## MICROCRACKING OF CFRP COMPOSITES DURING CRYOGENIC THERMAL CYCLING

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## ABSTRACT

With the fast-approaching targets to decarbonise industry, the use of cryogenic liquid hydrogen (LH2) as a zero-carbon fuel is gathering increased attention. For LH2 to be practical as a fuel, consideration must be given to methods of storage. The aviation sector is one of the key drivers for the use of LH2 due to its high gravimetric energy density, hence aircraft manufacturers have made commitments to have commercial LH2 fuelled planes flying by 2035 [1]. For LH2 to be a viable fuel for commercial aviation, tank mass must be reduced, and the gravimetric efficiency of storage solutions increased [2]. Currently metallic tanks are the primary method for cryogenic fluid storage, however upwards of 40% weight savings have been predicted by utilising carbon fibre reinforced polymer (CFRP) tanks [3].

LH2 is usually stored at low pressures, in the region of 1-12 bar, and at very low temperatures, 20 K (-253 °C) [4]. Utilising composite materials in applications that cover large changes in temperature is challenging, not least due to differences in coefficients of thermal expansion (CTE) of the constituents. In CFRPs the carbon fibres have a near zero CTE, whereas polymers tend to have much larger CTEs [5]. When cooled to cryogenic temperatures the mismatch in CTE causes internal stresses to develop which, along with the embrittlement of the polymer, may lead to microcracking [6]. Microcracks both reduce the structural performance and provide leakage paths through the material, causing tank failure.

To develop CFRP LH2 tanks, potential composite materials must be screened for their resistance to microcracking when cycled between the upper and lower operating temperatures of the tank. The reports in the literature generally describe a procedure that involves cooling the material to a cryogenic temperature, warming it to ambient (or above ambient temperature) and inspecting for microcracks. The reported works used different cooling and heating methods [7, 8], which provided varying heating and cooling rates and different upper and lower temperatures [9]. Several coupon sizes and shapes have been trialled [10, 11], which also affects the cooling rates throughout the samples. Nevertheless, it is important to note there is no standard approach for such tests, moreover very little work has been carried out to compare the effect of different test variables and specimen configurations.

The purpose of the work described in the paper is to systematically investigate the effect of cooling rates, temperatures, and coupon geometries to establish a standardised test methodology for screening materials for composite LH2 tanks. The paper describes the approaches that have been used to provide a range of cooling rates and temperatures alongside a range in sizes and shapes of coupons. The primary cooling methods investigated are liquid nitrogen (LN2), temperature 77 K, and liquid helium (LHe), temperature of 4 K. The rate of cooling was investigated through either directly immersing coupons into the cryogenic fluid for maximum cooling rate or through cooling the coupons in the vapour phase of the cryogen to achieve a predefined rate of cooling.

Three sizes of square flat plate coupons were tested with edge lengths of 25 mm, 75 mm and 150 mm. 150 mm diameter pipe sections were also tested at around 300 mm length. Three material systems were used with different expected levels of microcracking. Inspection was carried out by both optical microscopy of polished edges and X-ray computed tomography (XRCT) after of thermal cycling. Figure 1 shows a comparison between a micrograph and XRCT scan taken on the same specimen.

It was shown that cooling rate had a larger impact on microcracking than absolute temperature, with the coupons immersed in LN2 exhibiting more microcracking than those cooled in LHe vapour. In immersion, the flat plate specimens of 150 mm edge length appeared to cool unevenly and had evenly distributed microcracking on one side of the coupon that was not observed in other coupon geometries. It is proposed that for screening purposes, LN2 immersion of flat plate coupons with 25 mm edge length is a fair first screening test set up as the test is relatively fast, low cost and showed the most differentiation in microcracking behaviour between the material systems compared to the other test methods.



Figure 1: Comparison of XRCT scan and edge micrograph of microcracks in 25 mm Carbon-Untoughened Epoxy specimen subjected to 20 cryogenic thermal cycles between 77 K – 323 K through immersion in LN2.

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