

MICROCRACKING OF CFRP COMPOSITES DURING CRYOGENIC THERMAL CYCLING



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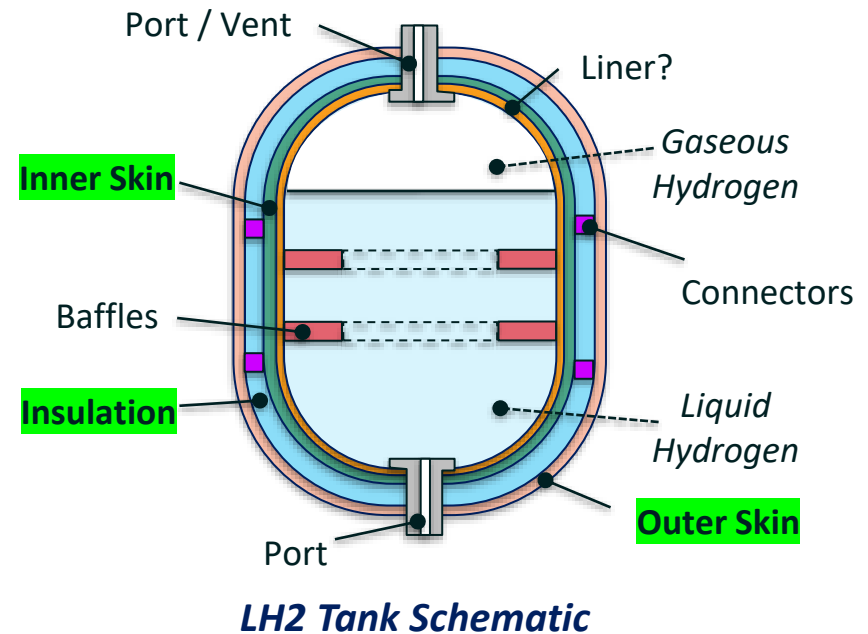
Hydrogen as an Alternative Fuel

Relative Mass And Size of Different Fuels

	Gravimetric energy density	Volumetric energy density
Jet fuel	1 (1)	1
Lithium-ion battery	60	18
700 Bar Hydrogen	0.35 (7)	8
Liquid Hydrogen	0.35 (0.5)	4

Values in brackets account for tank weight assuming composite hydrogen tanks.

Why Liquid Hydrogen?
Minimised mass and size compared to other alternatives



Why Composites?
Literature and modelling predict around 50% weight saving for composite inner skin over a Type I Al-Li baseline. (Vickers, 2013)

Stresses in Cryogenic Composite Tanks

Thermal Stress

Coefficient of Thermal Expansion (CTE) is a challenge in composite materials as the constituents can have opposing CTE.

- **Carbon Fibres** - Negative CTE axially – Elongate on cooling.
- **Polymer Matrices** - Positive CTE – Contract on cooling.
- **Ply Level** interaction between fibres and matrix.
- **Laminate level** interaction between plies at different angles.

Mechanical

- Internal Pressure – in the region of 2-10 bar
- Fluid sloshing during 9g crash case

Role of constituents in composite LH2 tank

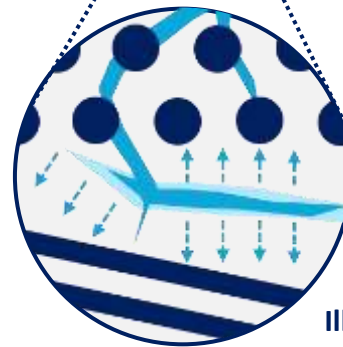
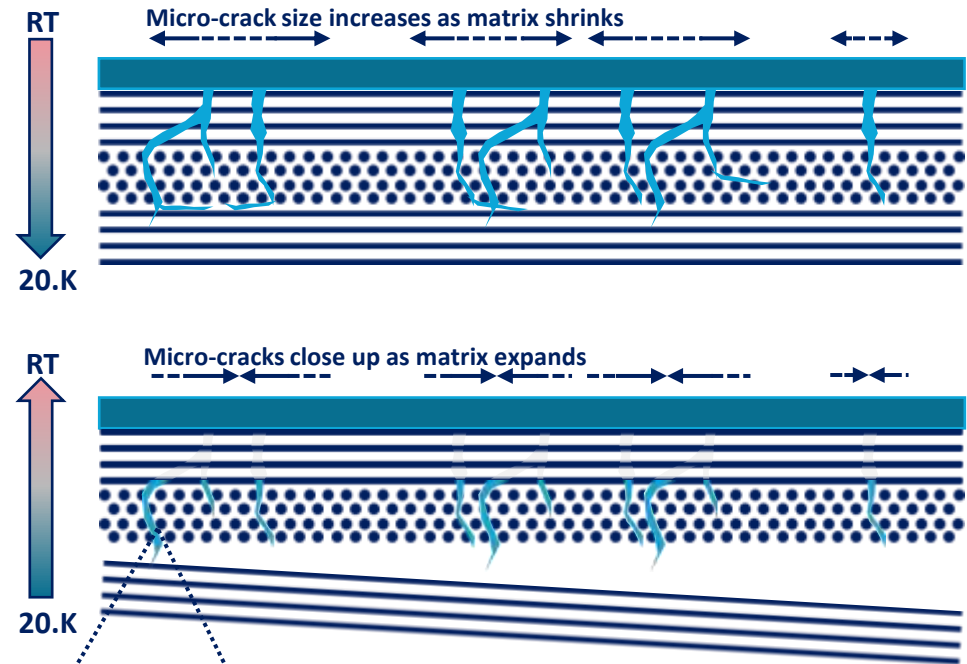
Fibres experience the majority of the mechanical stress from the pressure loading.

