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Thermal conductivity of thermally-isolating polymeric and composite structural support materials between 0.3 and 4 K

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ABSTRACT

We present measurements of the low-temperature thermal conductivity of a number of polymeric and composite materials from 0.3 to 4 K. The materials measured are Vespel SP-1, Vespel SP-22, unfilled PEEK, 30% carbon fiber-filled PEEK, 30% glass-filled PEEK, carbon fiber Graphlite composite rod, Torlon 4301, G-10/FR-4 fiberglass, pultruded fiberglass composite, Macor ceramic, and graphite rod. These materials have moderate to high elastic moduli making them useful for thermally-isolating structural supports.

1. Introduction

Cryogenic instruments frequently require rigid mechanical support of significant mass while minimizing conductive heat flow. In particular, optical systems require very stiff structural mounts. To increase the stiffness of a structure the designer generally has the choice of using more material or choosing materials with larger elastic moduli. However, materials with higher elastic moduli tend to have higher thermal conductivity. This situation motivates the use of the ratio of elastic modulus to thermal conductivity as the figure of merit for thermally-isolating structural materials.

This work was motivated by the need to support the cryogenic microwave receiver of the Spider balloon experiment [11,10,3]. The Spider instrument insert has massive components at 4.2, 1.4, 0.35 and 0.25 K. Each of these temperature stages are thermally and structurally referenced to an outer liquid helium cryostat. The cryostat will tip in elevation from zenith to horizon and the cryogenic support structures must minimize the gravitational deflection of the focal plane. Historically, we have used polyimide supports such as Dupont Vespel SP because of its low thermal conductivity [9,12]. Advanced polymers like Vespel are generally quite expensive. At the time of this writing, 1/2″ Vespel rods cost approximately half the price of gold by weight. Because the Spider instrument will include six duplicate focal plane structures, the cost and availability of the material required to support them is a significant concern.

There are a number of materials properties that are potentially important when designing thermally-isolating structural supports. As mentioned previously, low thermal conductivity and high modulus are desirable attributes. The elastic modulus of materials increases as they cool (for example, see Flynn [5]). This increase in stiffness is moderate in metals but can be significant for some polymers. In particular, the elastic modulus of Teflon increases by approximately a factor of 20 when cooled from room to cryogenic temperatures [2]. In addition to stiffness, the strength of materials is important for structures that may experience large forces or shocks.

The stability of the dimensions of the structure upon cooling may also be important. In this case, materials with small coefficients of thermal contraction, such as carbon fiber composites [14], may be desirable. For larger structures the support members themselves may comprise a significant fraction of the mass, in which case the density may become important. The enthalpy of the materials, particularly those with low thermal conductivity, may also be a significant concern since they will take longer to cool down.

2. Materials tested

We test two types of DuPont Vespel polyimides. Vespel SP-1 is the base polyimide resin and SP-22 is filled with 40% graphite by weight. Polyetheretherketone (PEEK) is a semi-crystalline thermoplastic and is available in both pure and filled forms. We test three types of PEEK. The first is unfilled Ketron PEEK 1000 manufactured

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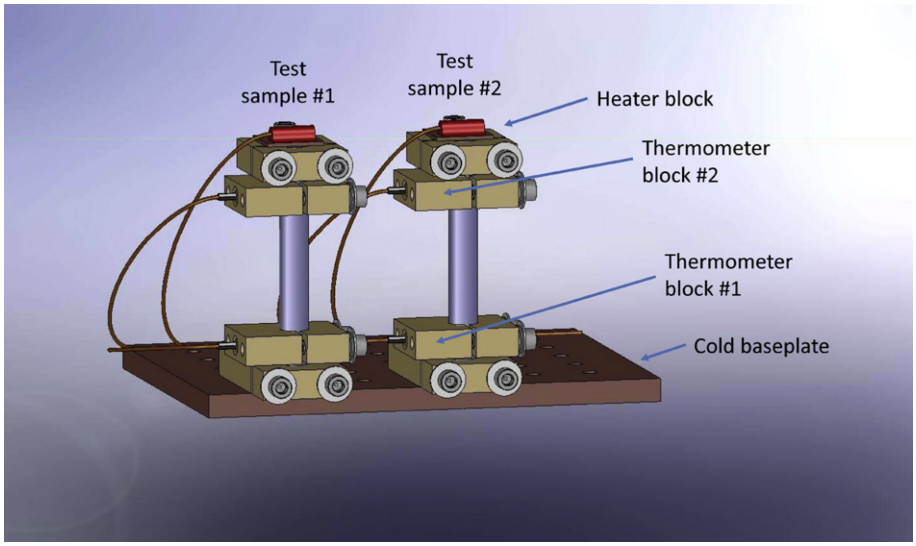

