THE 3 OMEGA TRANSIENT LINE METHOD FOR THERMAL CHARACTERIZATION OF SUPERINSULATOR MATERIALS DEVELOPED FOR SPACECRAFT THERMAL CONTROL

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The 3 Omega Method is a form of the transient line method for thermal characterization that has been in limited use since 1912. Through sinusoidal heating of a thin copper wire located on the specimen and varying the input frequency, a third harmonic voltage arises, the frequency dependence of which carries information about the thermal conductivity and diffusivity of the sample being tested. Thus the heating wire serves also as the sensing element in this method. Major advantages of this approach include very small temperature oscillations, on the order of a few degrees or less, significantly reducing errors arising from thermal radiation and gaseous convection that plague more traditional methods which typically require very large temperature gradients for low conducting materials. Additionally, in this method, thermal conductivity and diffusivity are extracted from the measurement simultaneously based on first principles, eliminating the need for complex and unreliable thermal modelling and calibration of the measurement system. The implementation of this method has been significantly facilitated and its sensitivity and dynamic range improved by the advancement of modern technology, especially the development of lock-in amplifiers and subsequently digital signal processing. Traditionally, this method requires fabrication of micro heating elements through vapour deposition of copper on the specimen to be tested. This method has been implemented at Active Space Technologies GmbH for testing aerogels developed in the context of the AerSUS - Aerogel European Supplying Unit for Space Applications – project FP7 (contract no: 284494), an initiative funded by the Seventh Framework Programme of the European Union. To the best of our knowledge, this is the first time that it has been implemented using macroscopic (60 micro-meter diameter) mass produced copper wire, significantly reducing sample preparation cost and time. Furthermore, this work represents the first use, to our knowledge, of digital signal processing techniques, namely discrete Fourier powers, to extract the third harmonic signal, eliminating the need for large time constant analog circuitry, and making feasible the extremely low frequencies (down to 0.001 Hz) which are required for thermal characterization of super-insulators. We present the implementation of this method, a comparison of results for known materials from the literature and thermal characterizations of super-insulators for space applications. Additionally, simulations and analysis are presented to showcase the use of such super-insulating materials developed by the AerSUS consortium in a realistic Mars lander scenario.

I. THE 3-OMEGA TECHNIQUE

Originally, the 3-omega (3ω) technique was developed to measure the thermal diffusivity of metal filaments used in incandescent light bulbs (Corbino 1912). Later, the method was used to measure the thermal diffusivity of liquids (Birge and Nagel 1987) and solids (Moon et al. 1996) by indirectly heating the specimen using a thin planar metal filament as both a heater and thermometer in thermal contact with the solid of interest. The first reported use of the 3ω method to measure the thermal conductivity of solids was by Cahill (Cahill and Pohl 1987). Cahill's 3ω technique utilizes a microfabricated metal line deposited on the specimen to act as a heater/thermometer. When an alternating current (AC) voltage signal is used to excite the heater at a frequency ω, the periodic heating generates oscillations in the electrical resistance of the metal line at the heater frequency of 2ω. Since voltage is given as the product of the current and the resistance of the wire, $V_0 = R_0 I_0$, multiplication of the sinusoidal current at the input frequency ω by the heater resistance fluctuations at 2ω results in sum and difference voltage signal at the first and third harmonics (1 and 3ω), which is used to infer the magnitude of the temperature oscillations (Corbino 1911). Comparing the input RMS power per unit heater length

$$p_{rms} = \frac{V_0^2}{2lR_0}$$

to the frequency dependent voltage oscillations, the amplitude and phase can be analysed to obtain the thermal conductivity and diffusivity of the specimen (Cahill and Pohl 1987, de Koninck 2008).