Matrix experience transverse thermally induced stress from the fibres expanding.

Adding more fibres does not improve performance, it increases the transverse matrix stresses!

Micro-Cracking

- Constituents expand and contract relative to each other leading to Internal stresses.
- Polymers become brittle at cryogenic temperatures.
- Material damage occurs through matrix microcracking.



If LH_2 becomes trapped in the composite during thermal cycling, it can boil, potentially leading to catastrophic delamination/rupture of the tank.

Illustration of Micro-Crack Development During Cryogenic Cycling

temperatures.

The Microcracking Problem

A fully linked network of microcracks in a tank will enable permeation of hydrogen

Any surface microcracks may cause catastrophic failure if hydrogen becomes trapped during warming

Image credit: NCC/Jenny Banks

Cryogenic Composite Screening Methods

- **Resistance to cryogenic microcracking** - The primary factor for assessing the suitability of composite materials for use in cryogenic tanks.
- **Thermal cycling followed by microcrack inspection** - The most common screening method In literature.
- Thermal Cycling Methods Vary in literature.

Cooling fluid

- LN₂
- LHe

Heating method

- Elevated
- Ambient

Cycle temperature

- Lower temperature
- Upper temperature

Cooling Method

- Immersion
- Vapour cooling

Cycle time

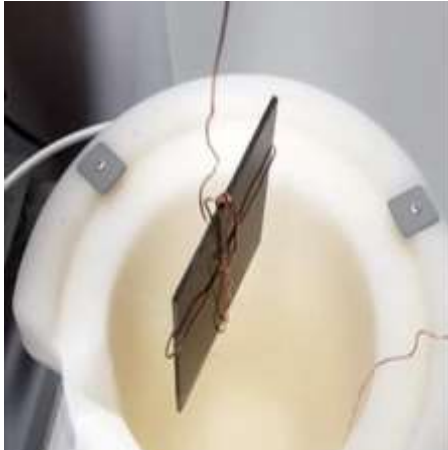
- Cooling rate
- Heating rate

Coupon geometry

- Shape
- Size

Areas Investigated

Cooling Methods

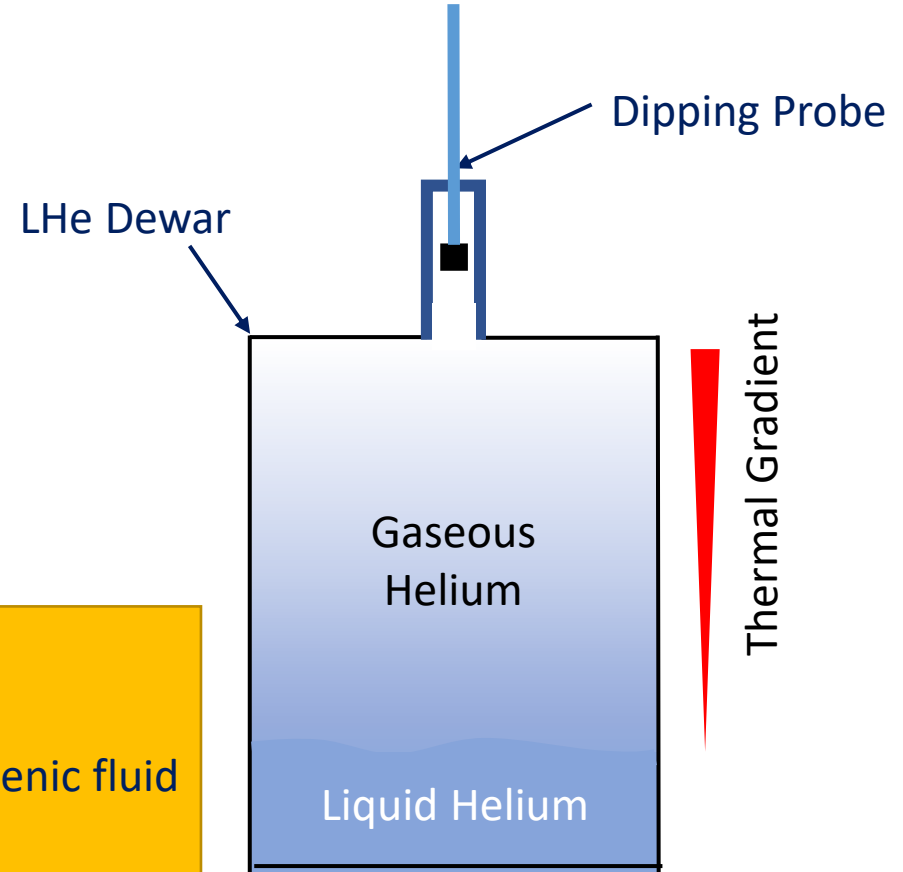


Immersion

No cooling rate control – thermal shock
Lower temperature fixed to that of the cryogenic fluid

