Radiation qualification of thermal interface materials for detector cooling

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Silicon sensor based particle detectors operated in a hadronic radiation environment need to be cooled to counteract the radiation induced leakage current and to prevent thermal runaway. To achieve this most efficiently, a low thermal resistance is required between the detector modules and the cooling structures. In many cases dry thermal contacts are sufficient, but especially for a large-area contact so-called thermal interface materials (TIM), largely availably on the market in many forms, are the preferred choice. However, in the use case for detector cooling there can be many requirements, such as non-liquid phase, no heat cure, low thermal impedance, no compression force, radiation hardness, making it challenging to find a suitable TIM. One option are room temperature curing two component thermal gap fillers. Three different types have been tested thermally and mechanically in this campaign. The thermal test setup determines the thermal conductivity of a test sample by measuring the temperature gradient with a controlled amount of heat flow through a sample. Mechanical tests are required to qualify the structural integrity of the thermal interface under thermal stress and mechanical stress. Resembling the style of an ISO 4587 lap shear test, and an ISO 25217 mode-1 fracture test, test samples were prepared with a large 5 × 5 cm² adhesion overlap using plasma cleaned carbon fibre plates to have a surface comparable to its intended use case. After testing of unirradiated samples, they have been irradiated to 600 kGy. The measured mechanical and thermal properties are presented and the results before and after irradiation are compared.
1. Introduction

Heat extraction of delicate detector components is critical for longevity in a high radiation environment. Particle detectors need to be cooled efficiently and for that to be thermally coupled to a cooling mechanics. Many commercial solutions of thermal interface materials (TIM) are not viable, as detectors may have specific requirements. For example heat curing or pressing on the detector might not be possible. Radiation resistance is typically not relevant and therefore not specified by manufacturers and must be assessed.

The goal of this study is the radiation qualification of potential TIMs for the use at the LHC experiments. Next to the radiation deterioration of the thermal conductivity, the change in mechanical properties could lead to a surface detachment and hence increased thermal resistance. Thermal tests have been devised to measure the deterioration of the material properties and mechanical tests were performed to study a potential attachment failure due to weakening of the material.

As candidate materials Thermal Gap Fillers have been chosen, which are typically silicone-based two component room temperature curing materials. As silicone is detrimental to electrical components these products without silicone or designed for low silicone outgassing have been chosen:

- Bergquist/Henkel TGF3500LVO – 3.5 W/(m·K) – Low silicone volatility
- Bergquist/Henkel TGF4500CVO – 4.5 W/(m·K) – Controlled silicone volatility
- Bergquist/Henkel TGF3000SF – 3 W/(m·K) – Silicone free

2. Irradiation

All thermal samples were tested before and after irradiation. Since the mechanical tests were destructive in nature, two sample sets per material were prepared, unirradiated and irradiated, respectively. A target dose of 600 kGy was chosen in this first irradiation campaign. The gamma irradiation was performed with a $^{60}$Co source at the Ruder Boskovic Institute, Zagreb (Croatia). A sample holder was designed to allow air flowing between the samples to avoid excessive heat due to the high dose rate and to prevent ozone buildup.

3. Mechanical qualification

The adhesive properties of TIMs are often weak, resulting in forces too low to be measured properly. Hence standard adhesive tests can’t be used. To increase the adhesion forces, a much larger contact area is used. The tests are meant to represent, to the closest extent possible, the real use case of the material under investigation. For this reason, all samples tested in this study were manufactured joining He/O plasma cleaned carbon fibre plates with a contact area of $50 \times 50$ mm$^2$ and with 0.2 mm TIM thickness. Two kind of mechanical tests were performed:
**Shear test** A test similar to ISO 4587 lap shear test [4] with modified sample geometry. The pull force is parallel to the plane of the TIM material. A photo of a sample being measured is shown in figure 1a. Results before and after irradiation are shown in figure 2. The peak breaking force for the silicone based variants (TGF3500LVO and TGF4500CVO) is increased by about one order of magnitude due to irradiation. The silicon free variant does not show a significant change.

**Fracture test** A test similar to ISO 25217 mode-1 fracture test [5]. The pull force is orthogonal to the plane of the TIM. A photo of a sample being measured is shown in figure 1b. Results before and after irradiation are shown in figure 3. Before irradiation the material is structurally weak and a cohesion failure occurs for all three variants. After irradiation, the silicone based variants are hardened and more stable with increase breaking forces, leading to an adhesion failure. Figure 4 shows the image of the breaking pattern before and after irradiation. Irradiated TGF3000SF still shows a cohesion failure pattern after irradiation consistent with the significantly reduced breaking force.

4. Thermal qualification

The TIM is applied between $30 \times 30 \times 2\text{ mm}^3$ Al carrier plates at various thicknesses. Aluminium was chosen as a carrier for its high thermal conductivity in order to minimise its contribution.
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Figure 3: Fracture test results before and after irradiation of all three gap filler variants.

Figure 4: Fracture patterns before and after irradiation

to the measured thermal resistance. Wires with a diameter of the target TIM thickness (0.2 mm, 0.3 mm and 0.4 mm) are used as spacers and removed after curing. The edges of the samples are coated with epoxy to increase the mechanical stability. Samples are pre-conditioned with 70 N of force to obtain the thickness in measurement condition. A photo of a sample is shown in figure 1c.

The thermal resistance through the sample is measured by sandwiching it between a heated and a chilled brass block, creating a controlled amount of heat flow. Temperature sensors are used to measure the heat flow and the temperature delta through the sample. Details of this setup are described in [6].

4.1 Relative degradation from irradiation

The measured thermal resistance ($r$) is compared before ($r_{\text{unirr}}$) and after irradiation ($r_{\text{irr}}$). A relative residual thermal conductivity (degradation - $d$) is defined as:

$$d = \frac{r_{\text{unirr}} - o}{r_{\text{irr}} - o}$$

The offset ($o$) is the thermal resistance of the Al plates used to make the samples and the thermal grease used to couple the sample to the test setup. In the degradation results shown in figure 5a an offset of $6 \times 10^{-5}$ K·m$^2$/W is used, which is based on direct measurements of the thermal grease and the Al plates. For TGF3500LVO the samples degraded only about 10 %, while TGF3500CVO and TGF3000SF showed a significant degradation. For TGF3000SF the 0.2 mm samples are significantly more degraded than the thicker ones, possibly due to its degradation of mechanical stability.
4.2 Thermal conductivity results

The slope of the thermal resistance as function of sample thickness gives the thermal conductivity of the TIM. The sample set consistency for TGF4500CVO and TGF3000SF was not sufficient to obtain quantitative thermal conductivity results. The measured thermal conductivity of TGF3500LVO matches its specification before irradiation (fig. 5b). After irradiation the measurement shows a reduced value of about 3 W/(m·K) (fig. 5c). Given the measurement error, the result suggest a reduced thermal conductivity of about 15%.

5. Summary

After irradiation to 600 kGy, silicone based gap fillers (TGF3500LVO, TGF4500CVO) show significantly hardened properties resulting in an increased adhesive bond. A problematic surface detachment due to the hardening was not observed. The fracture test designed to probe for cohesion failure showed adhesion failures, however at increased breaking forces. The silicon free gap filler material (TGF3000SF) did not show hardening, with a decreased structural strength. Thermally, TGF3500LVO deteriorated only about 10 - 15% due to an irradiation to 600 kGy. TGF4500CVO and TGF3000SF have a noticeably higher thermal resistance after irradiation.

The measurements presented here were performed at the Detector Assembly Facility (DAF) at DESY Hamburg (Germany).

References