II. THEORETICAL FORMULATION OF THE 3 OMEGA METHOD

The implementation of the 3ω method involves either fixing a wire heater/temperature sensor on the specimen or embedding it within the specimen. An alternating current of peak intensity I_0 at an angular frequency ω is applied to the wire of initial resistance R_0 is given by:

$$I(t) = I_0 cos(\omega t)$$

resulting in a thermal wave with penetration depth defined as:

$$\lambda = \sqrt{\frac{\alpha}{2\omega}}$$

where α is the thermal diffusivity of the specimen. Then the power dissipated in the wire can be described as the sum of a direct current (DC) component

$$P_{DC} = \frac{1}{2} I_0^2 R_0$$

and an alternating instantaneous current (AC) component

$$P_{AC}(t) = \frac{1}{2} I_0^2 R_0 \cos(2\omega t)$$

Given the radial symmetry of a line heat source embedded in a surrounding solid, cylindrical coordinates provide the most natural representation, where the Fourier heat equation takes the form

$$\frac{\partial^2 \varphi(r,t)}{\partial^2 r} + \frac{1}{r}\frac{\partial \varphi(r,t)}{\partial r} = \frac{1}{\alpha}\frac{\partial \varphi(r,t)}{\partial t}$$

Where $\varphi(r,t)$ is the specimen temperature profile as a function of radius r from the heater wire and time, t. Solving this partial differential equation under the assumption that the thermal penetration is much greater than the wire width, $\lambda \gg b_h$, de Koninck then derives the amplitude of the frequency dependent temperature oscillations in the wire as:

$$\begin{aligned}\Delta T_{AC}(\omega) &= -\frac{p_{rms}}{2\pi k}\int_0^{1/b_h} \frac{1}{\sqrt{\eta^2 + q^2}}\, d\eta \\ &\approx -\frac{p_{rms}}{2\pi k}\left(\ln(2\omega) + \ln\left(\frac{{b_h}^2}{\alpha}\right) - 2\zeta\right) - i\frac{p_{rms}}{4k},\end{aligned}$$

where k is the thermal conductivity, α is the diffusivity; de Koninck states that ζ is a fitting constant with a value of ≈ 0.922. This is in fact not correct, since the above integral is exactly solvable without approximation using trigonometric substitution yielding the correct answer:

$$\begin{aligned}\Delta T_{AC}(\omega) &= -\frac{p_{rms}}{2\pi k}\int_0^{1/b_h} \frac{1}{\sqrt{\eta^2 + q^2}}\, d\eta \\ &= -\frac{p_{rms}}{2\pi k}\left(\ln\left|\sec\left[\tan^{-1}\frac{\sqrt{\alpha}}{b_h\sqrt{i2\omega}}\right] + \frac{\sqrt{\alpha}}{b_h\sqrt{i2\omega}}\right|\right)\end{aligned}$$

Where b_h is the radius of the heating wire. In the low frequency limit, $b_h\sqrt{\frac{2\omega}{\alpha}} = {b_h}/{\lambda} \ll 1$, this can be approximated as:

$$\Delta T_{AC}(\omega) \approx -\frac{p_{rms}}{4\pi k}\left(\ln(2\omega) + \ln\left(\frac{{b_h}^2}{\alpha}\right) - 2\ln 2 + 2\Upsilon\right) - i\frac{p_{rms}}{8k}$$

Where Υ is the Euler–Mascheroni constant. This provides the separation of the real, or in-phase, component of the temperature oscillations at the third harmonic, and the imaginary, or out-phase, component. So the "fitting constant" should be just a constant with a value of $\ln 2 \approx 0.69$ instead of 0.922. The correct value here is important for determining the diffusivity but does not affect the measurement of the thermal conductivity.