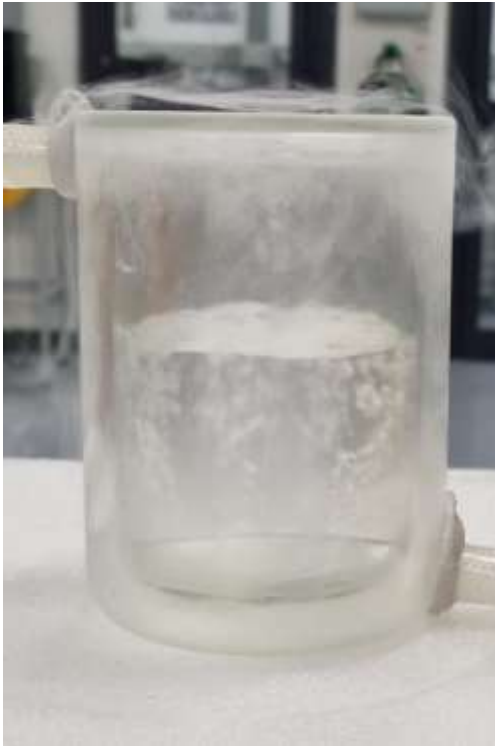
Vapour Dipping

Control of cooling rate and lower temperature



LHe Vapour Dipping

Film Boiling in immersion



Double Walled Glass Beaker



Video of film boiling on coupon

Film Boiling

Cooling rate influence by film boiling, reduces heat transfer between fluid and object

