Study on transient heat transfer at metal to dielectric interfaces in the temperature range between 3.5 K and 30 K

T Koettig, J Golm, J Liberadzka, P Borges de Sousa, J Bremer
CERN, CH-1211 Geneva 23, Switzerland
E-mail: torsten.koettig@cern.ch

Abstract. The thermal conductivity and diffusivity across a combination of metallic and insulating layers are important thermodynamic input parameters for cooling studies of composite materials or assemblies built out of layers of different electrical and thermal conductivity. The dynamic response of such thermal contacts across electrically insulating layers can be expressed in terms of a diffusivity-like value, which is giving insight on the interface thermal resistance. A two-stage cryocooler based test stand is used to measure the thermal conductance of samples. Variable base temperatures of the experimental platform at the cryocooler allow for steady-state and transient heat flux measurements up to 30 K. This paper describes the measurement methodology applied to such kind of non-uniform sample compositions, especially the frequency dependence of the diffusivity values is discussed.

1. Introduction

At low temperatures one has to pay attention to the boundary resistance at interfaces of materials. Bringing different bulk materials into contact leads to a lower thermal conductance than expected from adding up the individual values for each material. This is attributed to the thermal boundary resistance, which increases the overall thermal resistance of a stack. The thermal boundary resistance was first described for interfaces between solids and liquid helium, known as Kapitza resistance [1]. A thermal boundary resistance also occurs at solid-solid interfaces. It affects for example the heat transfer between parts of dielectric interlayers between metal components (stacked coil layers for magnet assemblies or cooling interfaces that need to be electrically insulated like electrode thermalisations). Here, the thermal boundary resistance is highly influenced by the actual contact area between the boundaries, which at interfaces can often be only a factor of $10^{-6}$ of the nominal area [2].

Transient heat transfer phenomena are best described by the thermal diffusivity $\kappa$, which is a material property characterising the dynamic thermal behaviour of a bulk material. It describes the change of the spatial temperature distribution with time, caused by a temperature gradient. The thermal diffusivity is defined as the thermal conductivity $\lambda$ divided by the product of specific heat $c$ and density $\rho$ as follows:

$$\kappa = \frac{\lambda}{\rho \cdot c} \quad (1)$$

A detailed introduction to the behaviour of thermal conductivity and specific heat at low temperatures can be found in [3]. Phonons are the sole heat carrier in insulators. In the
temperature range below the phonon peak of thermal conductivity the mean free path of the phonons is determined by scattering on crystal faces, lattice defects and amorphous structures and therefore tends to a temperature independent value. In that low temperature range, $\lambda$ becomes proportional to the heat capacity, which is the only temperature dependent quantity left in eq. (1) and is proportional to $T^3$, see [3]. The same happens in metals: when the mean free path of the electrons reaches the dimension of the lattice defects, the temperature behaviour of thermal conductivity is the same as that of the heat capacity. Therefore the thermal diffusivity should become constant towards very low temperatures, as defined in eq. (1). As stated at the beginning, the thermal diffusivity is a parameter defined only for bulk materials. How fast a heat disturbance propagates from one point of a sandwich made of different materials in series, depends also on its boundaries. The propagation velocity of the temperature wave is different in each layer. Instead of only adding up the parameters for each material in series, one has to add an additional resistance for the boundaries, which decreases the thermal diffusivity. For a sandwich consisting of many layers one obtains the total thermal resistance:

$$R_s = \sum_i R_i + \sum_j R_{Kj} \quad \text{with} \quad R_i = \frac{x_i}{\lambda_i \cdot A},$$

where $R_{Kj}$ is the thermal boundary resistance of the respective interface. The thermal resistance caused by the material itself $R_i$ can be calculated from the thermal conductivity considering its cross section $A$ through which the heat is transported and the thickness $x_i$ of each component. The total specific heat capacity multiplied by the density for the whole sandwich structure of a constant cross section can be determined by adding up the specific heat capacity $c_i$ times the density $\rho_i$ for each layer, multiplied by the percentage of the total thickness of the layer. This is shown in eq. (3), where $d$ is the total thickness of the sandwich:

$$c_s \cdot \rho_s = \sum_i \frac{x_i}{d} \cdot c_i \cdot \rho_i.$$

An effect caused by boundaries that is altering the heat capacity significantly for a sandwich is not known. Jun reports in [4] that the film thickness of Al can influence the heat capacity for a temperature range of 300 K to 420 K. A small film thickness enhances the heat capacity, but for a layer thickness of 1150 nm the reported specific heat is the same as for the bulk material. The thermal diffusivity-like value of a sandwich structure can be therefore expressed by $\kappa^*$ with different materials in series calculated by combining eq. (1) to (3):

$$\kappa^* = \frac{d}{A \cdot R_s \cdot c_s \cdot \rho_s}.$$

A common method to determine the thermal diffusivity of a material is the AC approach, which uses a sinusoidal heat signal. The temperature modulation is applied to one side of the sample. By crossing the sample the temperature wave gets attenuated and phase shifted (phase method). Therefore, the differential heat equation has to be solved. For a heat flow only in one dimension a solution can be found, defining the thermal diffusion length $\mu$ as the ratio of amplitudes $\hat{A}$ at position (0) and $(x)$ and extracting the thermal diffusivity-like value:

$$T(x,t) = T_{avg} + \hat{A} \cdot \cos \left(\omega t - \frac{x}{\mu}\right) \quad \text{with} \quad \mu = \frac{x}{\ln \left(\frac{\hat{A}(0)}{\hat{A}(x)}\right)} \quad \text{and} \quad \kappa^* = \frac{\mu^2 \cdot \omega}{2}.$$

2. Experimental set-up

A dedicated set-up (figure 1) is established for thermal conductivity and diffusivity tests [5]. The temperature oscillation at one side of the sample is generated by a glued heater of 100Ω that
is powered by a function generator (peak to peak voltages are ranging from 4 V to 20 V). The base temperature of the platform is stepwise increased from 3 K to 30 K by an additional heater circuit. The minimum temperature that is reached is determined by either directly linking the platform to the cryocooler or coupling it via a passive thermal attenuator, which is described in [6]. That attenuator guarantees a reduction in temperature oscillations, caused by the cryocooler and observed at the experimental platform from ±50 mK to values well below ±50 µK.

**Figure 1.** Picture of the tested samples, a) sandwich set-up mounted at the experimental platform. The components, which create twice a metal to sapphire interface are indicated. Two different samples with In or Ti-Au coating on sapphire were tested. The second sample of a representative 11 T dipole coil of the HiLumi LHC project is shown in b). The location of the temperature sensors made with indium foil is indicated at the first and last Rutherford cable, establishing the active length L between sensors.

### 3. Results and discussion

OFHC copper was chosen as a reference sample to validate the measurement set-up and the respective influence of a threshold frequency caused by the time constants of the sample and its thermal link to the cooling source [7]. The results of the measurement run on the cryocooler and of two points obtained in a vacuum chamber cooled by LN$_2$ at 78 K and water at room temperature are shown in figure 2. The determined values are in very good agreement with literature values and small deviations towards the lowest investigated temperatures are due to the strong influence of lattice imperfections on the thermal conductivity of the sample. The used Cu has values of thermal conductivity very close to literature data for RRR100.

The values for the Cu-In-sapphire sandwich in figure 3 are almost a factor ten higher in terms of thermal conductivity but only a factor 2 higher in thermal diffusivity than the more complex Cu-In-Au-Ti-sapphire one (having comparable values of heat capacity of the bulk materials in the sandwiches [7]). A numerical model has been established to verify the influence of interfaces on the thermal diffusivity and the comparison of the results will be discussed later in this section. The respective peak values in figure 3 appear always at lower temperature for the Cu-In-Au-Ti-sapphire sandwich, but approach the same values at the lowest tested temperature around 3 K. The values of thermal diffusivity are obtained with a wide variety of applied AC heat loads causing deviations at low temperature only for the highest values of heater voltage 20 V. Such applied heating powers create larger temperature gradients along the sample length, improving in general the measurement accuracy but also influencing the extracted diffusivity value by averaging across that $\Delta T$. The comparison with the numerical model shows a good agreement for the Cu-In-sapphire sandwich, while the Cu-In-Au-Ti-sapphire sandwich shows a
Figure 2. Plot of the determined thermal diffusivity values for OFHC copper, a) Frequency dependence in the low temperature range measured at the cryocooler set-up; b) Thermal diffusivity values of the plateau plotted vs. temperature and compared to literature data [8].