These temperature oscillations result in oscillating resistance given by $R_0\beta\Delta T_{AC}$, where β is the temperature coefficient of resistance, and, therefore, under current source $I(t)$, a superimposed voltage oscillation at the third harmonic arises given by:

$$V_{3\omega} = \frac{1}{2}V_0\beta\Delta T_{AC} \approx -\frac{V_0^3\beta}{8\pi k l R_0}\left(\ln(2\omega) + \ln\left(\frac{{b_h}^2}{\alpha}\right) - 2\ln 2 + 2\Upsilon\right) - i\frac{V_0^3\beta}{16klR_0}$$

(Cahill and Pohl, 1987). The first term is the real, or in-phase, component of the third harmonic voltage. It is log-linear in the input frequency, where the slope of the line is dependent on the conductivity, and the y-intercept depends on the diffusivity. The second term, the imaginary, or out-phase, component, on the other hand, is constant in frequency and depends only on the conductivity. Therefore, by measuring the frequency dependence of the in-phase and out-phase components of the third harmonic voltage, the thermal conductivity and diffusivity can be determined simultaneously.

In the low frequency limit, where the log-linear approximation applies, the thermal penetration of the heat wave is much greater than the width of the heater wire, i.e. $\lambda \gg b_h$. To be sure that we are in the "linear region", we take $\lambda > 5b_h$.

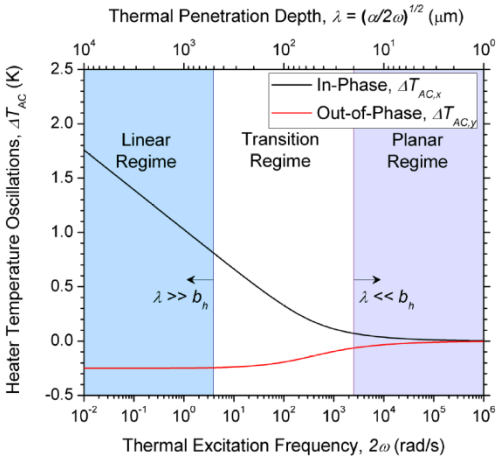


Fig. 1 – The in-phase and out-phase components of the 3ω temperature oscillations. In the low frequency limit, below the transition regime, the linear approximation may be used (taken from de Koninck, 2008).

III. EXPERIMENTAL SETUP

Samples, consisting of two pieces of at least 7cm were placed in a dish (Fig. 2). A copper heater wire of diameter 60 micrometers is placed between the two pieces. Low resistance lead-wires are soldered to the ends of the heater wire in pairs to allow for 4-wire resistance measurements for high precision resistance determination.

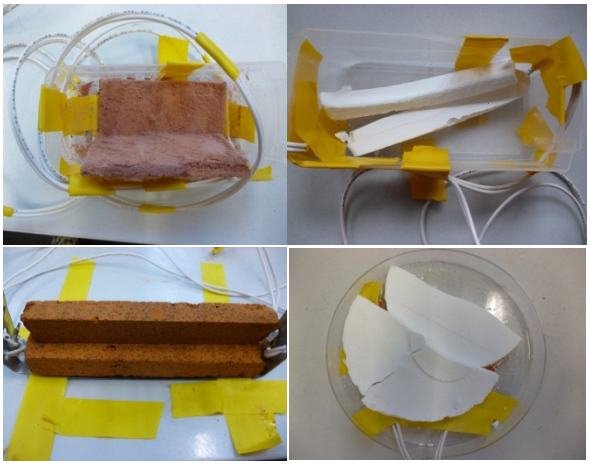


Fig. 2 – Aerogel samples tested as part of the AerSUS project

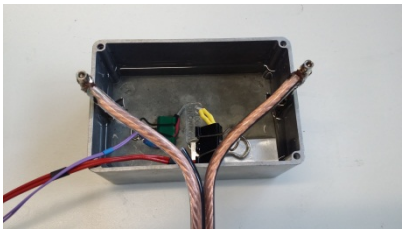


Fig. 3 – A first prototype test bed was also developed for measurements on liquids and phase change materials

The 3Omega measurement device first prototype developed at Active Space Technologies is shown in Fig. 4. This includes a National Instruments voltage input and output for driving the instrument and data acquisition. A next generation device is currently under development with partners with the design objective to accelerate, automate and simplify the measurement process.

