Trident™ Thermal Conductivity Application Highlight: Measuring Thin Film Samples using Flex TPS Sensor

The following Application Highlight addresses the measurement of polymeric thin films using the Flex Transient Plane Source (TPS) sensor and the Thin Films utility available on Trident.

Thin Films are used for a variety of applications in protective optical and electric coatings, thin-film photovoltaic cells and thin film batteries (**Figure 1**). While thin film materials have existed for decades, thermal conductivity measurement methodologies have traditionally been focused on exploring bulk samples, and the capability to characterize these specialty materials has generally lagged. In recent years, the knowledge gap has shrunk, prompted by new and exciting markets in nano and microscale fabrications where thermal management is significantly important. A novel tool for such characterization is the Transient Plane Source (TPS) adaptation for testing thin films as outlined in ISO 22007-2.

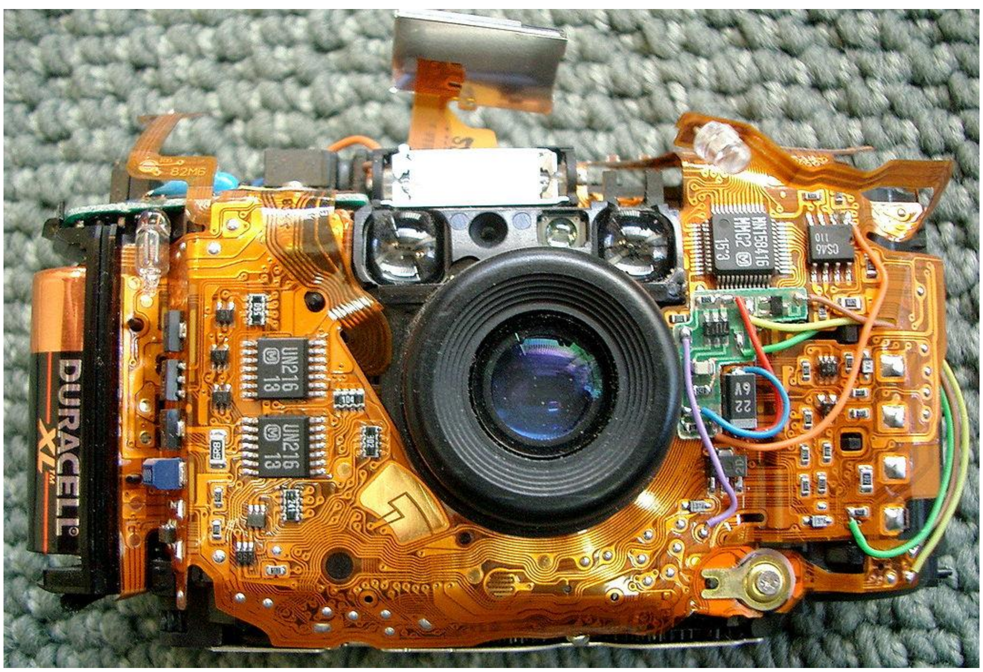

Figure 1. Thin Films have many applications ranging from flexible electronics, optics, and photovoltaics each with thermal management problems to solve. In this example photo, electronic components are embedded in a thermally insulative and electrically insulative material called Kapton. (Copyright: Wikipedia Commons, 2020).

Using a C-Therm FLEX TPS thermal conductivity sensor and Trident's *Thin Films* Utility, the measurement involves testing initially with just the selected backing material. The backing material is selected based on the type of sample being tested - according to the ISO guidance document it should be at least 10 times greater thermal conductivity than the test material.[i] This is followed by testing 1 layer of the thin film and then 2 layers of the thin film (in some cases up to 3 layers may be required). The successive addition of the thin film is used to generate a linear regression of temperature rise *vs* film thickness, which is then used to determine thermal conductivity (k). A general rule of thumb for the number of tested layers is to have a total layered thickness less than 1000 μm for best results. Measurement validation is determined by the line of best fit and an R^2 value of at least 0.999 is required. The TPS method requires only a few layers of film to achieve a valid result, making it appealing for such characterization. The procedure does require an accurate measurement of the film thickness (± 1 µm) to achieve a high-quality result.

