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Thermophysical Property Measurement: A Call to Action

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Abstract

Thermophysical property measurement using levitation processing in space, while expensive, has the potential for providing benchmark datasets of highest quality. Unfortunately, much of the previous historical record concentrates on data accuracy without adequately addressing precision making it hard to justify the added costs associated with developing and using microgravity facilities. Furthermore, analysis of measurement precision in the literature often does not fully distinguish the magnitude of relative contributions from both systematic and random error. This paper presents a brief review of the status of measurements for a subset of key properties that are appropriate for space investigation. From this, it is hoped that discussions are stimulated across the property measurement community to initiate development of reporting standards to better document relative variability for specific test platforms, both ground and space, to allow industry to better define the value-added for sponsoring space experimentation.

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1. Introduction

Materials Informatics has become a hot discussion topic within the Microgravity Physical Sciences Community. Across the globe, roadmaps guiding allocation of funds to sponsor scientific investigation have included material property measurement as a priority. The US Materials Genome Initiative proposed a national infrastructure for data sharing and analysis that would facilitate materials innovation.

The experimental input required goes far beyond a single set of measurements. In most cases, researchers must combine and calibrate data from many experiments into a single larger data set that represents the entire system and allows the determination of complex properties.¹⁾

From this, the US National Aeronautics and Space Administration (NASA) convened a cross-disciplinary group to develop a MaterialsLab Informatics document²⁾ to guide science prioritization for space materials investigations. In parallel, the European Space Agency (ESA) produced a Materials Science Roadmap³⁾ for the same purpose. Similar discussions are ongoing at the Japanese Space Agency (JAXA), the Russian Space Agencey (ROSCOSMOS), the Chinese National Space Administration (CNSA)⁴⁾, the German Space Agency (DLR) and the Korea Research Institute of Standards and Science (KRISS). The common theme is to highlight and prioritize key scientific topics and fundamental questions that guide the direction of future research initiatives.

The purpose of this paper is to outline the first steps in an international collaborative effort to promote data sharing and standardizing analysis protocol in the property measurement field. The key descriptors are collaboration and international. Collaboration is important because property measurement, especially in space, is expensive and use of multi-user facilities is cost-effective in order to leverage complementary science through efficient use of precious commodities such as crew time, telemetry, and access to space. The more eyes on a problem, the greater the gain, the better the synergy between related fields - now and in the future. Networks of scientists are built and future research initiatives hatched. International is important because common problems and transnational goals will become identified through sharing of facilities and analysis tools. Development of a scientific community with enhanced communication is a priority for creation of global standards. Ground-based property measurement has codified standards for test methods and analysis techniques but space-based techniques have yet to be addressed.

2. Background

Thermophysical property measurements are conducted in many ways using a broad variety of analysis techniques, experimental protocols, and facilities⁵). Reactive metals and high temperature oxides are often investigated using levitation techniques to minimize melt contamination and biasing of results. A wide variety of material properties may be selected

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for measurement but this paper concentrates on three paired sets of thermophysical properties – density/thermal expansion, surface tension/viscosity, and heat capacity/emissivity. Groundbased testing procedures used to evaluate these properties are not standardized and an extensive and varied array of specialized equipment is used by different research groups. Of central importance is the question – which platform is best for investigating which class of materials and why?

2.1 Density and Thermal Expansion

Molten metal density measurement (and thermal expansion measurement through evaluation of how density changes with temperature) using levitation processing is commonly accomplished using cinematographic techniques where a levitated sample of known mass, m, and temperature, T, is imaged with a digital camera having high spatial resolution. The image is usually back-lit to improve edge contrast when compared to a calibration sample of known diameter. Individual images are manipulated to identify spatial extent of the sample using an edge-fitting protocol and the volume, V, is then obtained by assuming rotational symmetry about the polar axis. Combining these measurements yields the density, ρ , tabulated as mass per unit volume at a given temperature⁶. In a similar manner, the volumetric thermal expansion, β , may be evaluated from a plot of volume as a function of temperature.

Density $\rho = \frac{m}{V}$ (1)

Thermal Expansion
$$\beta = \frac{1}{V} \frac{\partial V}{\partial T}\Big|_{T}$$
 (2)

It is common to obtain a series of measurements during cooling of the sample from superheated to undercooled liquid as seen in Fig. 1



Fig. 1 Density for a FeCo alloy showing the raw shadowimage and subsequent density calculation result –only the high-temperature liquid data is valid.

and density and thermal expansion are obtained simultaneously.