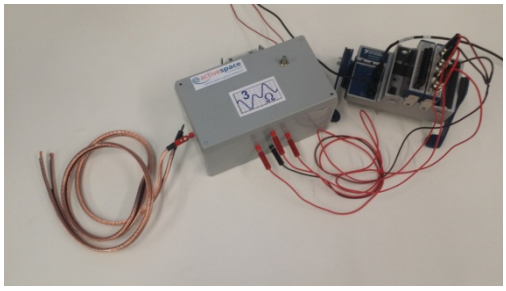


Fig. 4 – The 3Omega measurement device prototype developed at AST including NI instrumentation

IV. MEASUREMENT RESULTS AND ANALYSIS DATA

The voltage read across the heating wire using a Wheatstone bridge is dominated by the first harmonic voltage, i.e. the input heating voltage. Subtracting the voltage read on the other leg of the bridge removes most of the first harmonic as well as common mode harmonic distortion. Finally, the 3rd harmonic in-phase and out-phase components are determined from a Fourier transform which effectively removes noise at other frequencies. This in-phase component is measured as a function of frequency to determine the conductivity and diffusivity of the sample as shown in Fig. 5, Fig**. 6** and Fig**. 7**.

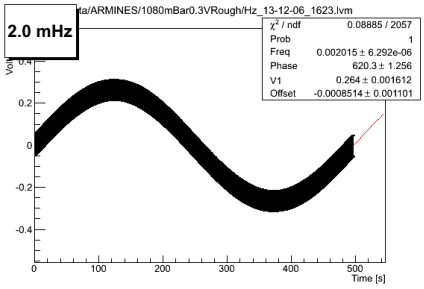


Fig. 5 – Heater wire voltage reading, dominated by 1st harmonic.

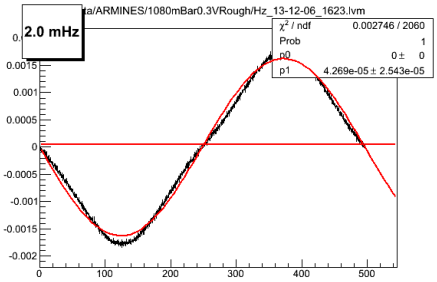


Fig. 6 – Voltage across the Wheatstone bridge removes most of the 1st harmonic as well as common mode harmonic distortion.

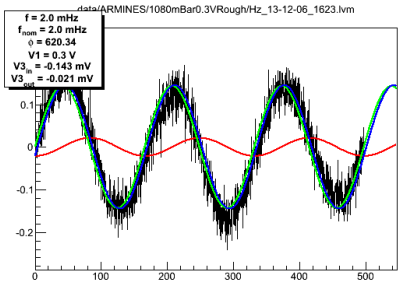


Fig. 7 – 3rd harmonic (0.006 Hz) voltage after subtracting the remaining 1st harmonic (input frequency of 0.002 Hz) power from Fig. 6

In Fig. **7** green shows the in-phase Fourier power of the 3rd harmonic, red shows the out-phase Fourier power and blue is the combined signal, showing a nearly pure 3rd harmonic signal. The frequency dependence of the power of the in-phase component yields the thermal conductivity and diffusivity of the tested material, according to the equations given above.

Analysis and Corrections

Small corrections for the resistance of the connecting wires are required. The resistance of the heating wire and connecting wires are determined separately using a 4-wire resistance measurement with mΩ accuracy which can then be efficiently removed from the measurement. Additionally, the precise width of the heater wire was not originally known. The manufacturer stated the diameter to be 60 microns. Laser diffraction was later used to more precisely determine the diameter of the wire to 65 ± 2 microns. The square of this value is proportional to the determined diffusivity, so accurate knowledge of the wire width is necessary. It is not currently known how constant the wire width is across the length of the spool.