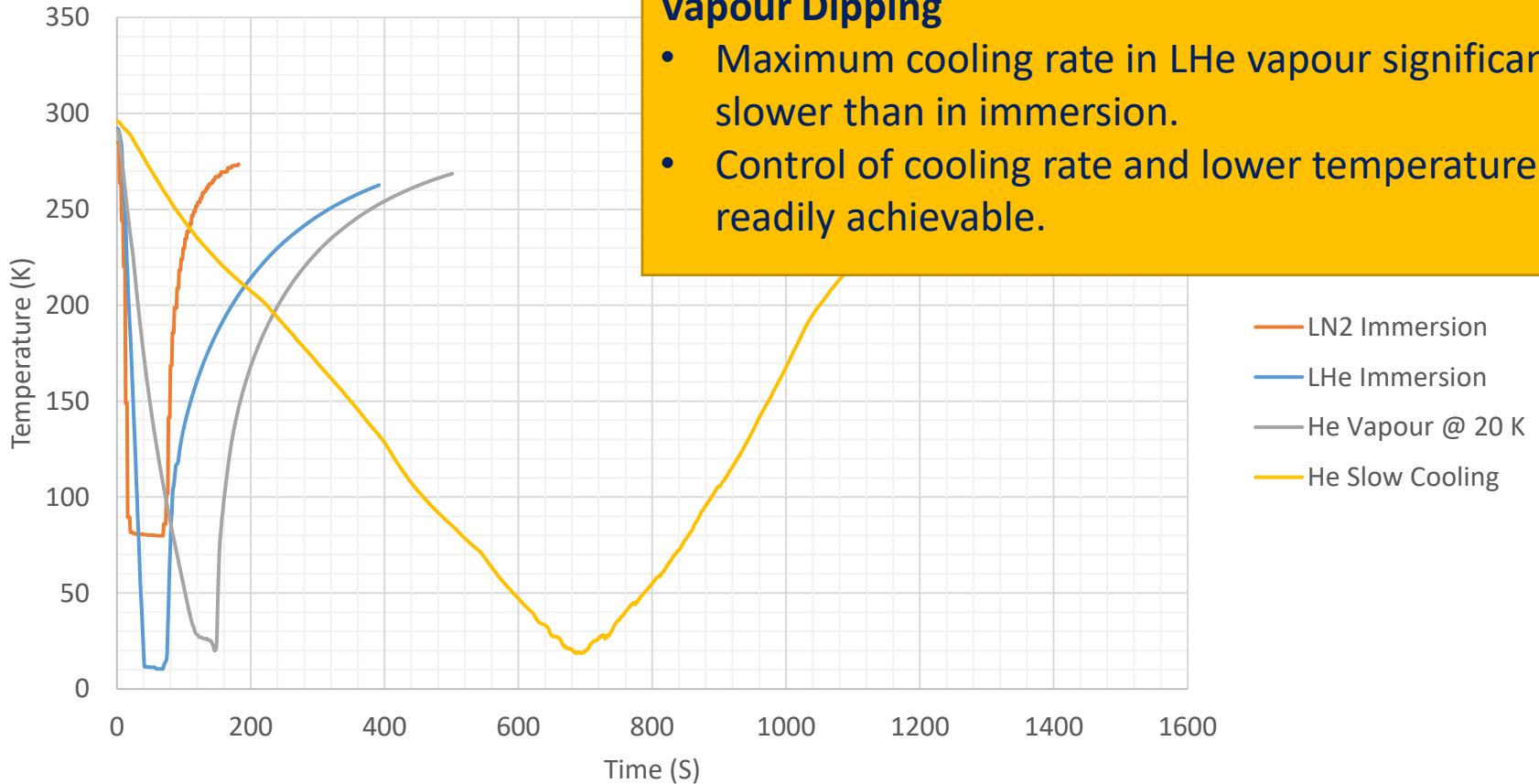
Cooling Methods

Immersion

- LN2 has faster cooling rate than LHe

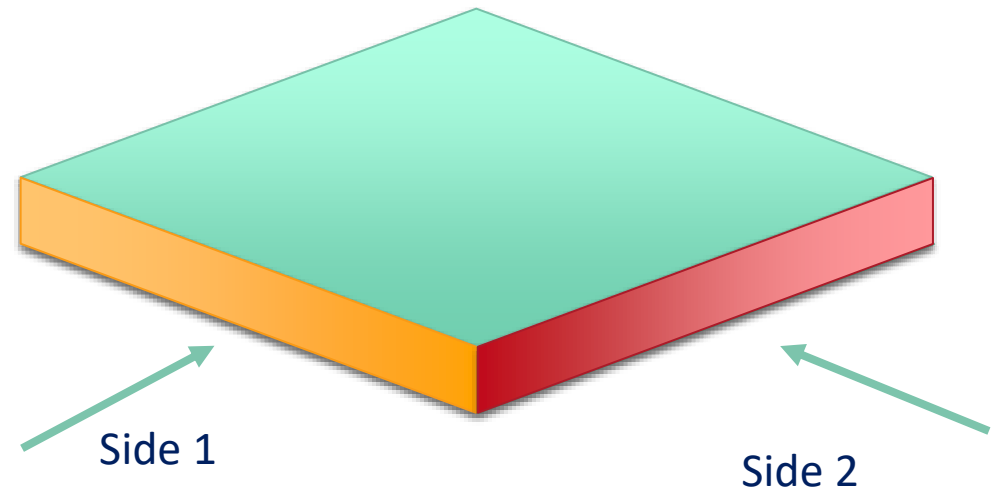
Vapour Dipping

- Maximum cooling rate in LHe vapour significantly slower than in immersion.
- Control of cooling rate and lower temperature readily achievable.



Specimens

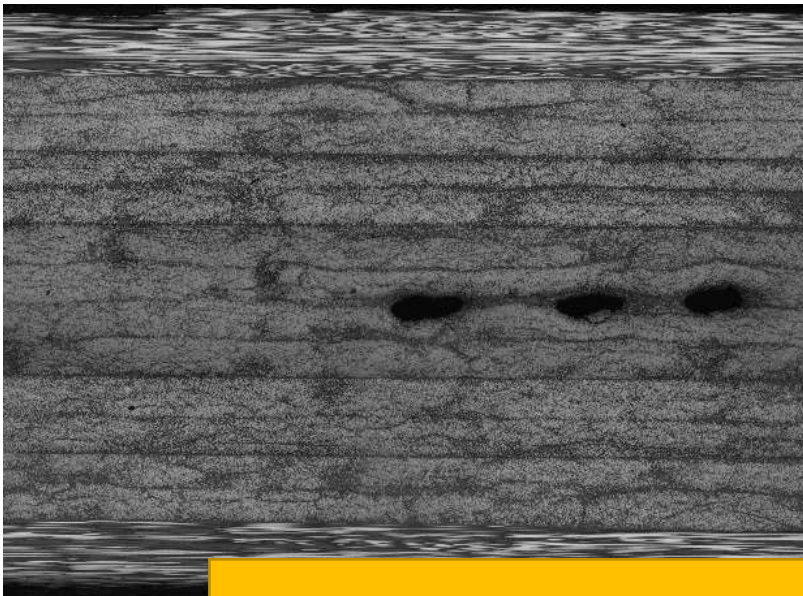
- Plate specimens with two polished edges to inspect for microcracks with optical microscopy
- Layup
 - 16 Ply QI Blocked
 - $[0_2/45_2/-45_2/90_2]_s$
- Sizes
 - 25 mm x 25 mm
 - 75 mm x 75 mm
 - 150 mm x 150 mm
- Materials
 - Multiple thermoset and thermoplastic materials tested