Once the sample is solid the apparent density has severely increased noise due to the non-spherical shape (and often the presence of a central solidification void). Surface deformations due to oscillations or internal flows are to be avoided since nonuniformities in shape create significant random signal noise due to the inherent assumptions required to convert the shadow silhouette to volume. Since it is advantageous to have quiescent conditions, electrostatic levitation (ESL) techniques are commonly employed⁸⁻¹⁰⁾ for ground-based testing. A novel new electromagnetic levitation (EML) technique has also shown promise where surface oscillations and internal flows are suppressed through application of a strong static magnetic field¹¹⁾. Evaporation must be tracked, as seen in Fig. 2, to eliminate the systematic error associated with reductions in overall sample mass^{12),13)}. Ground-based techniques which do not employ levitation include fast-resistive pulse-heating discharge¹⁴⁾ and sessile droplet techniques^{15), 16)}.

2.2 Surface Tension and Viscosity

During levitation, mode 2 surface oscillations with l = 2 can be intentionally imposed on a molten droplet⁷ and the natural



Fig. 2 Temperature profile for isothermal ESL (FeCo shown) and cooling curve EML (FeCrNi shown) tests. Significant evaporation is shown during ESL thermal hold due to the small sample size¹³).

(4)

frequency of these oscillations, f, is used to determine the surface tension given sample mass. In a microgravity environment, the relaxation time for sample damping defines the viscosity, μ , given the droplet radius, R, such that:

Surface Tension
$$\gamma = \frac{3 \pi m f^2}{(l)(l-1)(l+2)}$$
 (3)

Viscosity
$$\mu = \frac{\rho R^2}{(l-1)(2l+1)\tau}$$

The methods for imposing surface deformation are very different when comparing ESL to EML in ground-based tests. In ESL, the positioning field strength is modulated at near to the droplet natural frequency and the magnitude of the axial deformation increases slowly with time until motion is clearly visible; this excitation is then stopped in order to obtain a clear measure of oscillation damping¹⁷⁾. To maintain sample stability system perturbation must be minimized and it takes significant time for the oscillations to reach the desired amplitude. Thus these tests must be conducted isothermally as shown in the first part of Fig. 2. Because the sample must be continuously heated to counter radiative losses, the orientation of the heating lasers influences the test conditions - for a symmetric arrangement surface gradients are minimized but for an asymmetric arrangement (such as from one side) significant temperature gradients develop which produce surface tension gradients which may induce a moderate amount of stirring due to Marangoni flow (for a spinning sample this would be equator-to-pole with a hot equator and cold poles). Sample size has been shown to influence the measured value for surface tension¹⁸⁾ while the positioning control circuit feedback frequency influences the measurement of viscosity¹⁹. These potential biases to measurement precision are often not documented fully in the literature.

In EML, a sudden pulse to the heater field creates a balanced radial compressive force that does not destabilize the sample and thus the oscillations may be imposed quickly softening the requirement for maintaining an isothermal sample. If a sample is held isothermally the superposition of the magnetic positioning and heating fields creates significant convection and thus it is common to simultaneously minimize both evaporation and stirring by allowing the sample to cool quickly. This is shown in the second part of **Fig. 2** where a sudden pulse using the heater field is used to excite oscillations at the sample melting temperature. In the figure, cooling is slowed by imposing a moderate heater control voltage. Oscillation damping is shown in the first part of **Fig. 3** and the decay of the deformation decay is used to obtain viscosity while the frequency is used to obtain surface tension.

EML samples are larger than ESL samples, on the order of 1 gram and 50 milligrams, respectively, and thus the influence of sample evaporation is reduced. Due to differences in



Fig. 3 Characteristics of Sample Oscillation (a) viscous damping of sample deformation amplitude¹⁷ and (b) mode splitting from a Fourier transform of the signal frequency⁵ for AuCu.

sample mass, the natural oscillation frequency for a given material will be very different using different facilities – for FeCo alloys on the order of 35 Hz and 200 Hz for EML and ESL, respectively, – thus requiring different data acquisition frequencies to capture the full extent of sample deformation.