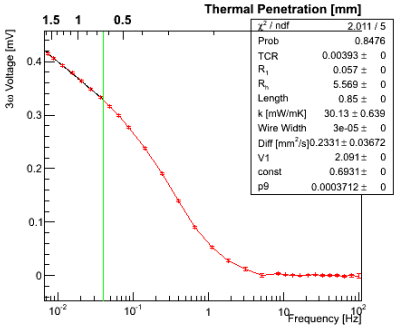


Fig. 8 – An example of the in-phase third harmonic power from a frequency sweep of a Rohacell sample.

In Fig. **8** above 10 Hz is the "planar" region, below 40 mHz is the log-linear region which is used to determine the thermal conductivity and diffusivity of the specimen. Between these two is the "transition" region. Notice the similarity to the theoretically predicted curve depicted in Fig. 1.

Error Estimations

The uncertainties in the measurement system come from statistical errors, which can be reduced by repeating the measurement or increasing the measurement time, and systematic errors which are not reduced by extended measurements. Statistical errors originate in the voltage reading which has a random noise on the level of 50 microvolts per voltage reading. Taking a large number of measurements reduces the uncertainty. The systematic errors originate in uncertainties in values which are read once, i.e. the resistance, length and width of the heater wire. Estimates on the uncertainties in the heater wire resistance, length and width are assumed to be Gaussian and 1,000 random variations of these parameters are simulated to determine the total systematic uncertainty for each measurement. Propagation of uncertainties is used to find the combined final measurement uncertainties.

V. TESTS ON KNOWN MATERIALS

The 3 Omega method, as implemented by AST-DE, was tested on several materials with known properties for verification. First, a sample of Rohacell which was tested by the Physikalische Technische Bundesanstalt in Germany, with measured conductivity of 32 mW/m·K ± 10% and diffusivity of 0.26 mm^2/s ± 15%. The results of measurements performed at AST are shown in Fig. 9 resulting in measured conductivity of 29.9 mW/m·K ± 4% and diffusivity of 0.22 mm^2/s ± 17%. Increasing the heating power applied to the sample in the measurement process by a factor of 3 resulted in measurement values consistent within a few percent. The high probability of consistency with a constant (>0.05) indicates an over-estimate of the uncertainty in the measurements, such that true uncertainty must be lower. In Fig. 9 green lines show the nominal values for the sample.

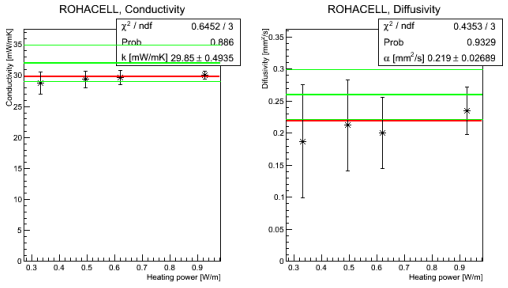


Fig. 9 – Thermal conductivity and diffusivity measurement of Rohacell performed at AST-DE.

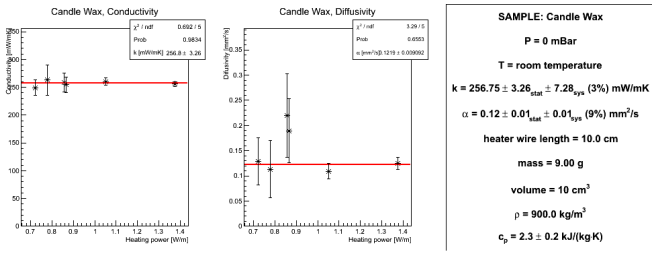


Fig. 10 – Thermal characterization of candle wax using the 3 Omega instrumentation implemented at AST-DE.

VI. AEROGEL MEASUREMENTS

A total of four samples were tested, samples A131 and A132 supplied by ARMINES / MINES ParisTech / PERSEE, sample 396 supplied by the University of Coimbra and RC1500-020 supplied by DLR. The samples were tested at atmospheric pressure and at 10 mbar vacuum pressure, not controlling for the temperature (lab temperature ca. 20-23°C).

The measurements taken at 10 mbar are used for the simulations of the Martian lander application, with conductivity values obtained in the range of 16 to 23 mW/m·K. At atmospheric pressure, values were obtained ranging from 29 to 42 mW/m·K.

