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Development of Thermal Conductivity Apparatus for Ablative Composite Materials

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Rafael has developed a transient thermal conductivity apparatus specifically for ablative composite materials. This innovative apparatus is capable of determining thermal conductivity of materials over the temperature range 330 to 1200 k, covering the pyrolytic stage of the composite material. The target specifications of the apparatus are the measurement of thermal conductivity in the range 0.05-2.0 W·m^{-1}·k^{-1} using specimen thickness in the range 7-25 mm. This is achieved using a new design of a symmetric heater-specimen assembly, forcing an adiabatic state along its centerline. The temperature-controlled system is sustained in a closed chamber, capable of introducing specific gases, enabling tests under a wide range of environmental conditions. This device will provide experimental results of thermal conductivity for ablative materials over a wide range of temperatures, enhancing the modeling capability of ablative materials performance in solid motor rockets.

I. Introduction

The need for thermal conductivity data in the industry grows with the growing effort to develop materials for high temperature applications. Moreover, the development of new composite materials for the aerospace industry requires modeling of heat transfer management and thermochemical ablation of these materials in high temperature. Rafael LTD has many years of experience in developing products for military, aerospace and civil applications. Although Rafael has powerful modeling capabilities of complex heat transfer problems, experimental data such as temperature depended thermal conductivity of composite materials in general, and particularly, composite ablative materials, is still necessary.

Thermal conductivity devices can be divided into two categories: Steady-state and transient devices. The steady-state devices include "Guarded hot plate"[1,3,6,11], "Heat Flow Meter"[2,5,6], "Radial Heat flow"[7], "Laser Flash"[8], "Divided Bar"[9],and "Panel test[10]. Transient devices include "Transient hot wire"[4,13] and "Transient line source"[12]. All of these devices are less accommodated to test composite ablative material, since ablative materials possess special characteristics: they have considerably lower heat conductivity, gas is generated during heating, thermal properties change non-monotonically, and the thermal characteristics of composites depend on rate of heating[14]. Steady-state measurements are not suitable for ablative materials since they undergo thermochemical degradation during the period of temperature stabilization, prior to actual measurement at elevated temperatures.

An appropriate concept which was found suitable for determining thermal conductivity of ablative materials is mentioned by Dimitrienko[14] dealing with thermomechanical behavior of

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composites at high temperatures. This concept takes into account the thermal characteristics of ablative composites and outlines the main characteristics of the device.

II. Apparatus Design

A. Principle of technique

The concept of the device[14] consists of a uniform ramp heating of a composite material specimen, while measuring the temperature difference on both sides of the material. A symmetric assembly of heater-specimen is built up in order to establish an adiabatic boundary condition at the middle interface (Fig. 1), assumed according to heat transfer definition. The heat flux to the specimen is measured during the test and the whole device is encapsulated in a blanket with very low thermal conductivity. In order to maintain inert environment required for the pyrolysis of the specimen, specific gases can be introduced in the sealed chamber.

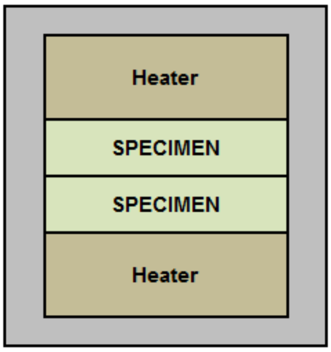

Figure 1. Scheme of conceptual device for measurement of the thermal properties of composites under high temperatures.

In order to regulate the heating rate and uniformity of the temperature field, it is desirable to add a metallic plate between the heater and the specimen. Holes in the metallic plate are essential for preventing pyrolysis gases accumulation at the interface of the metallic plate, causing intra-pore pressure elevation in the composite material. Using this assembly, the author[14] states the feasibility of determining thermal conductivity, along with thermal diffusivity and heat capacity.
Although the concept of the device is highly feasible meeting the main goals of the present work, some accommodations had to be made, especially in the raw data interpretation. Dimitrienko's suggestion[14], as outlined in detail, was found far too complex, with more variables to be assessed. In the present work, a detailed procedure is described for raw data interpretation towards thermal conductivity determination.

B. Governing equations

The present heat conduction problem is considered as a one-dimensional transient heat conduction problem with a boundary condition of uniform ramp-type temperature elevation with time. In order to determine the thermal conductivity of the specimen, there is a need to quantify the heat flux, as it varies during the test (Fig. 2).

