University of BRISTOL

Smart Chemistries for Advanced Composite Materials

ACCIS DTC Conference 2012

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17th April 2012

DOCTORAL TRAINING CENTRE

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ADVANCED COMPOSITES CENTRE FOR INNOVATION & SCIENCE

Outline

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2. **Microcapsule Systems**
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Aerospace materials

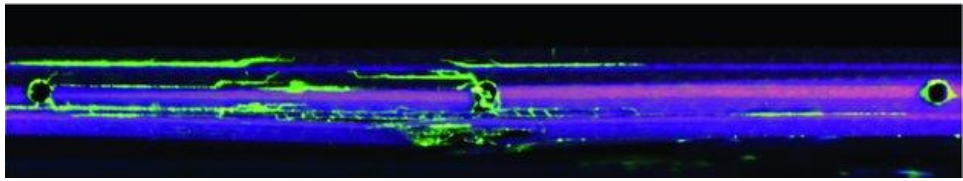
Changing planes

A new breed of aircraft built from lightweight carbon composites is taking flight. But are these materials all they're cracked up to be, asks Hayley Birch

Self-Healing for FRP Composites

- **Barely Visible Impact Damage (BVID)**
  - Difficult, time consuming and expensive to detect
  - Predominantly caused by matrix cracking
  - Microcracking and hidden damage – ***Potentially Fatal***

- **Self-Healing**
  - Damaged structure repaired by reagents contained within the composite material
  - Maintaining structural integrity and material performance
  - Adding to the designed structure



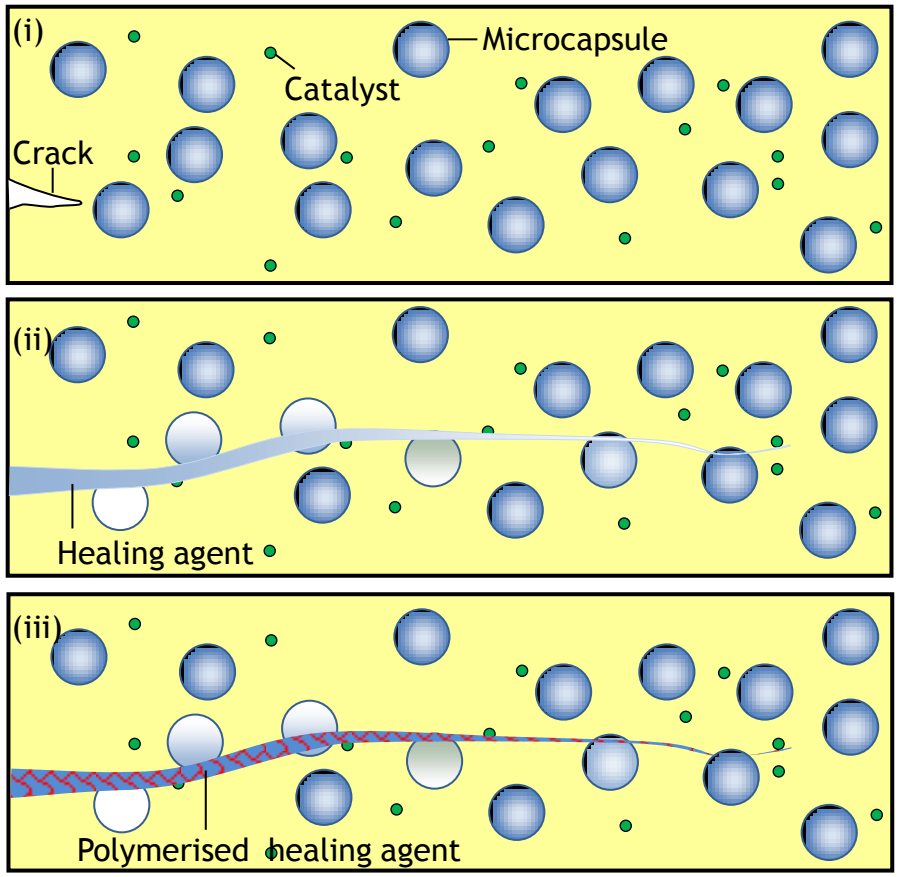
Norris *et al.*, *Adv. Funct. Mat*. **2011**, *21*, 3624-3633

- **Autonomous Systems**
  - Respond to damage; triggering the 'healing' process
  - No external action required

