



Advanced Manufacturing Research Centre



Project Part-Financed
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European Regional
Development Fund



Science and Manufacturing: Ingredients for Innovation

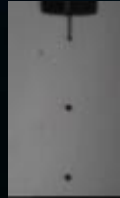
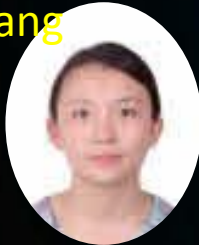
Professor Alma Hodzic
AMRC Research Director
17th December 2013
AFOSR, Washington DC



Self-ameliorating inkjet printed composites for higher survivability

Programme Managers: Dr Lee "Les" Byung-Lip, Sc. D. and Lt Col Randall "Ty" Pollak, PhD

Yi Zhang
ME



Andrew
Cartledge
ME



Hannah
Crunkhorn
AMRC



PhD Candidates

IJ printing & IJPC analysis

Fatigue tests & FEA

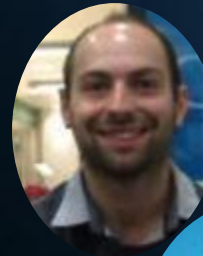
Machining & characterisation

Research Fellows

Dr Jonathan
Stringer, ME



Dr Richard
Grainger, AMRC



Supervisors

Patrick Smith, ME



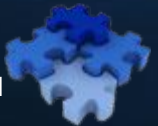
Alma Hodzic, AMRC



Christophe Pinna, ME

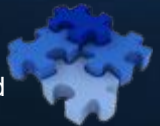
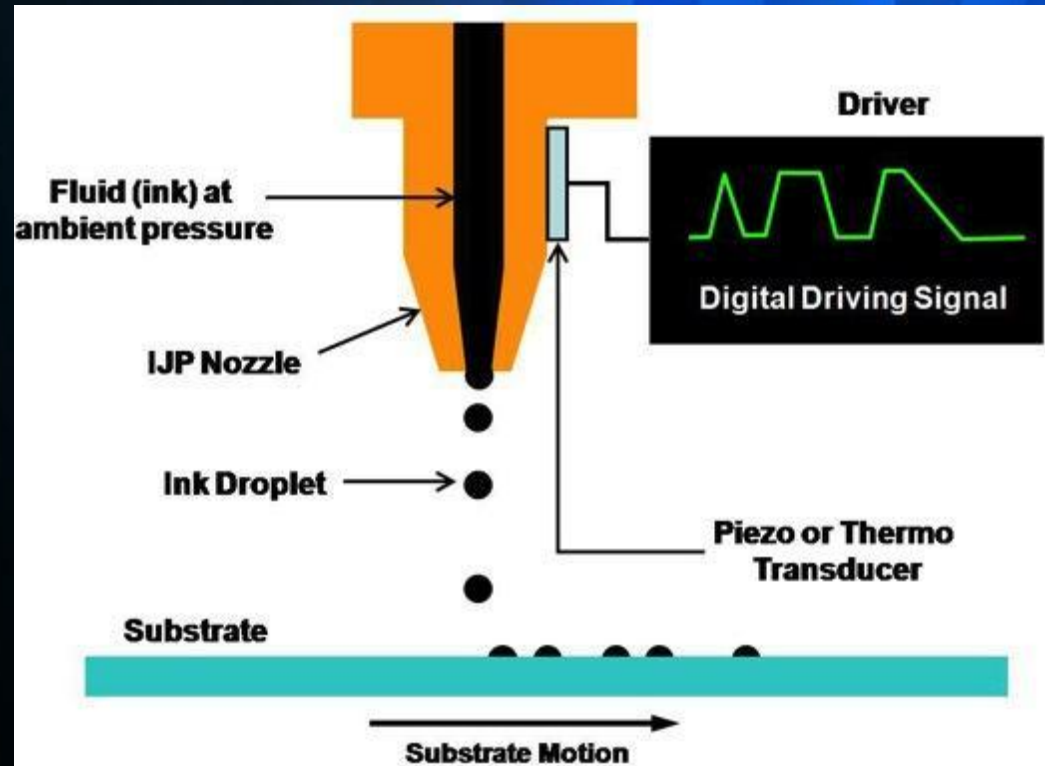


