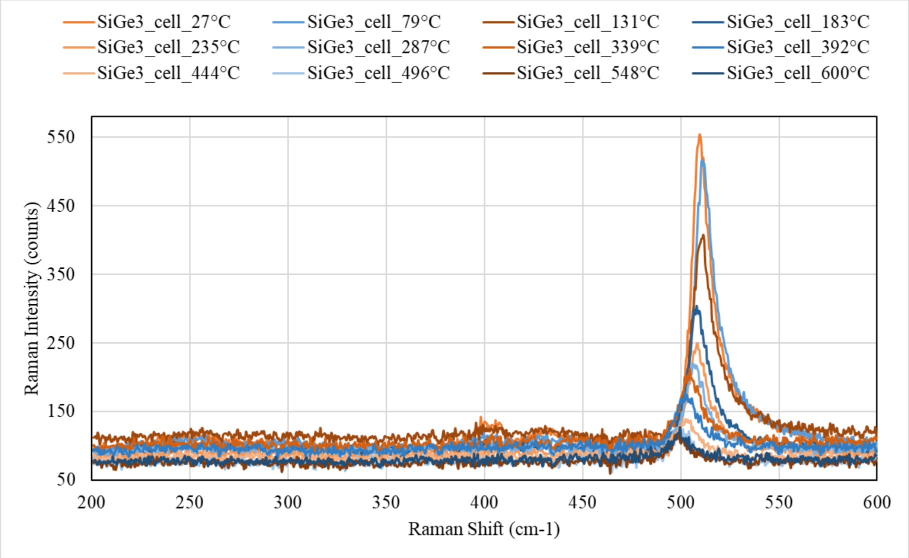


Figure 3: Raman spectra of the Silicon germanium (SiGe) sample corresponding to different temperatures

However, the peaks in the Raman spectra display a shift as the temperature is increased. To get a better statistical representation, the measurement is repeated on four samples. The peak position vs temperature is plotted to obtain the calibration curve as indicated in Figure 4.

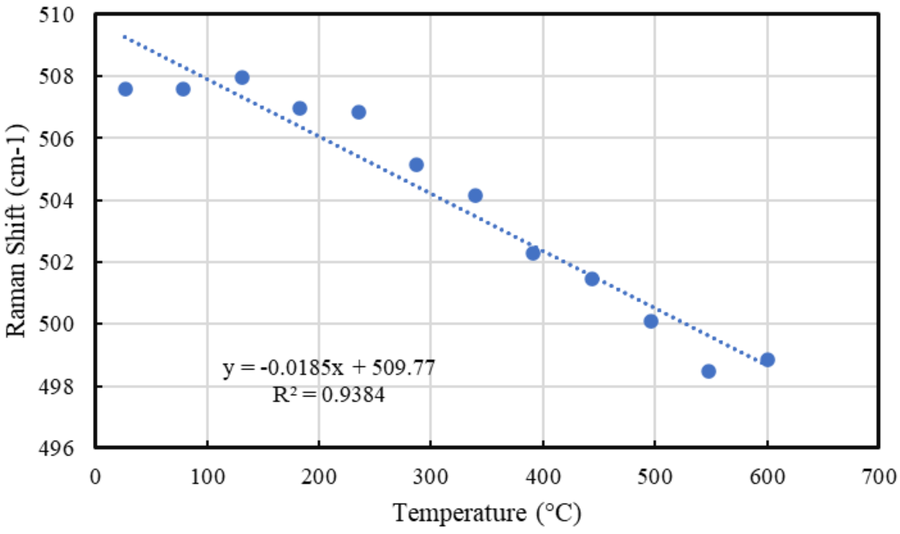


Figure 4: Temperature vs Raman peak shift curve to be used for calibration of Silicon Germanium wafer material

The relation between peak shift and temperature is given by the slope of the linear fit applied to the data, as given in the equation below.

$$\Delta\omega = \kappa\Delta T \tag{1}$$

where $\Delta\omega$ is the peak shift, ΔT is the temperature change, and κ is the value of the slope. In this case, the slope of the calibration curve is 0.0185. Now the laser power is increased and localized heating is induced in the SiGe wafer material. The Raman spectra are measured at each power setting with incident powers of 0.1,1, 3.2, 5, 10, 25, 50, 100 mW. This is repeated for four samples, and the peak position at each power setting is averaged. The averaged peak position is plotted with the Power absorbed, and a linear fit is applied as shown in Figure **5**. The absorbed power is given by the equation given below.

$$P_{absorbed} = P_{incident} * a \tag{2}$$

Where, $P_{incident}$ is the incident power used in each power setting, and is the absorption coefficient of SiGe. In the current study, a value of 0.6 is used based on the individual absorption coefficients of Si and Ge.