Project Summary

- **AIM:** *<u>Development of novel self-healing agents for composite materials</u>*
  - Relatively **cheap**, **stable** and applicable to **industry** applications (AUTOHEAL – Team MAST)
  - Compatible with existing material
  - Quantify healing performance (**mechanically**)
  - Further development (PhD *to date*)

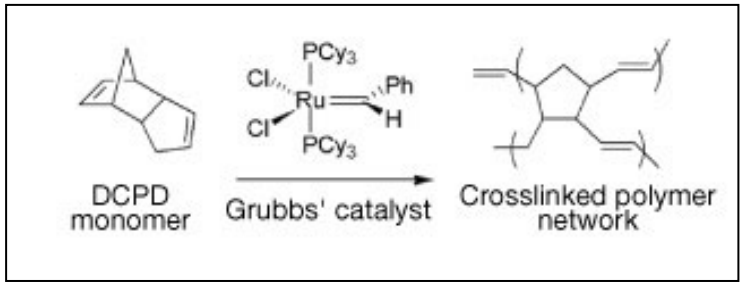
- **Potential applications**
  - Maintain integrity in personal protective equipment
  - Self-healing adhesives for military hardware
  - Anti-corrosion coatings

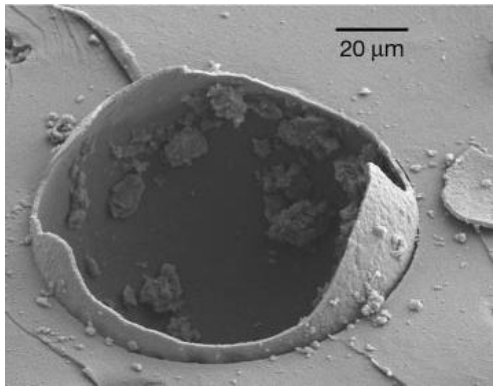


Microcapsule Systems

- **2001:** S. R. White *et al*. "Autonomic healing of polymer composites", *Nature*, **409**, 794-797
  - Ruthenium initiated ring-opening metathesis polymerisation (ROMP) of dicyclopentadiene (DCPD)

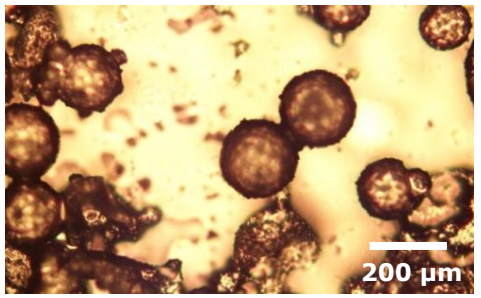
- **Limitations**
  - Expensive reagents; >£250/g
  - Air and moisture sensitive catalyst; *requires wax coating*
  - Poor adhesion between poly(DCPD) and epoxy resin matrix
  - Unable to withstand conventional autoclave composite processing techniques





Microencapsulated Epoxy / Catalyst Selection

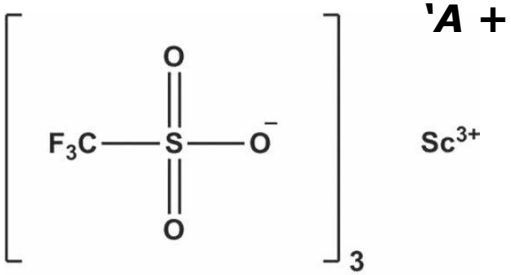
- **In-situ microencapsulation**
  - DGEBA/ethyl phenylacetate (EPA) solution within a poly(urea-formaldehyde) shell
  - Emulsion in H_2O with surfactant stirred at ~50 °C for 4 hours, cooled and filtered
- **Analogues**
  - DGEBA:EPA ratios; 75:25, 50:50, 25:75, confirmed by NMR to within 1%
- **Size**
  - *Optical microscopy:* 150-250 μm in diameter

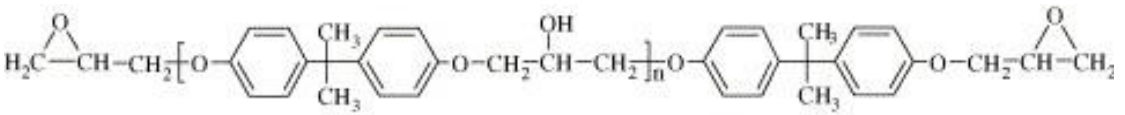


B. J. Blaiszik *et al*. *Polymer*, 2009, **50**, 794-797

- **Active catalyst based on metal salt with weakly coordinating anions**
  - Lewis acid
  - Scandium triflate [$Sc(OTf)_3$]
  - Air and moisture stable
  - Solid fine white powder
  - Melting point >200 °C
  - Relatively non-toxic
  - Catalyst soluble in EPA
  - Non-stoichiometric mixing;