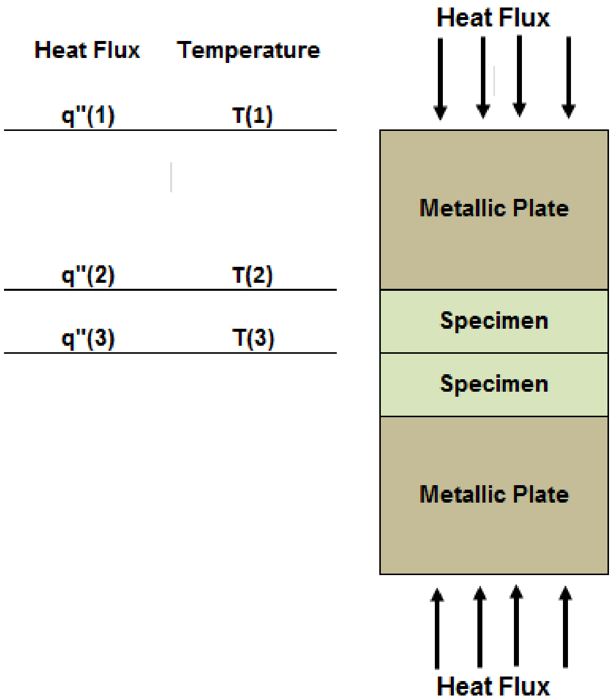

Figure 2. Updated concept scheme of a device for determination of thermal conductivity of ablative materials.

Using the Fourier law,

$$\rho_{(T)} \cdot C_{(T)} \cdot \frac{\partial T}{\partial t} = \frac{\partial}{\partial x}(\lambda_{(T)} \cdot \frac{\partial T}{\partial x}) \tag{1}$$

Where ρ is the density, C is the heat capacity, T is temperature and λ is thermal conductivity, with the boundary conditions,

$$T_1(0,t) = T_0 + (t \cdot b) \tag{2}$$

$$q''_3 = 0 \tag{3}$$

Where T_0 is the initial temperature at t=0, b is the heating rate, we obtain the temperature propagation profile in the metallic plate and the specimen, as illustrated in Fig. 3.

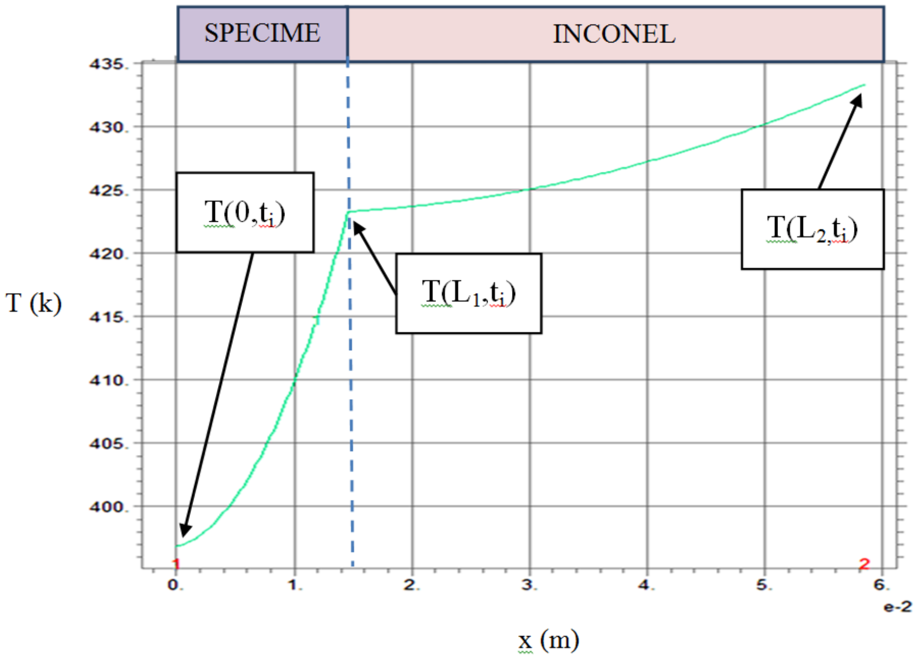

Figure 3. Example of temperature profile in the metallic plate and the specimen at time t_i . This example is illustrated with the help of a numerical solution, calculated in the finite-element Software FlexPDE Ver. 6.0 .