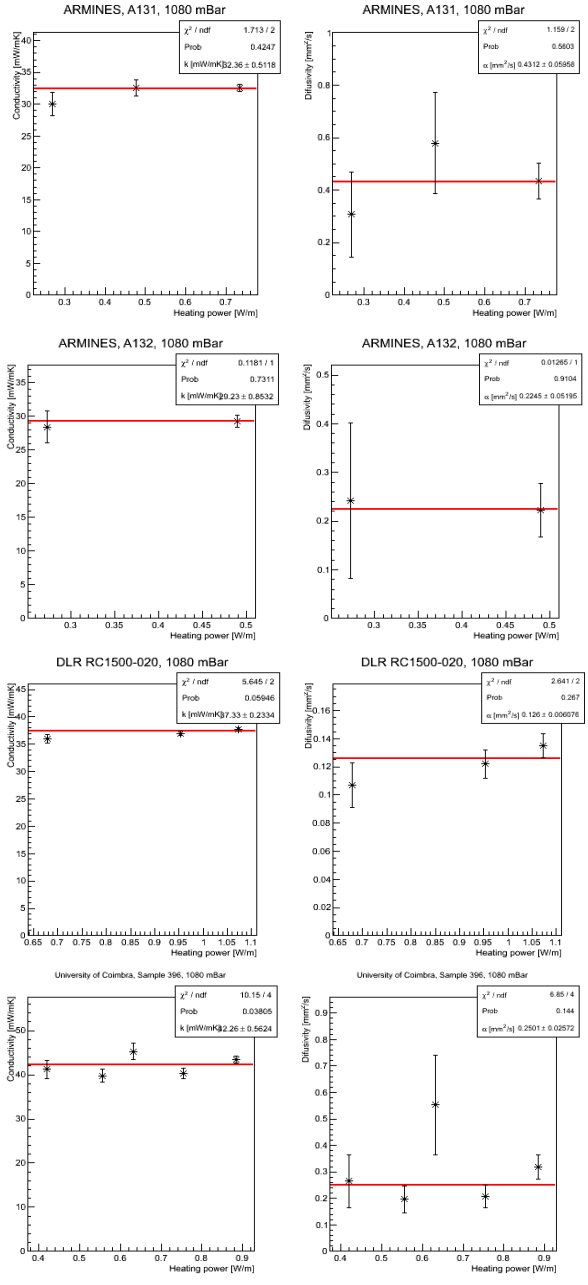


Fig. 11 – Measurements at atmospheric pressure

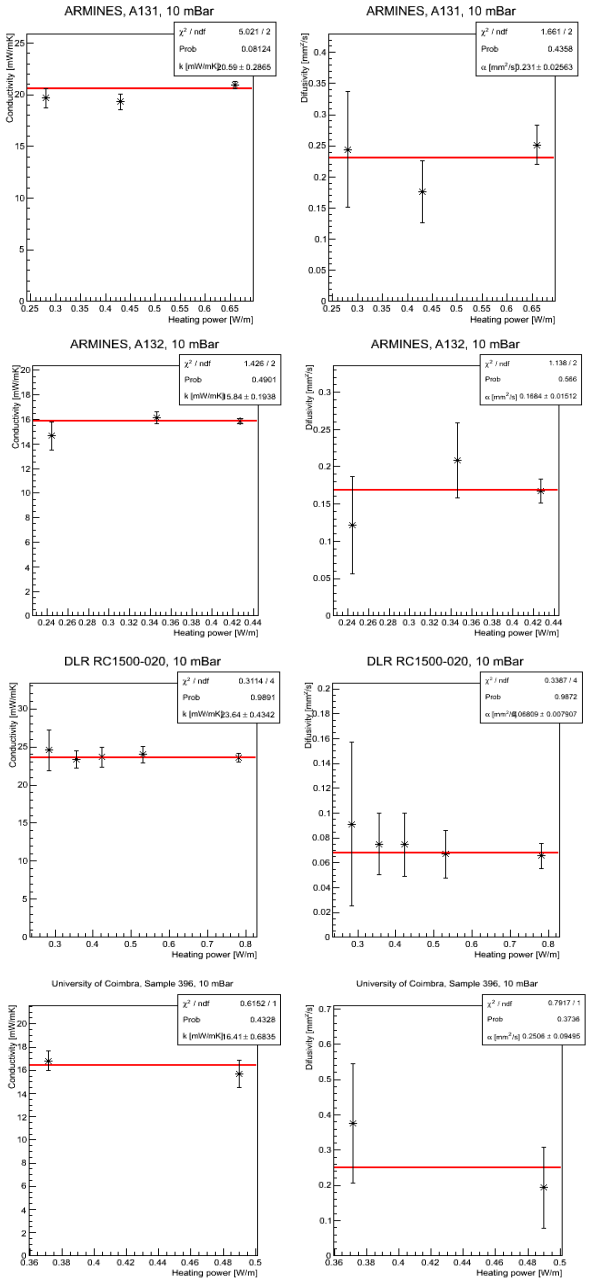


Fig. 12 –Measurements at 10 mbar vacuum pressure

VII. SIMULATIONS OF AEROGEL INSULATION IN REALISTIC APPLICATION

In order to do a first assessment of the performances of the aerogels for a realistic application, Active Space Technologies performed a preliminary analysis. The selected application is, taking into account the target applications, a mission to Mars, with an instrument (part of a larger lander) on its surface. Low atmosphere environments are considered one of the most promising applications for aerogels, given the poor performances of MLI in cases with presence of atmosphere.

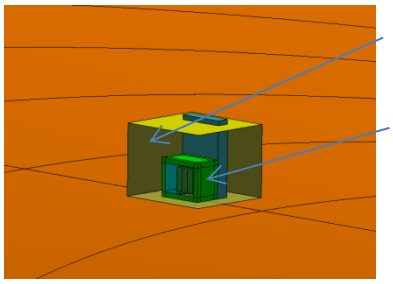


Fig. 13 – General view of AERSUS instrument on the ground

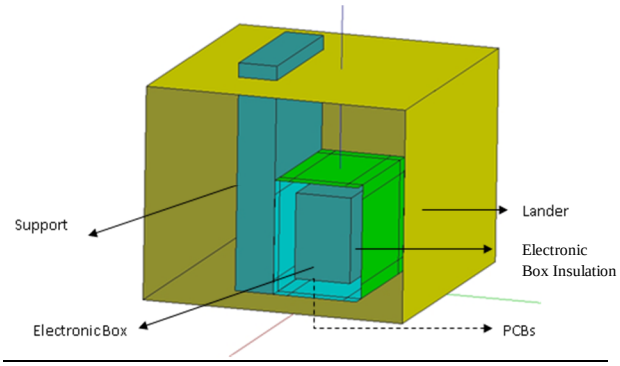


Fig. 14 – AERSUS Instrument Components

The above presented simulation is used to size the insulation layer of the internal Ebox with aerogel, in order to minimize the heaters power consumption during the night and to reach the switch-ON temperature of the Ebox and keep the maximum temperature below the maximum functional value for the internal boards.

The materials tested have a wide range of properties, with all of them (density, specific heat or conductivity…) showing very different values, meaning that already quite different results in the simulation can be foreseen. The differences in terms of thermal conductivity, specific heat, etc. are quite large, in particular considering together the specific heat and the density (volumetric heat capacity).

Table 1 – Aerogels materials used for the simulation

Insulation material	Density [kg/m^3]	Specific heat capacity [J/kgK]	Volumetric heat capacity [J/m^{3}K]	Conductivity [W/mK]
DLR RC1500-020	203.2	1700	345440	0.02364
ARMINES A131	97.4	915	89121	0.02059
ARMINES A132	37.3	2500	93250	0.01584
UC396	69.4	943	65444	0.01641

The strongest differences observed are between the first prototype (DLR) and the others, since the way in which these different materials are synthetized by the partners is different due to different manufacturing processes and different target material characteristics and applications.