'A + B mixing' not required

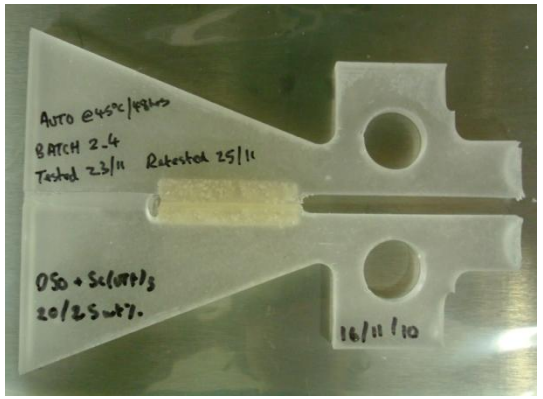


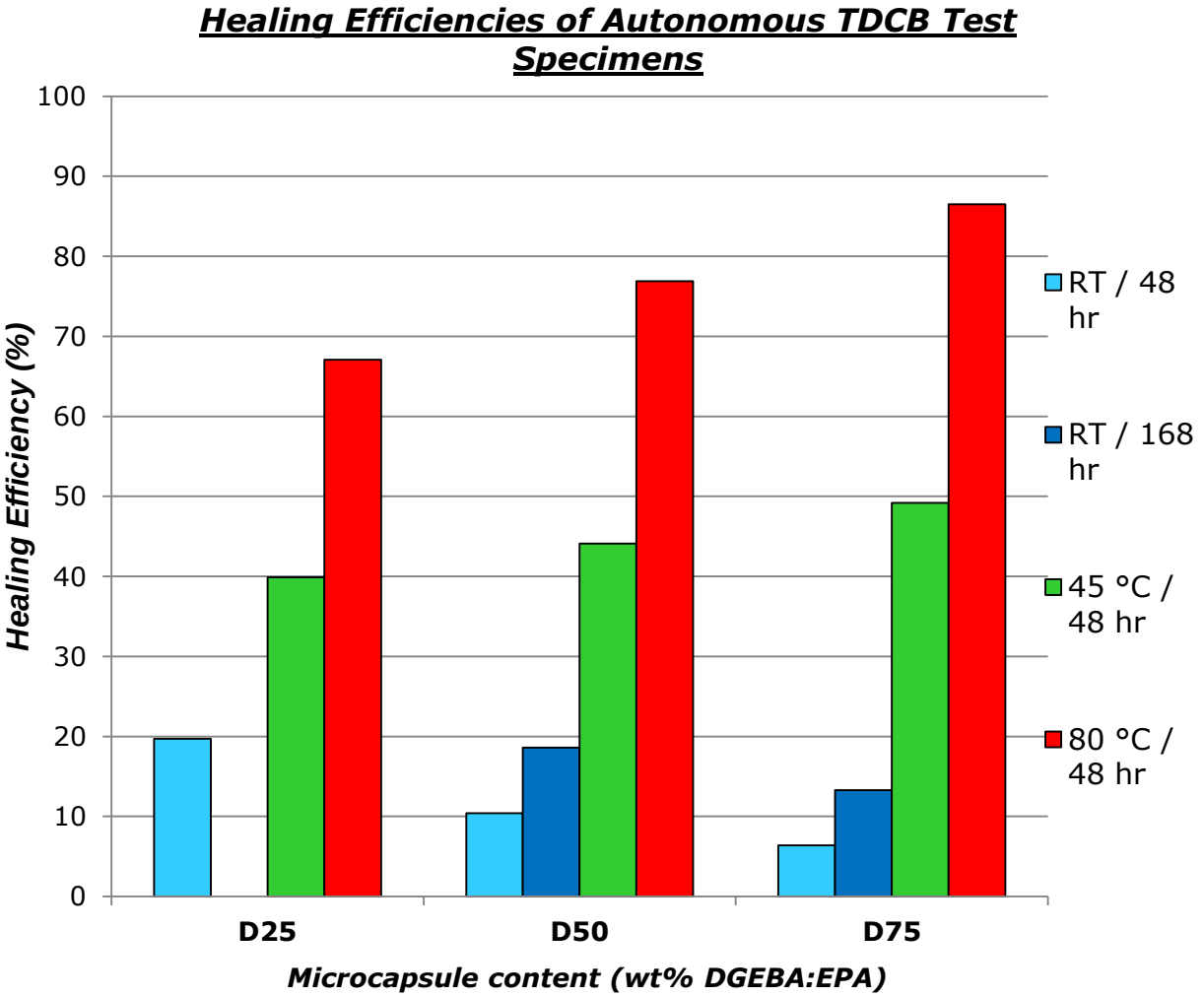


Diglycidyl ether bisphenol A (DGEBA) polymer

Microencapsulated System: Mechanical Testing

- **Tapered Double Cantilever Beam (TDCB)**
  - Principally to demonstrate proof of concept
  - Self-healing agents (SHAs) embedded in an epoxy resin
  - Crack propagation region along a central trench (CT) section
  - Actively optimise 'healing' performance and efficiency



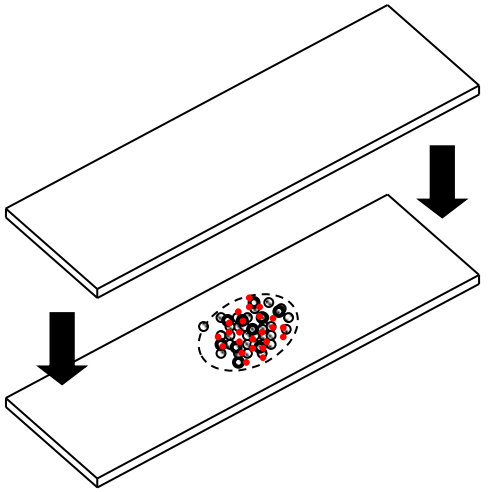


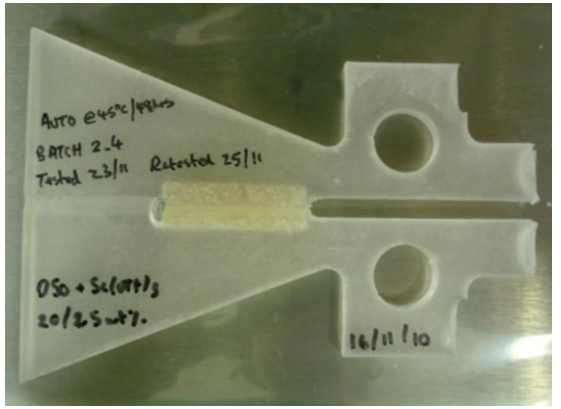
Coope *et al.*, *Adv. Funct. Mat.* **2011**, *21*, 4624-4631

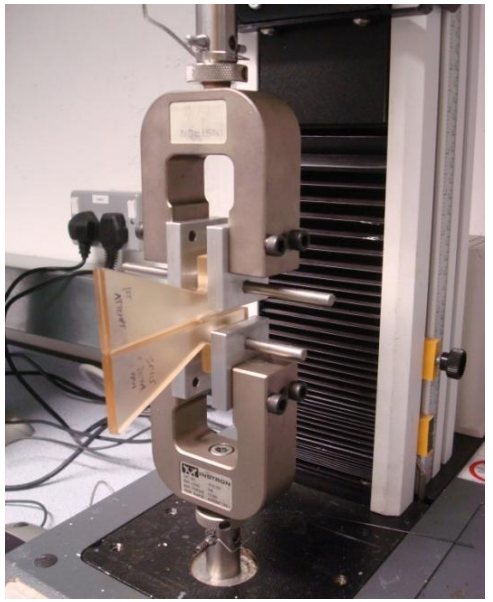
- **Healing efficiency**
  - Significantly increased at a higher temperature over the same time period

Conclusions

- Development and implementation of a novel microcapsule and solid catalyst based self-healing system
- Conclusive quantitative analysis from TDCB testing
  - >80 % fracture strength recovery for a pure epoxy resin
- A stable and tailorable self-healing DGEBA-based material system towards the application, operating conditions and required healing cycle







Acknowledgements

EPSRC
Engineering and Physical Sciences Research Council

Team MAST
BAE SYSTEMS

University of BRISTOL

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