The time constant, is given by,

$$\tau = \frac{L^2}{2 \cdot \alpha} \tag{4}$$

Where τ is the time constant, L is thickness, and α is thermal diffusivity.

The time & displacement-depended temperature profile equation, is established as follows, given $t > [\tau_{specimen} + \tau_{metal}]$,

$$T(x,t) = \begin{cases} T_0 + (b_{specimen} \cdot t) - \Delta T_{specimen} \cdot \left(1 - \frac{x^2}{L_{specimen}{}^2}\right) - \Delta T_{metal} & ,0 < x < L_1 \\ T_0 + (b_{metal} \cdot t) - \left[\beta_0 \cdot \left(1 - \frac{(x-L_1)^2}{L_{metal}{}^2}\right)\right] - \left[(\Delta T_{metal} - \beta_0) \cdot \left(1 - \frac{x - L_1}{L_{metal}}\right)\right] & ,L_1 < x < L_2 \end{cases} \tag{5}$$

Where ΔT_{metal} is the actual temperature difference on the metallic plate and β_0 is the temperature difference on the metallic plate if it were sandwiched as the specimen and had an adiabatic interface, as well ($\beta_0 = b_{metal} \cdot \tau_{metal}$).

Using the rule of energy conservation at the interface between the metallic plate and the specimen, the "leaving" heat flux from the metallic plate equals to the entering heat flux to the specimen,

$$\lambda_{metal} \cdot \frac{\partial T}{\partial x}]_{x_{-2}} = \lambda_{specimen} \cdot \frac{\partial T}{\partial x}]_{x_{2_-}} \tag{6}$$

Where $\frac{\partial T}{\partial x}]_{x_{-2}}$ is the temperature gradient calculated for the metallic end, and $\frac{\partial T}{\partial x}]_{x_{2_-}}$ is temperature gradient calculated for the specimen's end, both co-existing at the same theoretical point (metallic plate-specimen interface).

In this way, the calculated thermal conductivity of the specimen is given by:

$$\lambda_{specimen} = \lambda_{metal} \cdot \frac{(\Delta T_{metal} - \beta_0) \cdot L_{specimen}}{2 \cdot \Delta T_{specimen} \cdot L_{metal}} \tag{7}$$

Note that Eq. (7) is valid if the time duration of the test is greater the summation of the time constants values of the metallic plate and the specimen ($t > [\tau_{specimen} + \tau_{metal}]$).

C. Specifications

The target specification of the apparatus is absolute measurements of composite materials with thermal conductivity in the range 0.05 to 2.0 $W \cdot m^{-1} \cdot K^{-1}$, using specimen thickness in the range 7 to 25 mm, from room temperature up to 1200 K. The device will be able to conduct tests with various gases introduced in the chamber. The target measurement uncertainty is ±5% or better. Heating rate of the specimen should be maintained at a constant value in the range 0.5 to 10 [K/min]. Heating symmetry should be maintained in the range 0 to 2 K.

D. Design and Analysis Tool: FlexPDE[15] Multiphysics 6.0

The complex design of the present device requires the use of numerical investigation of the heat transfer problem. The software "FlexPDE Multiphysics 6.0" has been used for designing the metallic plate and the specimen's dimensions, heat conduction to different parts of the assembly, energy requirements from the heater and considerations in choosing appropriate building materials. FlexPDE can model and analyze a variety of engineering problems in fluid dynamics, heat transfer, mechanical stresses, optics, electricity and more. FlexPDE uses finite element method together with adaptive meshing and error control by using numerical solvers.

E. General overview of the apparatus

The transient thermal conductivity apparatus, as described in Fig. 4, meets all initial requirements, except the target measurement uncertainty, which can be assessed only by a reference material. Verification and calibration procedures will be held during early tests of the apparatus, by using MICROSIL™ micro porous ceramic material. Final assembly of the upper part of the device consists of: (1) covering the heating chamber with an insulation blanket characterized by a very low thermal conductivity and (2) metallic cover for ensuring specific environmental gas inside the heating chamber. The heart of the device resembles a pressing machine, equipped with 25 thermocouples for measuring temperature elevation on both sides of the metallic plate and the specimen.