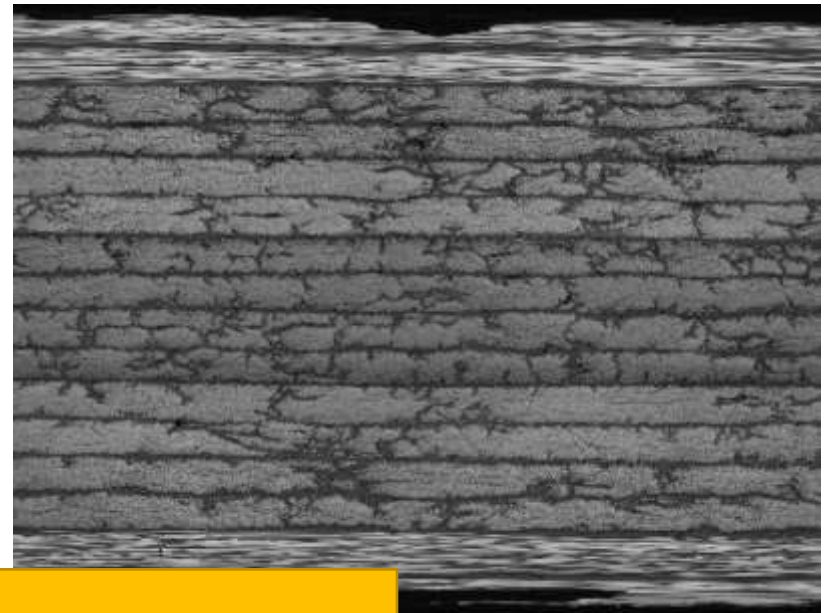
Two Materials Presented

Initial

Low Quality



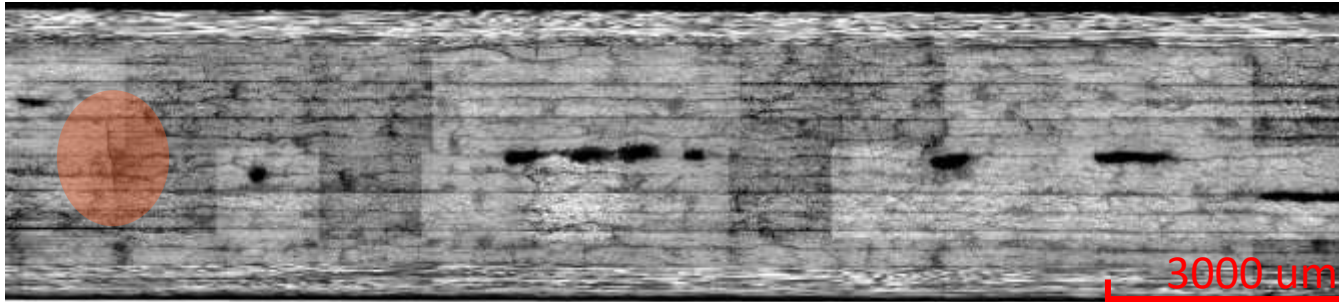
High Quality



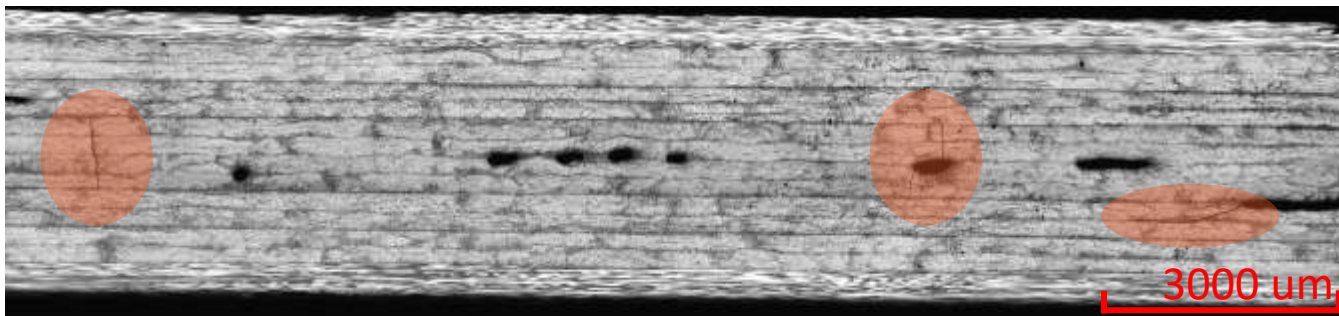
Low Quality material has more porosity / Voids
High Quality material has increased toughening