TPS Thin Films Utility Basics

The TPS method employs a double-sided sensor (**Figure 2**) which is comprised of a spiral of electrically conductive nickel, sandwiched within an insulating polyimide material. By applying a voltage to the sensor, a temperature gradient is generated at the sample sensor interface and the temperature rise is measured and recorded by a Wheatstone bridge circuit with a data logging system.

Figure 2. C-Therm FLEX Transient Plane Source Thermal Conductivity Sensor (Available in 6, 13 and 30 mm Size Options).

This is the general function of a bulk measurement for TPS. By measuring the sample's temperature rise *vs* film layer from zero (just the backing material) to at least two total layers (**Figure 3**), thermal resistivity, thus thermal conductivity can be obtained. As noted above, for this method to work, the films must have a thermal conductivity much less than the backing material (more than one order of magnitude lower). The reason for this is because the backing material and sample are too similar with respect to their thermal properties, the film itself will become too thermally transparent for the TPS sensor to measure. For example. If stainless steel was used as the backing material (typical k of 15W/mK), the sample being analyzed would have to be less than 1.5 W/mK. An example of a linear regression of temperature rise vs film thickness is shown in **Figure 3**.

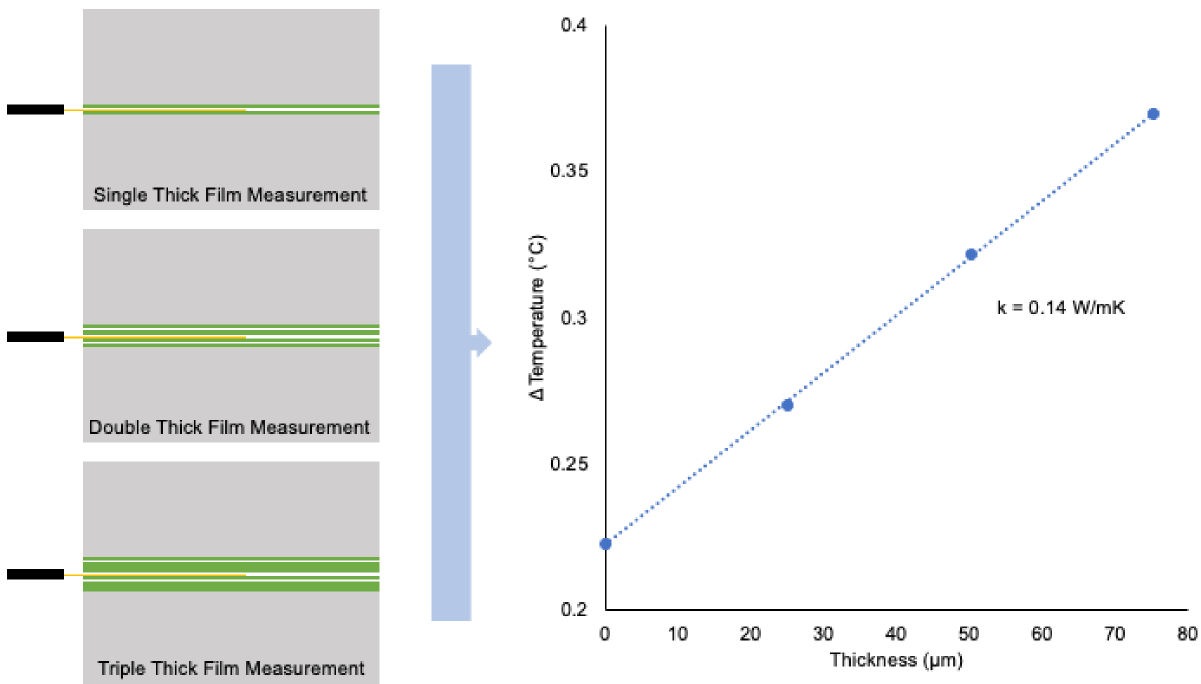

Figure 3. Schematic for measuring thin film TPS measurements. A single thick film measurement is conducted, followed by a double thick film measurement and a triple thick film measurement. The resulting relationship between change in temperature and film thickness can give the thermal conductivity by relating thermal resistivity to thermal conductivity.

Case Study: Testing the Thermal Conductivity of a Variety of Thin Films

To give a sense of accuracy of the *Thin Films* utility on Trident, a series of films were measured and compared to literature values for these films. In all cases, a good alignment of the measured value on Trident *Thin Films* utility is found for each of these standard thin film materials. Each thin film is free standing, non-compressible and fully consolidated, with minimal variation in the thickness throughout the film.