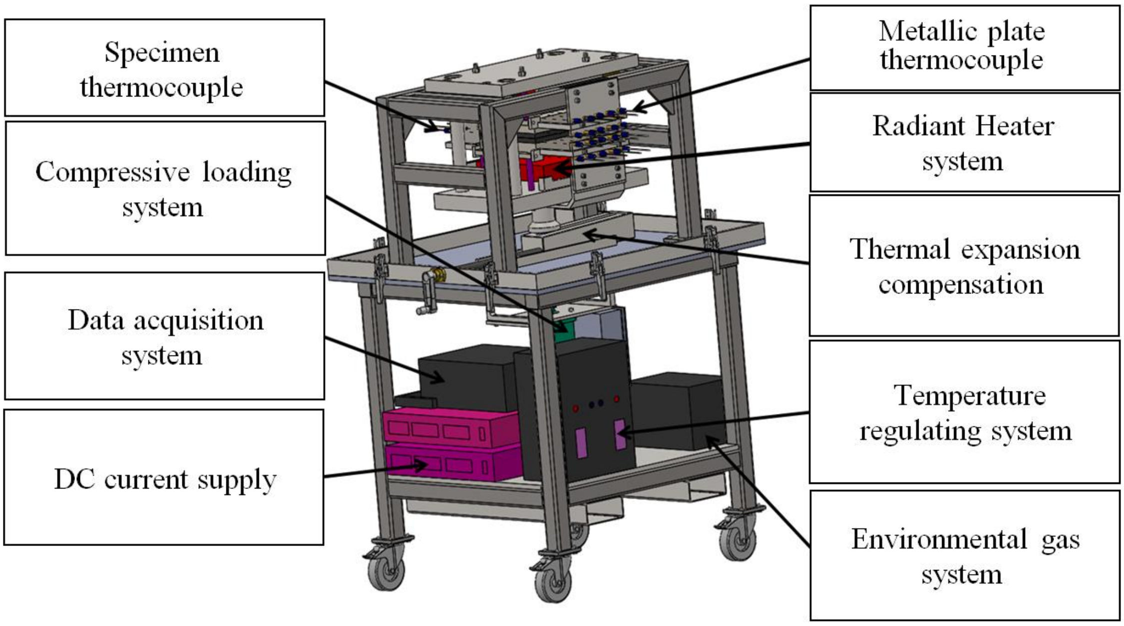

Figure 4. Main features of the transient thermal conductivity apparatus.

F. Radiant heater

The apparatus uses a commercially available ceramic-fiber radiant heater consisting of rows of iron-chrome-aluminum heater wires cast in a 50mm thick ceramic. The heating element is single phased, generating a maximal power of 1700W, with maximal voltage of 50V. The overall allowable temperature of the heating element is 1400 K, controlled by a temperature overload regulator. The temperature regulating system is capable of maintaining a linear ramp over hundreds of minutes with an accuracy of 0.5%. The ramp rate can be adjusted readily and by another adjustment – it is possible to run steady state measurements of thermal conductivity.

G. Temperature measurement

Each metallic plate is instrumented with ten metal sheathed, mineral insulated type K thermocouples, five on each side, at the center and edges of the "metered region" (Fig. 5). The thermocouples are not earthed to the metal sheath, in order to minimize measuring noise. The metallic sheath is made of 304 stainless steel, 0.5 mm in diameter. The metered region (Fig.5, dashed rectangle), sized 100 x 100 mm, is less than 20% of the overall area of the plate, defined as the measuring region in order to minimize inaccuracies caused by heat losses from the edges of the metallic plate and the specimen. The thermocouples are located between the holes which are intended for pyrolysis gas release.

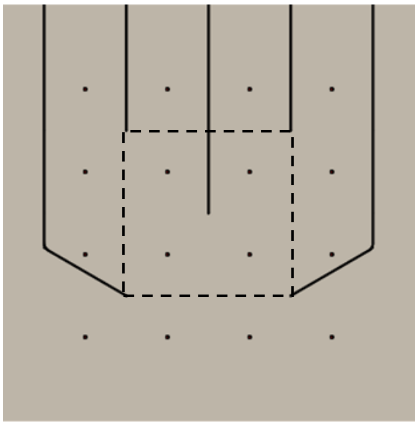

Figure 5. Thermocouples layout on the metallic plate.