Richard Scaife, AMRC

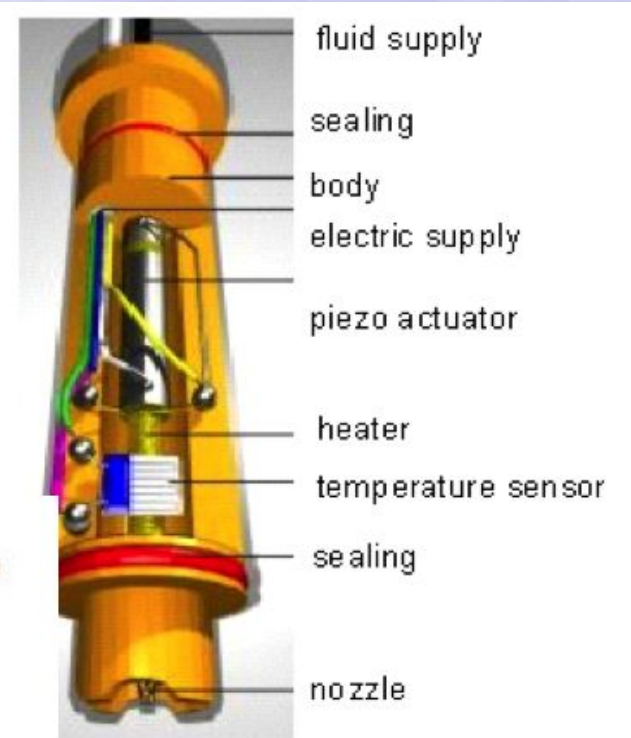
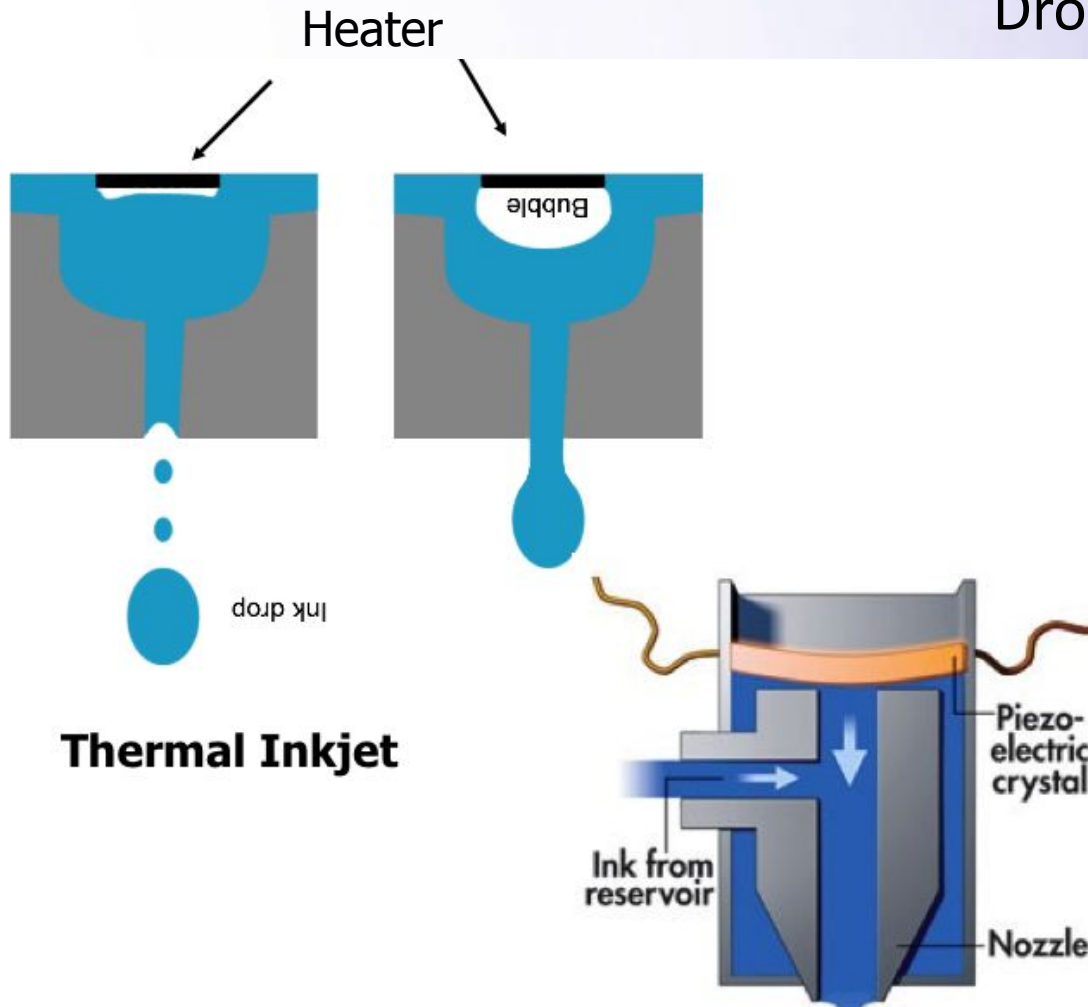


Benefits of Inkjet Printing

- Direct write technology (no masks needed)
- Additive technology
- Droplets of ink ejected from a nozzle to pattern substrate
 - Computer-aided which can pre-define patterns according to requirements
 - Rapid changing between patterns (no down-time)
- Non-contact deposition method (reduces/removes risk of contamination)