Different facilities use different means to measure the oscillation magnitude and frequency. High speed cinematography akin to that used for density measurement (up to 1 kHz but nominally 200 Hz) is used by researchers at DLR for ground-based and space-based EML²⁰, in Japan for ground-based EML²¹, ²², and at NASA Marshall Space Flight Center (MSFC) for ground-based ESL²³. Both ground and space-based ESL²⁴, ²⁵ at JAXA use an array of photodiodes arranged in a slit (up to 1 MHz) ²⁶. In contrast, researchers at DLR have pioneered use of an inductive technique known as Sample Coupling Electronics (SCE) ²⁷ that measures oscillation frequency and amplitude by monitoring changes to the EML coil/sample impedance which is a function of sample geometry.

Various researchers have compared ground-based test results to microgravity results with strong ESA and JAXA parabolic aircraft/sounding rocket programs^{25),28-32)}. The influence of gravity on oscillation frequency mode-splitting has been shown to be successfully modeled using the Cummings-Blackburn³³⁾ correlation in surface tension measurement^{18),34-35)}. This phenomenon is seen in the second part of **Fig. 3** where a spherical sample in microgravity has a single mode 2 oscillating frequency while a deformed ground-based sample shows multiple mode excitations due to the aspherical nature of the sample shape; the average frequency is also shifted higher on ground due to the greater magnetic pressure required to overcome gravity and levitate the sample. Competing groundbased viscosity measurement techniques that do not utilize levitation include the Ring Method and Rotating Crucible^{36),37)}.

2.3 Specific Heat and Emissivity

A liquid cooling curve that is similar to that shown in the second part of **Fig. 2** can be used to evaluate the ratio of emissivity, ε , to specific heat, C_p , from evaluation of a simple heat balance given droplet surface area, A, and using the Stefan-Boltzmann constant, $\sigma = 5.67 \times 10^{-8} \text{ W/m}^2 \text{K}^4$ and noting that the rate of change of temperature is negative during cooling. Analysis is significantly simplified if the test is done in vacuum such that conductive and convective losses are ignored and all heat is lost due to radiative cooling. Additionally, heat input, \dot{Q}_{IN} , is often negligible such that:

$$\dot{Q}_{N} - \dot{Q}_{OUT} = \text{Accumulation} - \sigma A \varepsilon \left(T^{4} - T_{env}^{4} \right) = m C_{p} \frac{\partial T}{\partial t}$$
(5)

$$\frac{\varepsilon}{C_p} = \frac{m}{\sigma A} \left(-\frac{\partial T}{\partial t} \right) \left(T^4 - T_{env}^4 \right)^{-1}$$
(6)

Data from cooling curves thus provides information on the linked properties of emissivity and heat capacity and separating these properties is often difficult. Individual resolution of emissivity is usually conducted on ground³⁸). Fukuyama³⁹ has proposed measuring C_p independently using a combined radiation/EML calorimetry technique based on previous successful space EML analysis technique proposed by Fecht and Johnson⁴⁰ which is ongoing as part of the current Material Science Laboratory (MSL-EML) ThermoLab program⁴¹. As seen in **Fig. 4**, by applying an oscillating heat pulse at various oscillation



Fig. 4 Modulation calorimetry thermal profile.

frequencies, the phase lag of the thermal response of the sample is used to evaluate the heat capacity. Two internal relaxation times are accessible as a function of the equilibrium temperature, T_o , and the thermal conductivity, κ , of the sample: the low frequency response, τ_1 , and the high frequency response, τ_2 .

$$\tau_1 = \frac{C_p}{4A\sigma\varepsilon T^3} \tag{7}$$

$$\tau_2 = \frac{3C_p}{4\pi^3 \kappa R} \tag{8}$$

3. Error Analyses

Traditionally, error analysis in thermophysical property reporting has often involved publishing the raw data in graphical form to indicate scatter and reporting some form of linear or curve-fit to describe averaged behavior. Unfortunately the error in slope and intercept for a linear fit (usually either how the property varies as a function of temperature or in the determination of constants from an Arrhenius analysis) are rarely reported. Additionally, researchers assume, without analysis, that error is normally distributed and often report values in the form of μ \pm n σ without indicating the confidence that is associated with the deviation. It is unclear if the data is reported with n = 1 to show the standard deviation (the obvious default) or reported with n = 2 or 3 to show 95 % or 99 % confidence. The relative contribution from random error and systematic error are also often not discussed but remain lumped together as "data scatter". Several recent publications have attempted to reverse this trend by careful and thorough reporting of the individual sources for both random and systematic errors^{8), 18)} and as a community we should strive to improve how data is presented.