Effects of LN2 Immersion – Low Quality Material 25mm

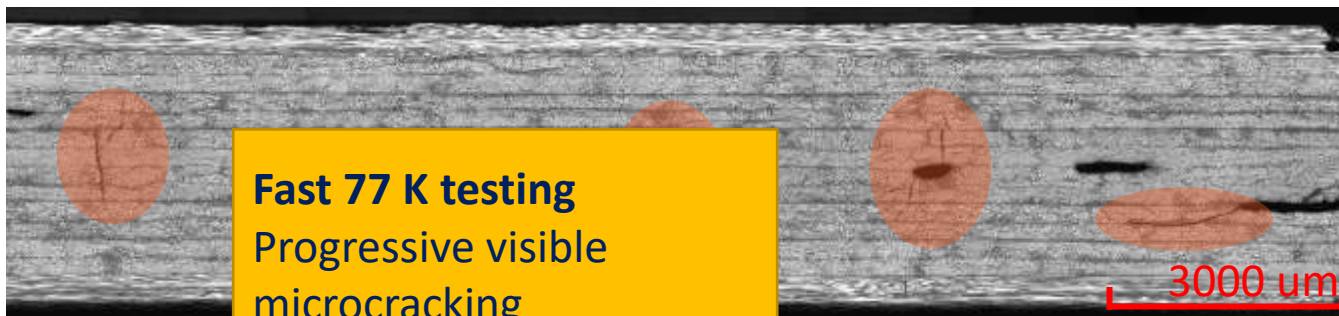
0 Cycles



10 Cycles



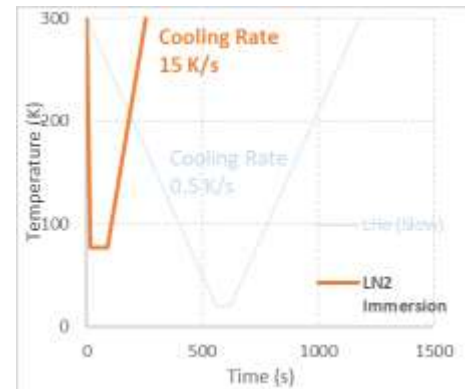
20 Cycles



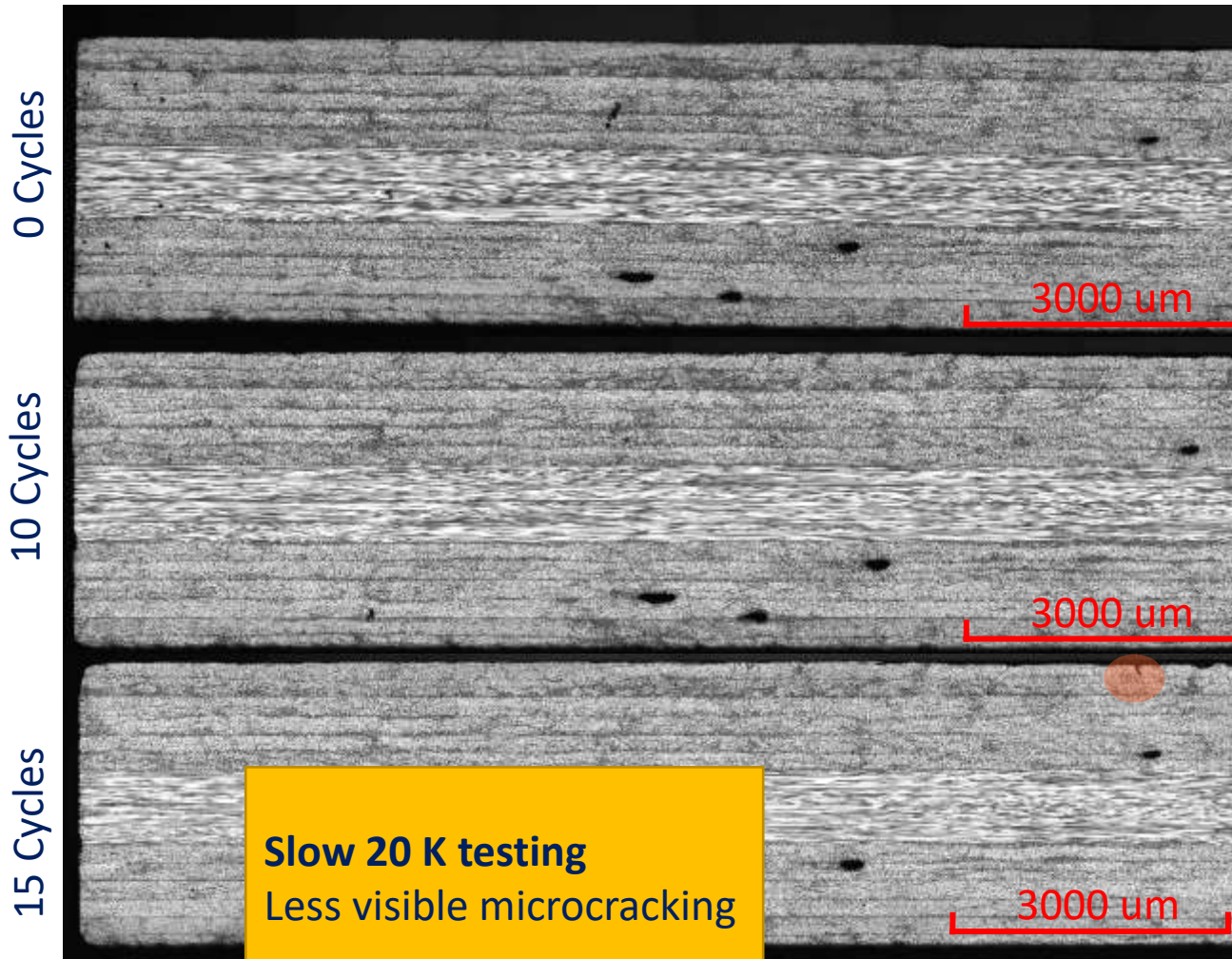
Fast 77 K testing
Progressive visible microcracking

For testing purposes, a low quality material was made with stress raisers and defects to generate microcracking.