Fig. 1. Schematic of the experimental setup. There are two samples in the test dewar but they are measured individually. The schematic shows the heater blocks, Cernox thermometer blocks and baseplate, which is attached to the sub-Kelvin ^{3}He sorption fridge.

by Quadrant Plastics.[1] The second is 30% carbon fiber-filled PEEK 450CA30 manufactured by Drake Plastics[2] (PEEK CA30). The short carbon fibers are not oriented in this material, although the extrusion process may introduce some anisotropy to its properties. We only measure the thermal conductivity along the extrusion direction of the rod. The third PEEK sampe is 30% glass-filled Sustatec PEEK manufactured by Rochling Sustaplast[3] (PEEK GF30). This is also an extruded product and we measured the thermal conductivity only along the axis of extrusion.

We measure two composite materials made by Avia Sport Composites.[4] The first is pultruded carbon fiber called Graphlite. Graphlite contains 67% carbon fiber by volume and a Bis-F epoxy matrix. The pultrusion process naturally orients the fiber bundle along the axis of the rod, but visual inspection of the material shows that the fibers are not uniformly oriented along this axis. The second material is a pultruded fiberglass rod with Bis-F epoxy which we will refer to as Avia Fiberglass in this paper. We only measure the thermal conductivity along the pultrusion axis of the rods. Both of these composites are supplied by CST Composites[5].

We measure a sample of graphite rod from Poco Graphite Inc.[6] The type of graphite measured is the same as that tested in Woodcraft et al. [18] (industrial grade AXM-5Q). This graphite has a particle size of 5 microns and an apparent density of 1.73 g/cc. Macor is a machinable glass-ceramic manufactured by Corning.[7] We also measure a sample of Torlon 4301. The Torlon base (4203) is a polyamide-imide (PAI) thermoplastic manufactured by Quadrant Plastics and the 4301 grade includes both 12% graphite powder and 3% PTFE.

The final material tested is a commonly available G-10/FR-4 Garolite fiberglass rod purchased from McMaster-Carr.[8] The manufacturer of this sample is unknown, but the distributor states that it meets MIL-I-24768 specification for G-10/FR-4. Although a cryogenic grade of G-10 is available, our own experience and that of Walker and Anderson [17] suggests that for many cryogenic applications the readily available G-10/FR-4 is an acceptable material. However, FR-4 may be unsuitable for ultra-high vacuum applications because of the presence of a halogen flame retardant. These rods are fabricated by bonding layers of woven glass fabric into a sheet and then grinding them into rods along one of the fiber axes. We confirmed the orientation of the fibers in our sample by dissolving away the binding epoxy. We measured the thermal conductivity along the plane of the woven glass fabric.

3. Experimental procedure

The thermal conductivity measurements are performed in an IR Labs HD-3(10)L liquid nitrogen/liquid helium cryostat. Temperatures below 0.27 K are obtained with a ^{3}He sorption fridge operating from the pumped helium bath of the cryostat. The experiment is conducted within the 1.4 K radiative environment of the pumped helium cryostat.

The sample test stage (shown schematically in Fig. 1) has provision for two independent samples. The C10100-alloy copper mounting blocks are two piece clamps. Because the coefficient of thermal contraction of the test samples is generally higher than copper we use stainless belleville spring washers to maintain clamping force. A mounting block holds the sample upright and thermally sinks it to the cold stage. There are two thermometer blocks along the length of the sample, each containing a calibrated Cernox thermometer read out with a 4-wire resistance bridge. At the top of each sample is a 1 MΩ metal-film resistive heater. In order to track any temperature coefficient of the resistive heater, we monitor both voltage and current across the heater to determine the heater power. Over the range of temperatures reported in this paper, the heater impedance is observed to vary by 7%. A thin layer of Apiezon-N thermal grease is applied to all interfaces. The test samples were all turned down to 0.25″ in diameter with the exception of the Poco Graphite sample, which was 3/16″ diameter and shimmed in the mounting blocks. The overall length of each sample was $\sim$2″. The two thermometer blocks are spaced approximately 1″ apart and their separation is measured with calipers.

An important element of the experimental setup is the separation of the thermometers from the heaters and heatsinks. When heat flows across a thermal interface a temperature gradient is formed. This thermal boundary resistance can lead to a significant temperature offset between the thermometer block and the sample under test if heat flows through the clamp to the material. If

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[2] http://www.drakeplastics.com.
[3] http://www.sustaplast.com.
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