In addition to failing to record systematic errors, many researchers compare their recent measurements to those in the literature only in terms of the data accuracy without addressing relative precision. A common theme is "*these results (do/do not) agree with previous findings*" without a further discussion of how error in each set varies – an understandable approach when the historical results did not report error well enough to fairly compare with the new measurements. Further complicating the issue, error bars in figures are often intentionally omitted for clarity given the overlap induced by limited scatter for highly populated datasets. With continued better reporting of precision, this trend will wane and as the community puts more emphasis on expanding error analysis to enable coherent facility and analysis-approach comparison^{6), 8)}.

A brief review of facility-specific factors that are often <u>not</u> reported, but that may introduce significant systematic error, includes: surface charging^{10), 25}, positioning control feedback frequency¹⁹⁾, sample oxygen contamination²⁹⁾, and oxygen

partial pressure in the processing environment²²⁾. Quantification of fluid flow is particularly important and researchers do not often attempt to bound this key variable - probably because this is computationally intensive and involves extensive specialized expertise⁴²⁾ to properly include key parameters such as deformed sample shape, coil or electrode geometry, thermophysical properties of the melt, and details of the processing environment. Magnetohydrodynamic (MHD) model predictions for convection during EML testing has been experimentally validated both with⁴³) and without⁴⁴) suppression of induced-flows but the specifics of how convection may develop during the process of conducting individual tests is most often ignored. Additionally, factors that may introduce significant random errors during processing of the data are often not controlled. An example is curve-fitting following edge detection to track the physical boundary of a levitated droplet for density evaluations. Bradshaw⁷⁾ at the MSFC/ESL recommends use of a 7th-order Legendre polynomial while Lee⁸⁾ at KRISS determined that 4-7th-order polynomials converged on the same fit if the aspect ratio of the deformation was less than 1.4285 and 6-7th order were almost the same up to an aspect ratio of 5 - thus recommending use of a 6th-order polynomial. Fukuyama³⁹⁾ recommends a 6th-order polynomial for EML without static magnetic damping²¹⁾ and a 5th-order polynomial for EML with static magnetic field flow-damping¹¹⁾. How much error is introduced by adoption of a specific analysis technique for multiple facilities has not been fully reported to date.

The bottom line is that with the wide diversity of techniques, control of variability is lost (or at least obscured). Key questions arise. What is the relative fidelity for using impedance to track surface motion versus using cinematography at different acquisition rates versus using an ultra-high speed linear array of photodiodes? How do we best conduct edge-detection and how does this impact inherent random versus systematic error? As a community, we need to initiate the definitive first-step to understand and control the sources of measurement error and to provide a baseline dataset for future re-evaluation of microgravity thermophysical property determination techniques.

4. Microgravity Relevance

Microgravity testing is used to provide enhanced fidelity of results³⁴⁾ purportedly due to three main effects. First, without strong gravitational accelerations and with reduced levitation forces a more spherical sample allows for better analysis of experimental behavior. Second, better control of convection in space results in higher measurement precision. Third, sedimentation and buoyancy induced segregation are eliminated in reduced gravity. But how much better do we do in space as compared to ground? The obvious solution is to quantify

Facility	ESL	EML
Ground	Metals and limited other materials P_{vac} to limit arcing Limited stirring $(0 \rightarrow L)$	Metals and doped semiconductors P _{vac} to P _{amb} gas environment Significant stirring (T)
Microgravity	HT Oxide, slag, semiconductors P _{amb} to limit evaporation No induced stirring (0) ELF ²⁴	Metals and doped semiconductors P_{vac} and low P_{gas} Wide range $(L \rightarrow T)$ MSL-EML ^{2(), 45)}

 Table 1
 Comparing Levitation Techniques on ground and in microgravity.

variability across platforms but access to space is expensive and hardware is specialized. **Table 1** presents a comparison of two common levitation techniques presently employed in microgravity testing.

The ESA TEMPUS⁴⁵⁾ (German acronym Tiegelfreies Elektromagnetisches Prozessieren Unter Schwerelosigkeit or Containerless Electromagnetic Processing under Weightlessness) for parabolic flight and MSL-EML²⁰⁾ facility for ISS space processing uses electromagnetic levitation to process conductive samples in high vacuum or gas environment with a wide range of convective conditions spanning the laminar to turbulent regimes. The JAXA Electrostatic Levitation Furnace (ELF) ²⁴⁾ facility uses electrostatic levitation to process materials including high temperature oxides with limited (to zero) stirring in a gas environment. Unfortunately, these facilities were specifically designed to target different material classes - MSL-EML for metals and ELF for high temperature oxides - and thus no common sample is currently available for comparison. If a common sample can be found, it must be conductive since EML cannot process non-conductive samples.