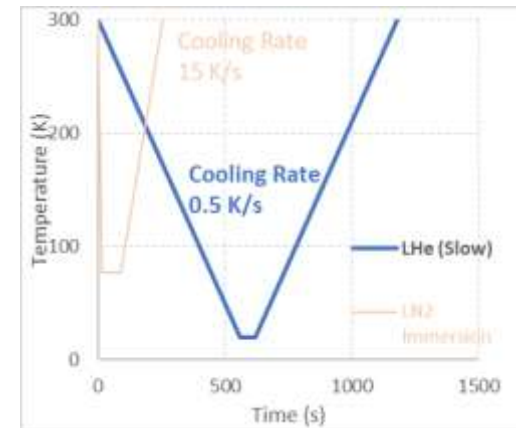
Significant microcracking occurred when cooled rapidly at 15 K/s to 77K.



Effects of LHe Cycling – Low Quality Material 25mm

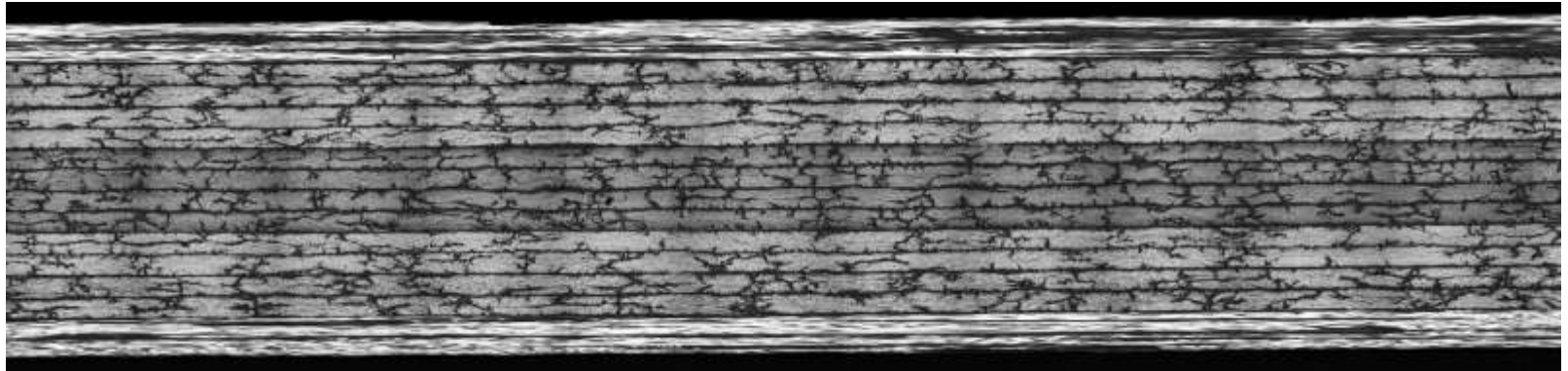


In comparison when cooled slowly at 0.5 K/s to 20K reduced microcracking was visible, in some coupons there were no microcracks.



Effects of LN2 Immersion – High Quality Material 25mm

0 Cycles



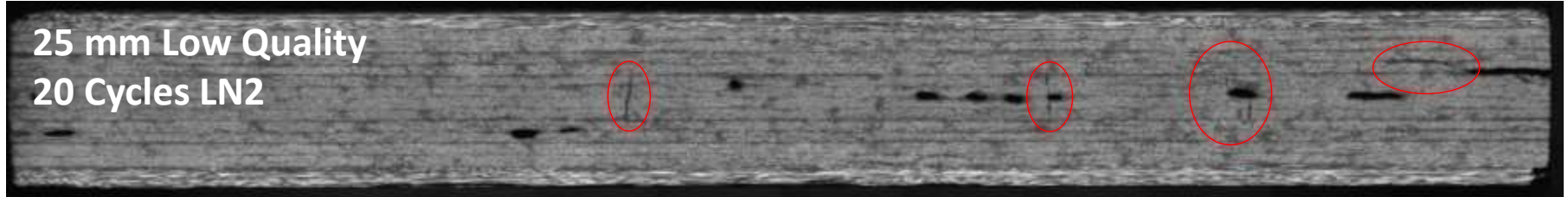
20 Cycles



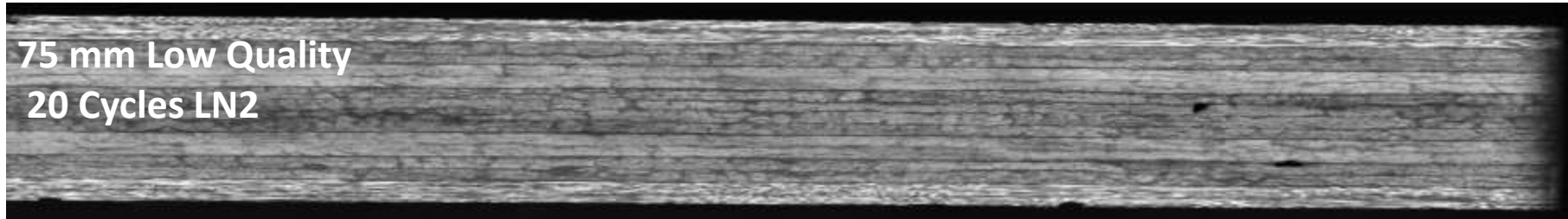
No Cracking in high quality material in any cycling tests up to 20 cycles