The following guidelines were followed to prepare samples:

(1) Test specimens must be the same size or larger than the TPS sensor.
(2) For films which are less than 50 μm in thickness, 3 layers of film are used along with the additional measurement of just the backing material (no film). For films greater than 50 μm thick, 2 layers of film are measured in addition to the measurement of just the backing material (no film).
(3) Contact pressure is important for this measurement. In order to get valid results, the sample must be tested under a high force load. This is most easily accomplished using the TPS Clamp accessory. (**Figure 4**)

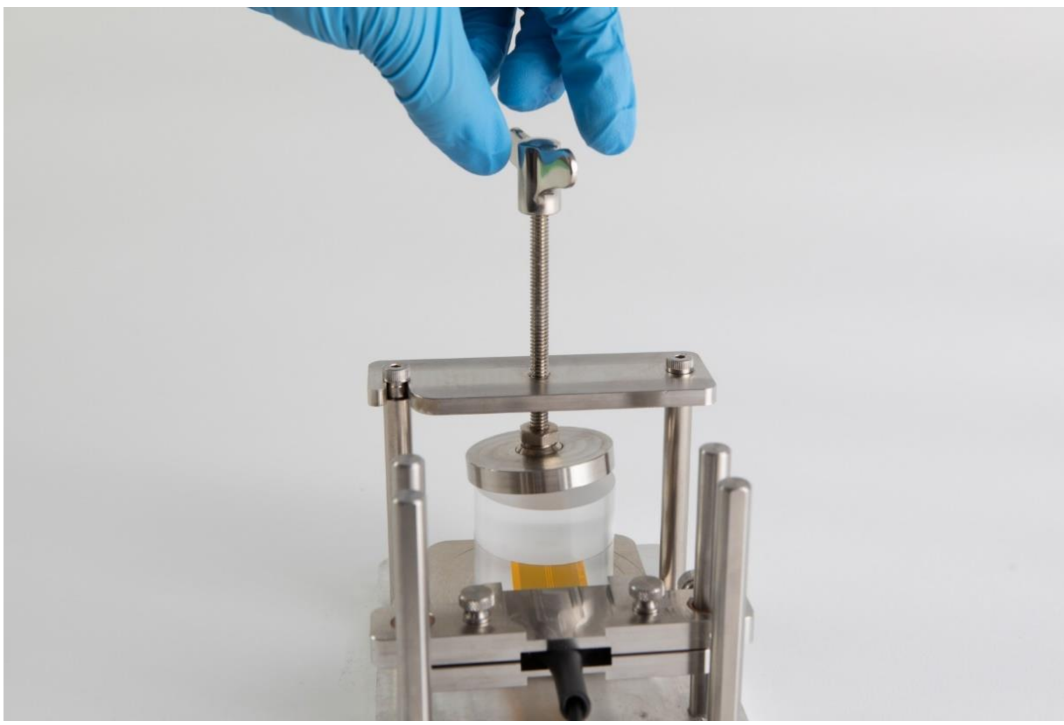

Figure 4. TPS Clamp Accessory Recommended for Use in Testing Thin Films (Note picture shows bulk sample testing - provided for illustrative purposes on use of accessory).