5. Future Directions

In order to validate test methods, analysis protocol, statistical evaluation, and reporting and archiving requirements, the Microgravity Physical Sciences Community must consider how to develop standards to facilitate information exchange and promote innovation in industry. The first step in this process is to organize an international interlaboratory program to perform complementary independent tests multiple times to build a database that will be shared across the community. This process is affectionately known as a Round Robin.

The idea of a Round Robin approach is neither new nor particularly innovative. It is, however, something that *must be done* if the microgravity materials property measurement field is to move from niche to mainstream. Fecht challenged the ESA Topical Team⁴⁶ membership during organizational meetings in 2008 to think about how to develop standards for the various techniques employed for thermophysical property measurement around the globe. Wunderlich, Matson and Pottlacher discussed the possibility of a Round Robin at the NIST 18th Thermophysical Symposium in 2012 but were unable to recommend how to proceed given limited funding opportunities at the time. Chiaramonte introduced the concept of Physical Sciences Informatics at AIAA-2012 and at the NASA MaterialsLab Workshop²⁾ in 2014 to promote sharing of data and broad dissemination of archived evaluation results. Pottlacher, as a NIST affiliate, publicly questioned each speaker at the NIST 19th Thermophysical Symposium in 2015 on how error analysis and recording of precision was accomplished for each measurement reported.

But the idea of a Round Robin approach has recently been gaining momentum and what is innovative is the strategy of expanding the ELF capabilities to include metals so that the two space facilities, EML and ESL, can be compared simultaneously to ground platforms (both levitation and traditional methods). The selection of pre-existing common samples is cost efficient – and per the definition of a Round Robin this means running what is already being run elsewhere.

The timing is right. We have the appropriate set of materials, we have the facilities on-line aboard the ISS, we can save space resources by running in parallel with existing programs, and we have a targeted funding opportunity supporting this work. Plus, we have community support from a broad array of international researchers⁴⁷⁾ who are already talking together as part of the ESA Topical Team and are interested in participating in the Round Robin.

By employing a Round Robin we get the broadest exposure for evaluation of facilities, experimental techniques, and analysis protocol. By expanding the subset of materials to be tested on ELF to metals, as is done terrestrially⁴⁸, we are not only broadening the usefulness of space hardware, we are increasing the potential throughput for physical science space investigations. The main risk is that by selecting metallic samples for processing, evaporation and subsequent deposition on facility components is certain to occur, which eventually requires cleaning of the chamber. This is a serious problem for MSL-EML with significant operational controls established to ensure safety of the astronauts, maintaining facility health, and preserving the useful life of the hardware. With ELF, the sample cartridge can be returned to earth for cleaning and recertifying and while this approach results in additional launch costs and some added crew time, this involves no change in the standard facility operation and represents a significant advantage for use of the ELF facility with metals.

Of particular interest is the combined use of modeling and experiment to understand how gravity influences both the accuracy and precision of each type of measurement technique. An example of this is use of TEMPUS by Egry to show how correction factors theorized by Cummings and Blackburn could be applied to oscillation frequency splitting during surface tension analyses⁵⁾. Archiving systems must work to promote public access⁴⁹⁾ and the thermophysical property measurement community needs to initiate activities prototyping the use of these tools to predict properties without the need for experiment. An example of this is use of ground-based oscillating cup viscometry to predict superalloy viscosity for a broad array of industrial alloys of complex chemistry from elemental measurements^{36), 37)}.

A final cautionary note on this approach - we know these techniques work on ground and many researchers use applications which differ in very subtle manners. We do not know how well the techniques will work in space. *Our goal should not be to say one technique is better than another*; we are trying to quantify variability and share analysis protocols to start to build a path to developing community standards for thermophysical property evaluations.

6. Conclusions

Microgravity thermophysical property measurement has the potential for providing material properties with superior accuracy and precision to better leverage and complement results obtained using existing ground-based facilities. The space physical sciences research community is in the process of developing standards for archiving the results from a wide array of test configurations to promote data sharing, innovative application, and deployment of new material classifications. The first step in this process is to investigate how to accomplish a collaborative/comparative project between research groups using a Round Robin format across test platforms – both in space and on ground. This requires a new perspective on the analysis of variability to include not only an understanding of accuracy, but also of measurement precision.

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