Figure 3. Plots of the determined thermal transport properties for the two sandwich structures with sapphire interfaces via In or Ti-Au deposits [7]; a) the resulting peak in thermal conductivity is a combination of the temperature dependent behaviour of sapphire, indium and copper (plus respective interface thermal resistances). In b) values of thermal diffusivity are plotted vs. temperature for both sandwich structures. The mentioned damper is a passive thermal attenuator placed in between the cryocooler cold-tip and the experimental platform [6]. Dashed and dash-dotted lines are the respective outcome of the modelled values.

deviation of a factor three, which only reduces towards lowest tested temperatures. The authors attribute that deviation to the formation of Ti-sapphire interlayers namely TiO and TiAl, which are essential for the well-known adherence effect of e.g. Ti on sapphire [9]. That interaction is covered in the model by the use of the measured thermal conductivity, but it is not included in possible heat capacity effects at the interfaces of Ti [4].

Further measurements are performed on a cable sample, representing the 11 T coil of the HL-LHC project [5]. Ten layers of Rutherford type cables were piled up and impregnated in a straight configuration. The temperature sensors have been placed directly on the first and last cable. There are two methods to extract the thermal diffusivity-like value from the plateau data of the frequency dependence, plotted in figure 4a). The first is the amplitude method described in eq. (5) and the second method uses the respective difference of the time constants
Figure 4. Plot of the determined thermal diffusivity a) vs. frequency and b) extracted values vs. temperature of the cable stack as shown in figure 1b).

\( \tau_i \) of the two temperature sensors in response to a step heating, see figure 1b). Hence, the thermal diffusivity-like value is determined from \( \kappa^* = L^2 / \Delta \tau \), with L being the distance between temperature sensors, indicated in figure 1b). The amplitude method delivers a comparable slope to the calculated component contribution, but shows an offset of a factor two, while the \( \tau \) method reaches the same values at low temperatures but has a different slope.

4. Conclusion

The investigation of transient thermal phenomena has been extensively tested on a cryocooler based test stand. The results are essential for numerical simulations of quench propagation studies in magnet coils or pulsed heat load cases. The thermal response to step and sinus-wave function heating was evaluated. For the amplitude method the threshold frequency depends on the time constants of the sample and the thermal link to the cold source. The determination of thermal diffusivity values from the time constants of the step function method giving reasonably close values and is therefore a valuable option to overcome the limitations of the amplitude method. The demonstrated analysis has been applied to bulk materials such as OFHC copper, bulk epoxy and tungsten alloys and can be used also for heterogeneous sample configurations.

References

[1] Kapitza P L 1941 Physics Review 60 354–355
[2] Gmelin E, Asen-Palmer M, Reuther M and Villar R 1999 Journal of Physics D: Applied Physics 32 R19
[3] Eness C and Hunklinger S 2005 Low-Temperature Physics SpringerLink: Springer e-Books (Springer Berlin Heidelberg) ISBN 9783540266198
[4] Jun Y, Zhen-An T, Feng-Tian Z, Guang-Fen W and Li-Ding W 2005 Chinese Physics Letters 22 2429
[5] Koettig T, Maciocha W, Bermudez S, Rysti J, Tavares S, Cacherat F and Bremer J 2017 IOP Conf. Ser. Mater. Sci. Eng. 171 012103
[6] Dubuis G, He X and Božović I 2014 Review of Scientific Instruments 85 103902 (Preprint https://doi.org/10.1063/1.4896049)
[7] Golm J 2018 Study of Thermal Diffusivity of Dielectric-Metal Sandwich Structures at Low Temperatures presented 18 June 2018 URL https://cds.cern.ch/record/2634825
[8] Jensen J, Tuttle W, Stewart R and Brechna H 1980 Thermal Diffusivity Brookhaven National Laboratory Selected Cryogenic Data Notebook (Brookhaven National Laboratory)
[9] Selverian J H, Ohuchi F S, Bortz M and Notis M R 1991 Journal of Materials Science 26 6300–6308 ISSN 1573-4803