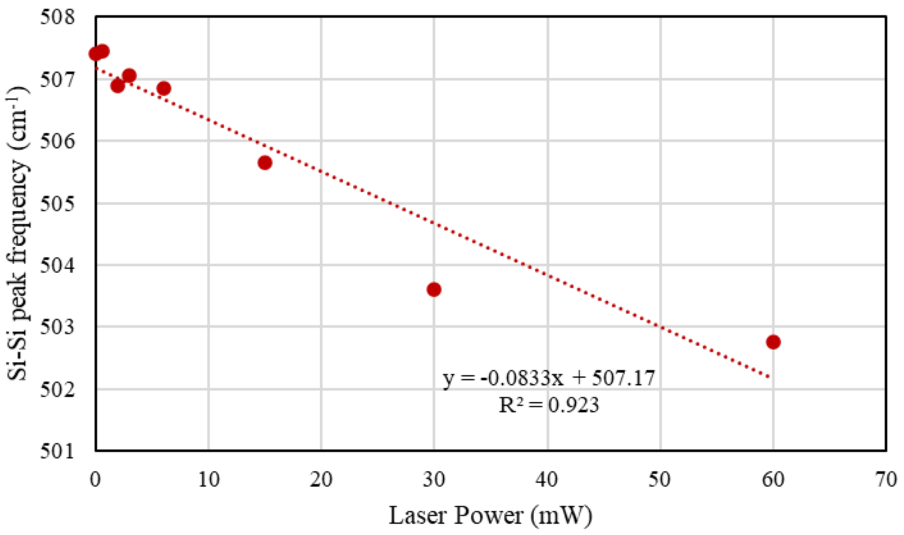


Figure 5: Average Peak position for the SiGe wafers for different power settings.

Based on both curves, the thermal conductivity of the material is given by the equation 3 below, which assumes heat conduction to a semi-infinite half-space.

$$\kappa_{thermal\ conductivity} = \frac{\frac{\Delta\omega}{\Delta T}}{4\sqrt{\pi}w\frac{\Delta\omega}{\Delta P}} \tag{3}$$

Where w is the standard deviation of the laser spot. The values used in the estimation of thermal conductivity are given in Table **1** below.

Table 1: Parameters for the thermal conductivity model estimated from the Raman spectrum data obtained at different temperatures and power settings

Parameter	Value
$\frac{\Delta\omega}{\Delta T}$	-0.0185
$\frac{\Delta\omega}{\Delta P}$	-0.0833
W	1.3 µm

Based on the estimated parameter, the thermal conductivity of SiGe is estimated to be 24.1 W/mK.

1.3 Conductive-Atomic force microscopy (AFM) of Tetrahedrite and Mg_3Sb_2 for local current probing

Both tetrahedrite and magnesium antimonide material, after heat treatment, are probed for variation in local current across the cross-section. A schematic of the TH and Mg3Sb2 cubes is given in Figure 6 below.

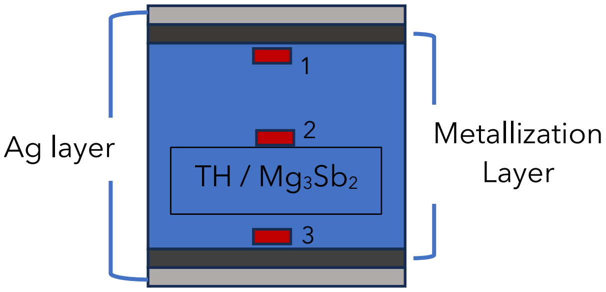


Figure 6: Schematic of the crossection of TH and Mg3Sb2 samples used in conductive AFM measurements. Regions which are scanned by C-AFM is indicated by red rectangles.

The AFM tip is scanned over a small area from the top, middle, and bottom of the sample to understand the variation in the local current flow across the sample cross-section.

In case of Mg3Sb2 samples, C-AFM scan results from all three regions for a bias voltage of 1 V and 2 V are given in the Table **2**.