H. Structural Materials

The metallic plate is a nickel-alloy INCONEL 625. This alloy exhibits excellent mechanical properties up to a temperature of 1230K (long term working temperature). It's thermal properties are defined up to 1270K. The upper frame of the device is made of stainless steel 304, capable of withstanding high temperature (up to 1270 K).

I. Thermal expansion compensation system

Two of the governing parameters of thermal conductivity determination are the thickness of the Inconel plate and the specimen. Since the change in temperature is quite high over the entire period of the test, the thickness of the Inconel plate and the specimen are subjected to substantial changes. A change in specimen thickness could cause mechanical damage to the specimen's structure, and for that reason a mechanical compensating system was added. Measurements were made up to 1200K in order to quantify the heating assembly gap intended for the specimen implementation.

J. Compressive loading system

The compressive loading system includes a pneumatic cylinder which presses the double-sandwiched assembly of Inconel-specimen (Fig. 2). This system minimizes the thermal contact resistance between the Inconel plate and the specimen, together with the thermocouples entrapped at that interface. The compressive load value is depended on the type of specimen, with a nominal value of 2.5 KPa.

K. Validation and Future work

The upcoming work following the manufacturing and commissioning of the apparatus includes uncertainty assessment and validation of measured thermal conductivity. A preliminary test has been carried out, consisting of the use of an EPDM-based insulation (Fig. 6). This material is an ablative insulation, widely used as internal insulation of rocket motors. There is a need of finding an appropriate material, suitable of validating data up to 1200K. Meanwhile, an uncertainty assessment procedure will be implemented.

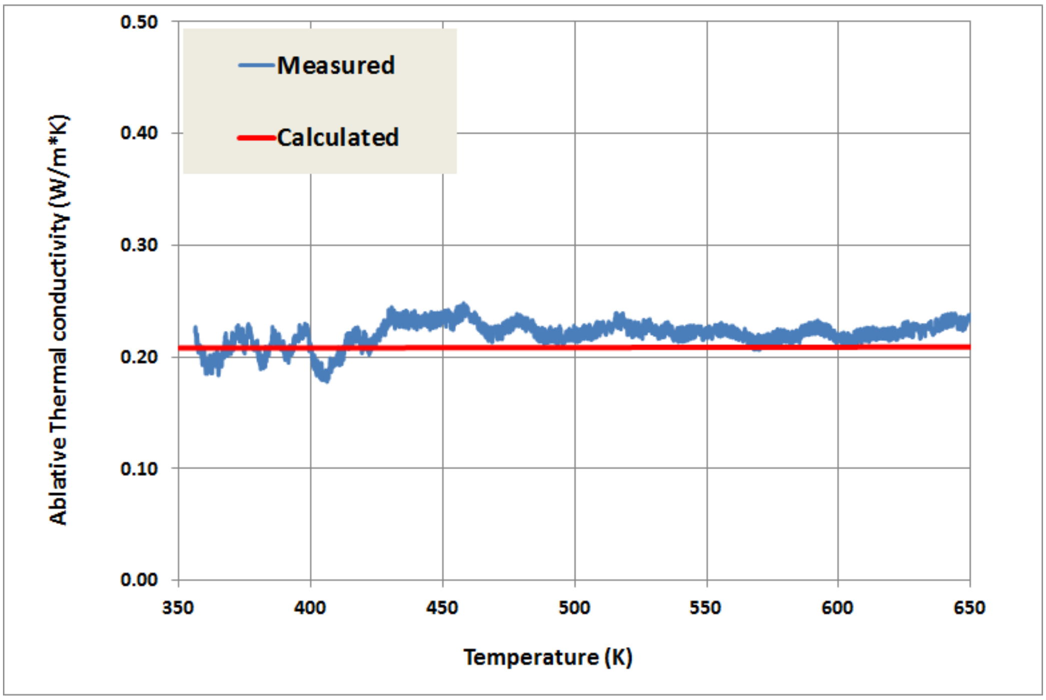

Figure 6. Thermal conductivity of EPDM-based insulation

III. Conclusion

The present work demonstrates a transient thermal conductivity apparatus design. This innovative apparatus is capable of determining thermal conductivity of ablative composite materials over the temperature range 330 to 1200 K, covering the pyrolytic stage of the composite material. This device can be reconfigured to accommodate test specimens of various sizes, and run in steady state mode.

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