Results – Coupon Sizes

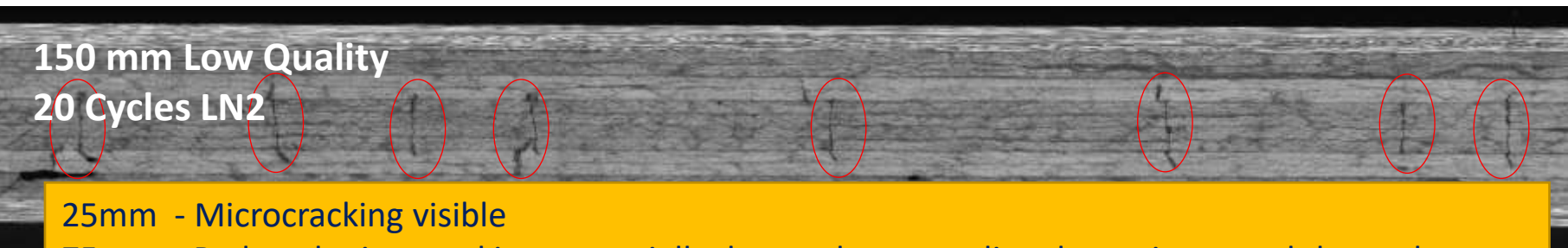
25 mm Low Quality
20 Cycles LN2



75 mm Low Quality
20 Cycles LN2



150 mm Low Quality
20 Cycles LN2



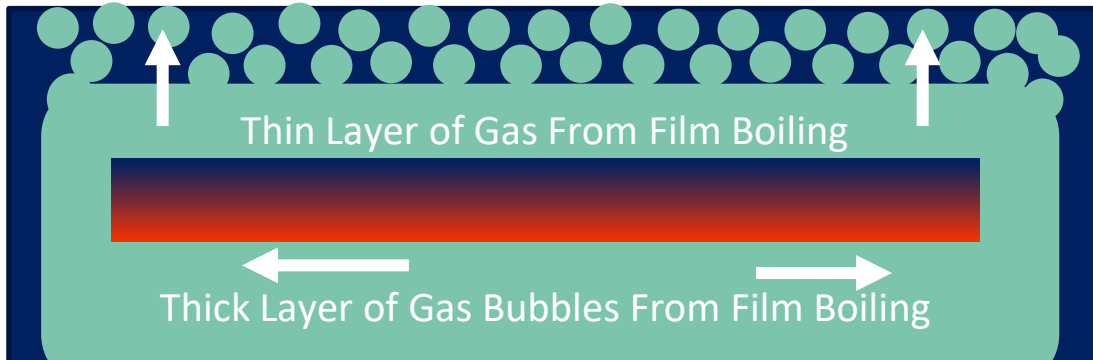
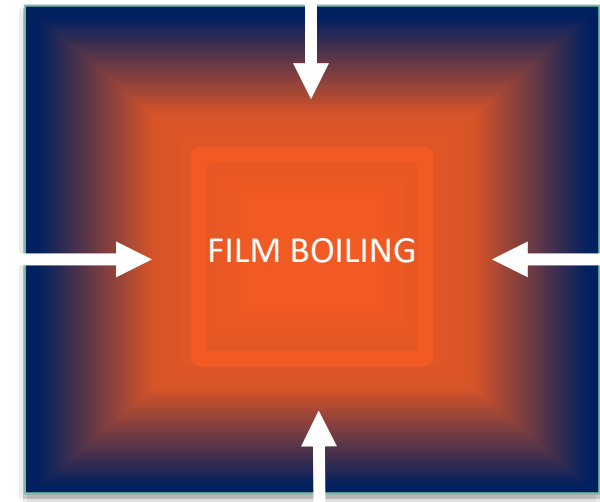
25mm - Microcracking visible

75mm – Reduced microcracking potentially due to slower cooling due to increased thermal mass

150mm – Significant microcracking, likely caused by non-uniform cooling

Cooling of 150mm specimens

- Film boiling recedes from edges to centre in a rectangular pattern.
- Indicate in plane cooling faster than through thickness cooling.
- Leads to temperature differential across specimen causing increased stresses.



- Coupons warped after cooling, cracking dominates on one side.
- Could be caused by cooling specimens horizontally.
- Film boiling could be insulating

150mm - non-uniform cooling can cause thermal gradient leading to significant microcracking

Initial Testing Trends

- For the composite materials tested, rate of cooling appears to have more effect on composite microcracking than absolute temperature, however this may vary from material to material.
- Coupon size has an impact on microcracking behavior under rapid cooling, potentially due to increased thermal gradients within the larger coupon.
- Tough materials may not crack under thermal cycling until several hundred cycles. An automated cycling rig has been developed to enable testing of tough materials to higher cycles.
- In addition to thermal cycling, other cryogenic composite characterisation methods are being developed including a “Cryogenic Microcrack Fracture Test” involving combined thermal and mechanical loading. - *To be presented later in the year*

Questions?

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