Table 2: Localized current detected at different regions in the Sintered Mg3Sb2 samples

	Voltage Bias 1 V			Voltage Bias 2 V		
Area	Average Current (pA)	Maximum Current (pA)	Minimum Current (pA)	Average Current (pA)	Maximum Current (pA)	Minimum Current (pA)
1	2346	92295	-764	5911	19424	-1309
2	8591	11465	117	17663	22186	-2117
3	4432	10506	115	8254	19829	-341

In the case of using both 1 V and 2 V voltage bias for measurement, the average current seems to proportionally increase, indicating ohmic behaviour. The significant variation in current values indicates the heterogeneity of the material. This points towards the presence of more conductive phases in the material. The current mapping given in **Figure 7**: Current mapping from C-AFM measurements done locally on different regions with the Mg3Sb2 sample points towards variation in the local electrical resistance of the material. For example, a higher current is observed in region 2 of the sample, indicating lower resistance. And in both regions 1 and 3, the currents can be observed to be higher, which can be a result of higher local electrical resistances in these regions. However, in all the regions the materials seem to be quite homogenous from the electrical point of view, without formation of defects or zones with different electrical behaviour.

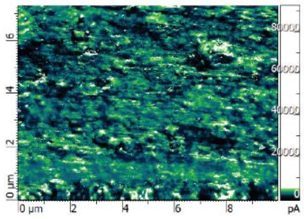
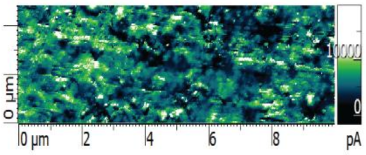
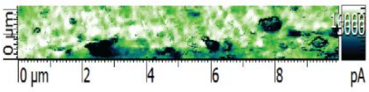
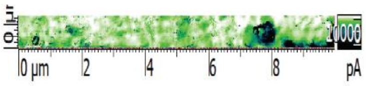
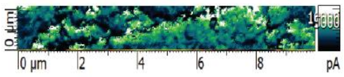
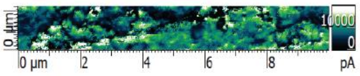
Region	1V Bias	2V Bias
Region 1		
Region 2		
Region 3		

Figure 7: Current mapping from C-AFM measurements done locally on different regions with the Mg3Sb2 sample

In the case of the tetrahedrite sample, due to the high conductivity of the sample, the measurements were highly unstable. Applying a bias of just a few millivolts resulted in currents on the order of microamperes. At higher bias voltages, the sample exhibited an anomalous response, likely due to saturation effects in the experimental setup, as indicated in Figure 8 given below.

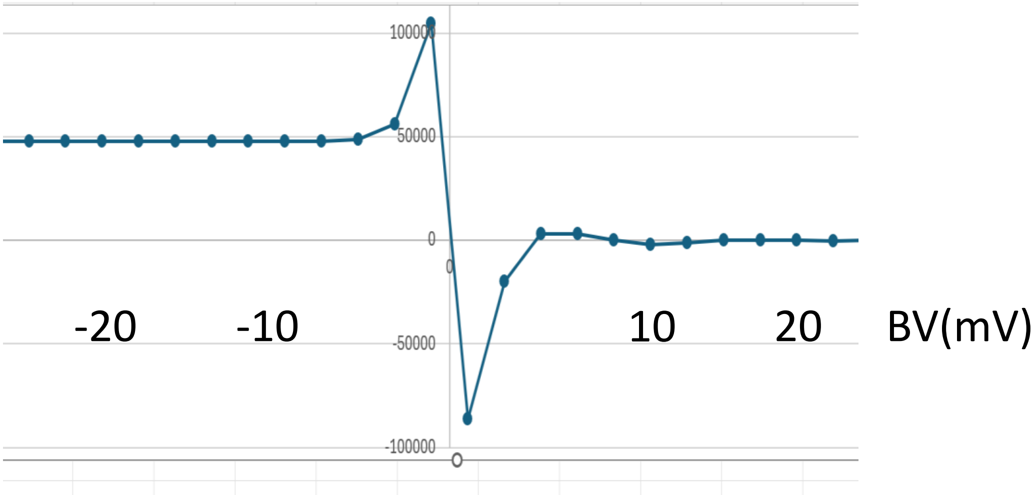


Figure 8: V-I curve obtained from the C-AFM measurement of Tetrahedrite samples

1.4 Evaluation of thermal interfacing at the hot side of the cascaded Thermoelectric Generator

A cascaded thermoelectric generator (TEG) module was attached to a graphite heater. We investigated the effect of a molybdenum plate on the heat transfer from the heater to the TEG. A thermocouple monitored the temperature of a steel plate within the TEG setup as we increased the power to the heater, and is presented in Figure **9**: Temperature profiles obtained from the thermocouple attached to the steel plate with and without a molybdenum plate for thermal interfacing with the heaterFigure **9**.