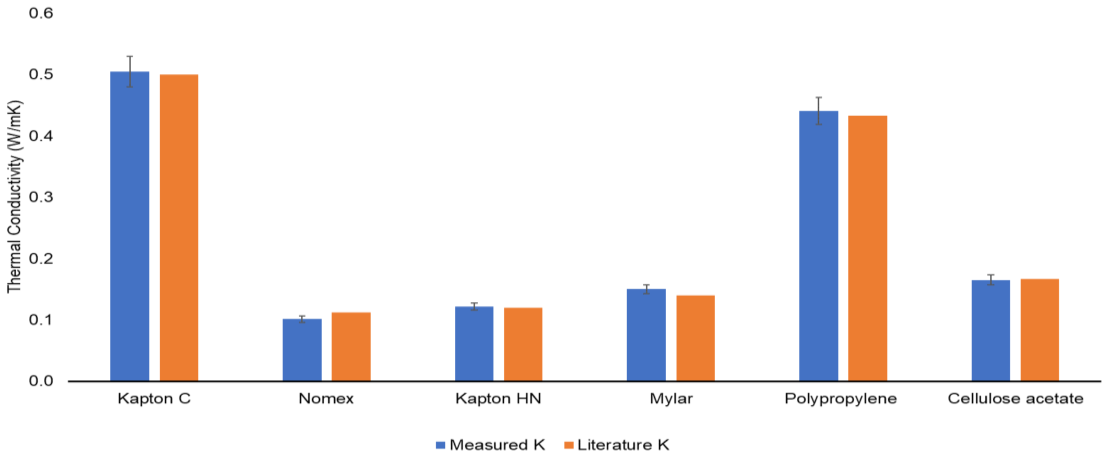

Figure 5. Comparison of thin film measurements obtained on Trident TPS using the Thin Films module as compared to literature values. The largest deviation was found for Nomex (10% as compared to literature values).

Figure 5 summarizes the performance of the FLEX TPS sensor with Trident's *Thin Films* utility for measuring industrially relevant thin film materials. Kapton C[ii] and polypropylene[iii] thin films were found to be the thickest films (500 µm thick) and demonstrated strong reproducibility and accuracy with respect to literature values. Additionally, thinner films like cellulose acetate[iv] (25 µm) and Mylar[v] (30 µm) additionally tested with accurate and reproducible results. In general, C-Therm recommends the *Thin Films* utility for studying polymeric thin films with a minimum thickness of 10 µm with a thermal conductivity between 0.05 – 5 W/mK.

Thin film testing is also possible using all sensor size options available on Trident (6, 13 and 30 mm). Trident offers maximum flexibility to allow for testing of samples of varying sizes, even those with extremely small diameters. Below is data highlighting the testing of a 127 µm polyether ether ketone (PEEK) film at room temperature. All results were within 5% of the reference value for PEEK (0.25 W/mK).

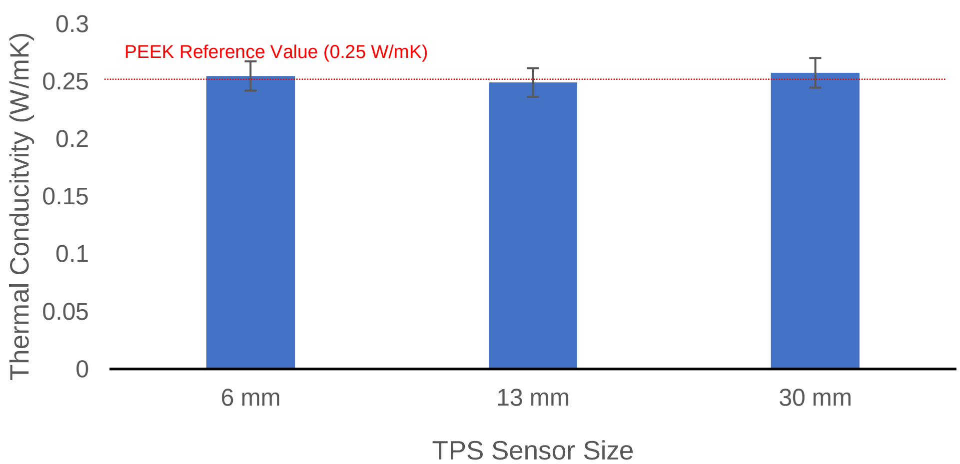

Figure 6 – PEEK Thin Film Measurements Using Different TPS Sensor Sizes.

The thin films utility offers a robust and widely applicable option for characterizing thin films with very low error and good reproducibility. As seen from the comparison study, low error can be expected with excellent accuracy for measuring a variety of industrially relevant thin film materials. The *Thin Film* utility is offered on C-Therm Trident instrument as an option for testing thin films.

To learn more about C-Therm's Trident thermal conductivity instrument, visit www.TridentThermalConductivity.com

[i] ISO 22007-2 Plastics – Determination of thermal conductivity and thermal diffusivity – Part 2: Transient plane heat source (hot disc) method
[ii] Kapton C Datasheet – DuPont Europe
[iii] High Density Polypropylene Datasheets – McMaster Carr.
[iv] Cellulose Acetate Polymer Film Datasheet – Good Fellow Materials.
[v] Mylar Datasheet – DuPont Europe.