Thiol-isocyanate chemistry for autonomous self-healing of epoxy thermosets

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1. Healing kinetics
Model study using online FT-IR spectroscopy
- Fast reaction (minutes) with a high yield
- Catalysed by a tertiary amine, esp. DIPEA
- Atom efficient: No side products generated
- Isocyanate can react with residual amines in the epoxy matrix

2. Healing potential
Healing through manual injection
- Tapered Double Cantilever Beam geometry (TDCB)
- The fracture toughness is independent of crack length
- Razor blade tapping to generate precrack (ASTM D 5045)
- Healing efficiency calculated according to formula below

3. Visualisation techniques
X-Ray Computed Tomography (CT)
- Non-destructive 3D visualisation method
- Analyse shape and distribution of microcapsules in the epoxy matrix (figure bottom left)
- Surface morphology analysis: slice formation (figure right), delamination of microcapsules and matrix (figure top left)...
- Air inclusions in matrix or capsules

Digital Image Correlation (DIC)
- Measuring strain in TDCB samples (see section 3)
- Follow the crack propagation and visualise strain asymmetries due to deviations or defects

4. Autonomous self-healing
Epoxy with microcapsules as containers
- Isocyanate microcapsules made via interfacial polymerisation
- Thiol microcapsules made using polycondensation of melamine-formaldehyde
- Healing efficiency > 50% (5 days) with both microcapsules embedded in the EPON 828 matrix
- Reinforcing effect of the microcapsules on the virgin peak load: increase up to 147% in load-to-fracture
- Healing efficiency increases with the amount of microcapsules (best results with 20 wt%)

Concluding remarks
- The thiol-isocyanate chemistry has been fully characterised and proved to be very promising for self-healing. Several issues with currently existing systems are resolved.
- A healing efficiency > 50% after 5 days is obtained so far. Optimisation is still ongoing.
- An extensive analysis of the mechanical properties of the new material is being performed in collaboration with the project partners. A full understanding of all the parameters is essential for further improvement of the system.
- The epoxy matrix material is easy to handle and adding the capsules does not change the processing conditions.

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