The results showed that using the molybdenum plate significantly lowered the temperatures measured on the steel plate compared to the setup without the plate. At the highest power setting, the temperature reached approximately 630 °C without the molybdenum plate, but only about 420 °C with the molybdenum plate in place. This indicates that the molybdenum plate created a substantial thermal resistance, hindering the transfer of heat to the TEG module. This can also be related to the high contact thermal resistance between the molybdenum plate and the steel plate due to the surface roughness of the molybdenum plate.

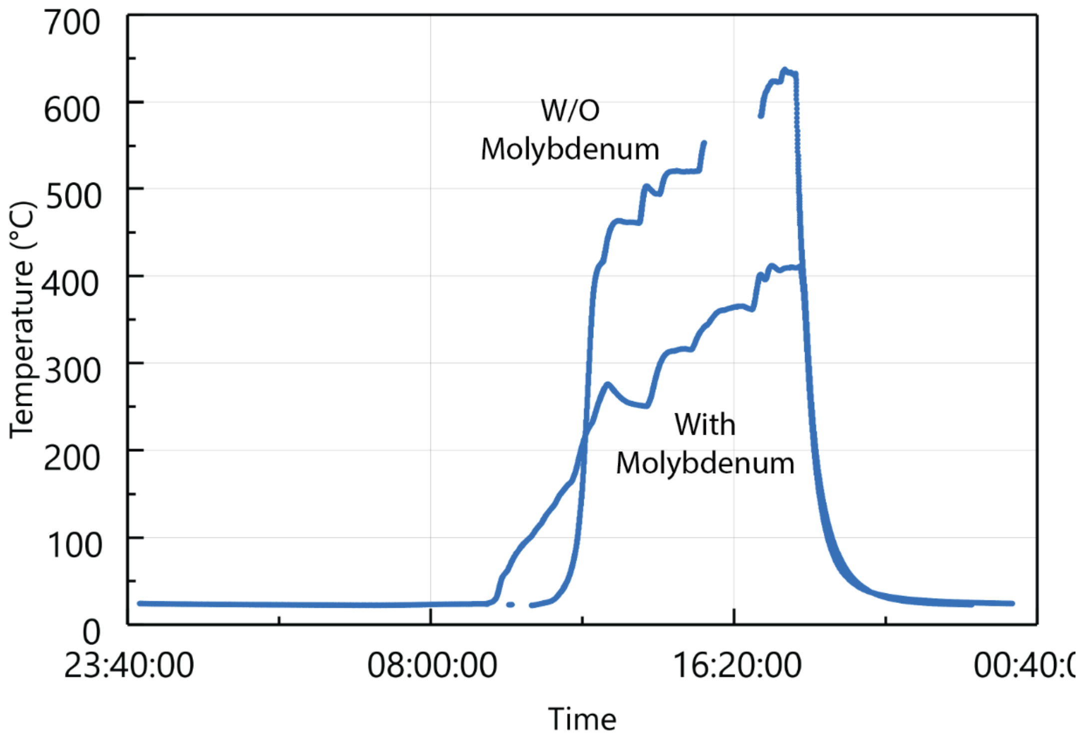


Figure 9: Temperature profiles obtained from the thermocouple attached to the steel plate with and without a molybdenum plate for thermal interfacing with the heater

Further analyses on the behaviour of the whole device are ongoing, considering the electrical performance.

Interpretation of the results (up to 400 words)

[Discuss the data obtained and describe the major scientific conclusions drawn.]

- The Tetrahedrite material is stable in the temperature range of 30-500 °C. However, a point to be noted is the time the material is held at each temperature. In the present study, the samples were held at each temperature for 3 min. This is much less than the general in-service time of these materials. The Magnesium Antimonide (Mg_3Sb_2) sample could not be measured as no Raman spectrum was observed even at room temperature.

- Spatial variation in resistances across the cross-section of MgSb is observed, pointing towards the material heterogeneity. Even within the same region, regions with high and low resistances can be observed. This can be due to multiple reasons, like anisotropy in material property imparted during sintering, due to surface roughness, or formation of conductive or non-conductive phases during sintering. Local variations in electrical resistance noticed between different regions, region 1, 2, and 3, can be due to the diffusion of elements during sintering or the oxidation of the material caused by the prior heat treatment.