These large differences have as a consequence that the materials evaluated via the currently used model can be quite hard to be compared between each material type. Moreover, a possible outcome of the analysis could be that different materials are more suitable for different applications than others.

It must be highlighted that the simulation has two separated objectives to be fulfilled by the optimal insulation thickness under evaluation:

1. To minimize the heating power during night and dawn periods;
2. To keep the maximum operating temperature of the PCBs below the maximum limits during operations.

Table 2 – Results of simulations on tested aerogels

Insulation	Thickness [mm]	Density [kg/m^3]	Insulation Volume [mm^3]	Mass [g]	Energy [kJ]
DLR RC1500-020	10	203.2	453000	92.05	5.90
ARMINES A131	7	97.4	309183	30.11	7.37
ARMINES A132	6	37.3	262776	9.80	7.37
UC396	6	69.4	262776	18.24	7.37

The performances of the various aerogel samples in realistic simulations of the selected Mars Lander

application are summarized in Table 1. As expected from a comparison between the physical properties of the different materials analysed, the results vary significantly. This can be noticed in particular in the overall insulation mass and the heating power necessary in order to switch ON the Ebox (reaching the minimum temperature requirement).

For this application case, the best mass saving material is ARMINES A132, while the best heating power saving material is the DLR Aerogel. One overall best material for this application cannot be identified. The selection of a proper material for a particular case can only be evaluated taking into account the specific mission requirements. In particular different materials can provide the best mass saving configuration (ARMINES A132), best heat power saving configuration (DLR RC1500-020), or the smallest insulation thickness (UC396 or ARMINES A131). The simulation model allows the evaluation of the insulation material for a specific application and for different optimization parameters and materials selection can thus be tailored to mission constraints such as power consumption, mass budget, operating temperatures, etc.

VIII. CONCLUSIONS

These tests show the success of the 3Omega method, showing good agreement with measurements using more traditional methods, but achieving total measurement uncertainty, including statistical and systematic errors, much lower. For the thermal conductivity, uncertainties as low as a few per cent are achieved and uncertainties on the diffusivity as low as 7 - 10% are possible. Relative errors, in general, can be reduced by using a longer sample (and heating wire), thus making the errors in its length and resistance negligible, which can be measured to an accuracy of about 0.3 mm and 0.001 Ω respectively. The main source of systematic error on the diffusivity is poor knowledge of the diameter of the heating wire, which is stated by the manufacturer to be 0.06 mm. Our own measurements using laser diffraction found the wire diameter of 0.065 ± 0.002 mm. Future plans to use laser diffraction to more precisely determine the width of the wire and ensure consistency along its length should reduce this uncertainty. Additionally, precise resistance and length measurements could be used to determine the average width, given the standard resistivity of copper.

These tests show excellent agreement with other measurements over a wide dynamic range of conductivities and diffusivities, with repeatability on the order of a few per cent even while changing the applied heating power by a factor of 2, 3 or even 4 without the need for thermal models to correct for convection losses, radiation losses or rising temperature of the sample. Additionally, the minimum sample size is significantly smaller with this method than with many other measurement techniques, requiring as little as $10\,cm^3$ of material, which can represent significant savings for materials researchers and developers, where manufacturing of new materials in minute quantities can be very costly. Sample preparation is shown to be simple and low cost with this implementation of the 3Omega method and, since measurements can be performed in 20 minutes or less, multiple measurements can easily be performed rapidly to reduce statistical uncertainty. These tests demonstrate the success and advantages of this technique as a measurement method, especially for very low conductivity samples. Future R&D efforts planned by AST in this measurement system intend to further simplify and accelerate the measurement process and reduce minimum sample size to ~1 cm, as well as prove the usability and advantages of this system in extreme testing environments, such as at 20K in cryogenic chambers.

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