- The thermal conductivity of the material measured by Raman spectroscopy is 24 W/mK. This is much higher than the value of thermal conductivity of bulk SiGe with similar composition reported in the previous literature. Generally, the values reported in the literature for SiGe are obtained with laser flash analysis. The most probable reason for the high conductivity values measured with Raman spectroscopy is the measurement geometry. In the case of laser flash analysis, the heat flow is perpendicular to the wafer surface, offering maximum resistance to heat transport. This enables the averaging of the thermal conductivity. However, in the case of Raman spectroscopy, the heat spreads radially to the surface, and a Gaussian distribution is assumed. This heat is localized and happens in a very small region in the sample. Another possible reason is that in the Raman method, the laser spot might be positioned on a region that is a single, large grain. The measurement is more indicative of the thermal conductivity within the grain rather than the bulk average.

- The evaluation of thermal interfacing of the hot side of the cascaded TEG with the heater evidences the higher temperature achieved when a molybdenum plate is used. This can be associated with the material thermal resistance and high contact thermal resistance of the molybdenum plate in contact with the steel plate. It can also be observed that there is an offset between the temperature profile obtained with and without the molybdenum plate. Though the highest power setting reaches temperatures of 630 °C when Molybdenum late is not used, the temperatures at the hot side are much less than the temperature region in which SiGe offers the highest efficiency.

Main achievements during the TA related work (up to 250 words)

[Describe the main achievements during your stay at the site(s), Outputs (results, publications, models, etc.), conclusions, next steps, potential impact]

- The thermal stability of Tetrahedrite was characterized in the 30-500 °C temperature range. It was found that TH is stable in the evaluated temperature range. It was determined that the Raman spectrum of the Mg_3Sb_2 sample could not be obtained, indicating a limitation of the Raman method for this specific material under the tested conditions.

- Spatial variation in electrical resistance was measured and observed across the cross-section of an MgSb sample, demonstrating material heterogeneity.

- The thermal conductivity of a SiGe sample was measured using Raman spectroscopy, yielding a value of 24 W/mK. This included preparing a calibration curve and understanding the significant difference when compared to LFA results from previous literature.

- A Cascaded TEG was prepared with a high temperature module and three low temperature modules of Bismuth Telluride. The performance of a thermal interface at the hot side of the cascaded TEG was evaluated, specifically comparing the use of a molybdenum plate to the case when no molybdenum plate is used. It was determined that not using a molybdenum plate led to higher temperatures being achieved at the interface, likely due to its contact resistance with the steel plate. It was concluded that despite reaching high temperatures, the hot-side temperature of the TEG without the molybdenum plate was still below the optimal efficiency range for the SiGe material used. This is a critical finding for device design.

Data Management

[Describe the further usage and storage of project data. State where the data will be kept and name a person responsible for the data. Define data]

The project data will be stored at RGS development and can be made available on request.

Difficulties during the TA related work (up to 250 words)

[List problems and issues, you had, completing out your research project: Did you get access to all the necessary equipment, facilities, databases, etc.?
If not, please specify the problems that occurred and list the equipment was not working or accessible.]

The Research facilities were readily available and accessible. The people in charge of the equipment were very helpful and proactive. A couple of small issues that we faced during the trials were the following :

- SiGe bonded samples to be evaluated under SPM could not be mounted due to the geometry and large size of the sample
- Mg3Sb2 sample, when evaluated with Raman spectroscopy, did not show any relevant spectra on an oxidized and unoxidized surface.

Intended publications

[Explain where and how you expect to publish the outcomes of your project work. Include also anything already published (What and where?)]

The outcomes of the project work require further work for validation. After the additional work, the results are intended for publication in a conference proceeding.

Expected impact

[The impact the expected results will have on current and future research or practice, public safety, European standardization, competitiveness, integration and cohesion and on sustainable growth. any follow on proposals, projects, collaborations, commercialisation]

A deep understanding of thermoelectric materials' physical, chemical, and mechanical properties is fundamental to overcoming the current limitations of TEGs and unlocking their full potential for sustainable energy solutions, particularly in waste heat recovery across various sectors (industrial, automotive, residential) and for remote power generation. The present study focuses on different thermoelectric materials used in different temperature ranges.

The characterization of TH and $Mg3Sb2$ material helps in understanding the stability and electrical resistance corresponding to the thermoelectric material for mid-temperature Thermoelectric generator applications.

The thermal conductivity measurements with Raman spectroscopy of SiGe help in finding an accessible alternative method for thermal conductivity measurement of thermoelectric material suitable for high-temperature thermoelectric generator applications. Thermal conductivity is one of the most important thermal properties of these materials, which affects the performance and working of TEGs.

The evaluation of thermal interfacing in Cascaded modules helps in maximizing the Temperature Difference (ΔT) across the module. In case of good thermal interfacing, heat loss at the interface between the heat source and TEG is minimized, ensuring more heat reaches the TEG, thus maximizing the ΔT for power generation. This, in turn, can lead to higher electrical output.

Conclusions / additional comments

[Provide any other comments you might have on your work]

Did you complete the European Commission User questionnaire https://ec.europa.eu/eusurvey/runner/RIsurveyUSERS?

☑*Yes* ☐ *No*

Feedback – HSE, Ethics and Satisfaction

Please rate on a scale from 1 (excellent) to 5 (poor). Feel free to provide additional comments

Practical information on how to apply for Transnational Access and the overall application process	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☑	☐	☐	☐	☐
Comment					
Information provided, once your project was accepted, on how to proceed	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☑	☐	☐	☐	☐
Comment					

Support received at the site(s) regarding technical/scientific matters and logistics	Have you got sufficient support from the RI staff during the project? If not, please, specify the problems. ☑ *Yes* ☐ *No*
Please specify any problems	
RI extension / upgrades required	In your opinion, is the RI needed to be upgraded? If yes, please give an explanation. ☐ *Yes* ☑ *No*
Please specify	
Problems with local regulations	Have you had any problems with regulations of the visited RI owner (HSE, lab working hours, etc.)? If yes, please, specify ☐ *Yes* ☑ *No*
Please specify	
Health and safety issues	Did you encounter any health or safety issue during your research? Please provide details. ☐ *Yes* ☑ *No*
Please provide details	
Environment & Ethics	Did your research involve the use of elements that may cause harm to the environment, to animals or plants? Please provide details. ☐ *Yes* ☑ *No*
Please provide details	

<table>
<tr><td>Environment & Ethics</td><td>Did your research deal with endangered fauna and/or flora and/or protected areas? Please provide details.
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Environment & Ethics</td><td>Did your research involve the use of elements that may cause harm to humans, including research staff? Please provide details.
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Environment & Ethics – Dual use</td><td>Does your research have the potential for military applications? Please provide details.
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Environment & Ethics – Misuse</td><td>Does your research have the potential for malevolent /criminal/terrorist abuse? Please provide details.
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Environmental issues</td><td>Were any potentially dangerous substances (materials / gases etc.) released into the environment (atmosphere, water, or land)? Please provide details.
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Ethics issues</td><td>Are there any other ethics issues that should be taken into consideration? Please specify
☐ Yes ☑ No</td></tr>
<tr><td colspan="2">Please provide details</td></tr>
<tr><td>Overall impression of communication and interaction after finishing your TA and related work</td><td><table><tr><td>1 (excellent)</td><td>2</td><td>3 (neutral)</td><td>4</td><td>5 (poor)</td></tr><tr><td>☑</td><td>☐</td><td>☐</td><td>☐</td><td>☐</td></tr></table></td></tr>
<tr><td colspan="2">Comment</td></tr>
<tr><td colspan="2">Suggestions for facilities not included in RISEnergy which you would use for your research</td></tr>
<tr><td colspan="2">[Please provide suggestions for specific type of facilities missing (RI gaps) or measurement / experiments you would like to perform which cannot be done on current RISEnergy facilities.]</td></tr>
</table>

Measurement setups for High temperature material properties like Thermal conductivity, Specific heat, Seebeck, and Electrical resistivity Mechanical properties like hardness, tensile strength and nano indentation up to temperatures of 1000 °C.
Suggestions how RISEnergy can improve future TA programme, how to make the TA more impactful and how to enable the achievement of high TRL levels
[Your suggestions]

Feedback – Pro-active Innovation Support

Awareness	Did you know about the pro-active innovation support of RISEnergy? ☐ Yes ☒ No
[Please specify how you learned about the pro-active innovation support]	
Personal experience	Have you taken advantage of or benefited from the pro-active innovation support? ☐ Yes ☒ No
[*Please provide details*]	

Information/service provided by the pro-active innovation support?	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☐	☐	☒	☐	☐
[*Please provide details*]					

I declare that the above provided information and especially that information on the number of days visited the RI is correct.

☒ *I have read the RISEnergy privacy policy for participation in the RISEnergy TA and consent to participation and the associated data processing.*