Drop on Demand Printheads



Piezo Inkjet



Effect of Z on Droplet Volume

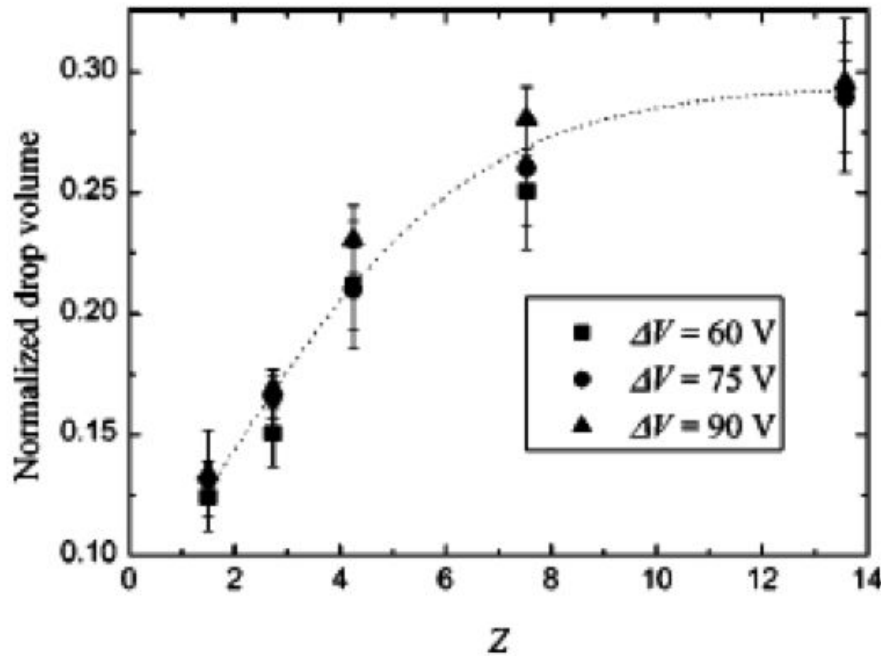
1 < Z < 10
Optimum printing

$$\frac{Re}{We} = \frac{(\gamma \rho a)^{1/2}}{\eta}$$

Droplet volume increases Z as predicted by Fromm

$$Z = (d\rho\gamma)^{1/2} / \mu$$

Droplet volume is normalised to the volume displaced by the actuator at different driving voltages

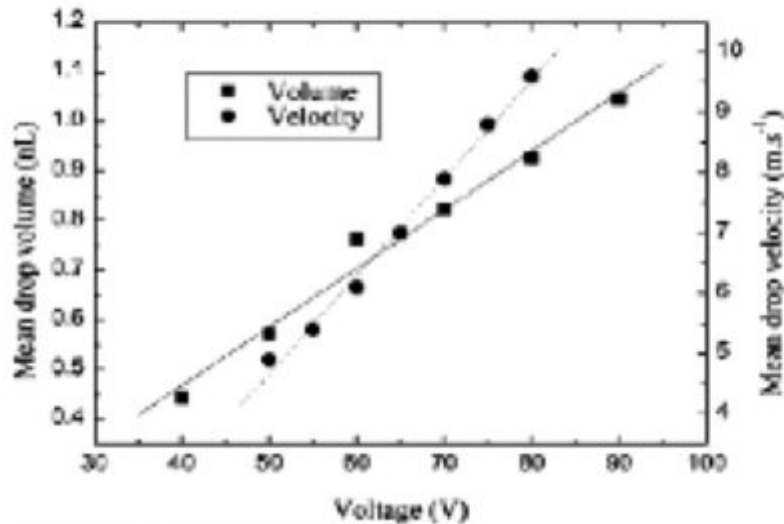


Reis, Derby J. Appl. Phys. 2005, 97, 094903

By varying the ink's rheology (viscosity or surface tension), we can vary the size of the ejected droplet



Influence of Voltage on Droplet Volume



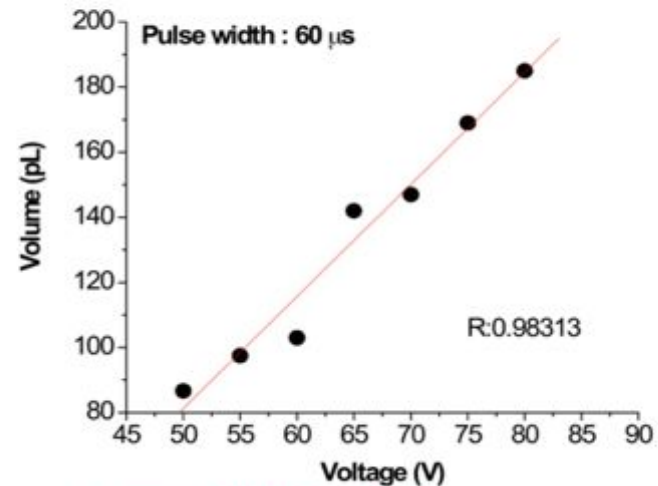
Molten paraffin wax

Reis, Derby, J. Appl. Phys. 2005, 97, 094903

By varying voltage, we can vary the size of the ejected droplet

Applied voltage is proportional to droplet volume

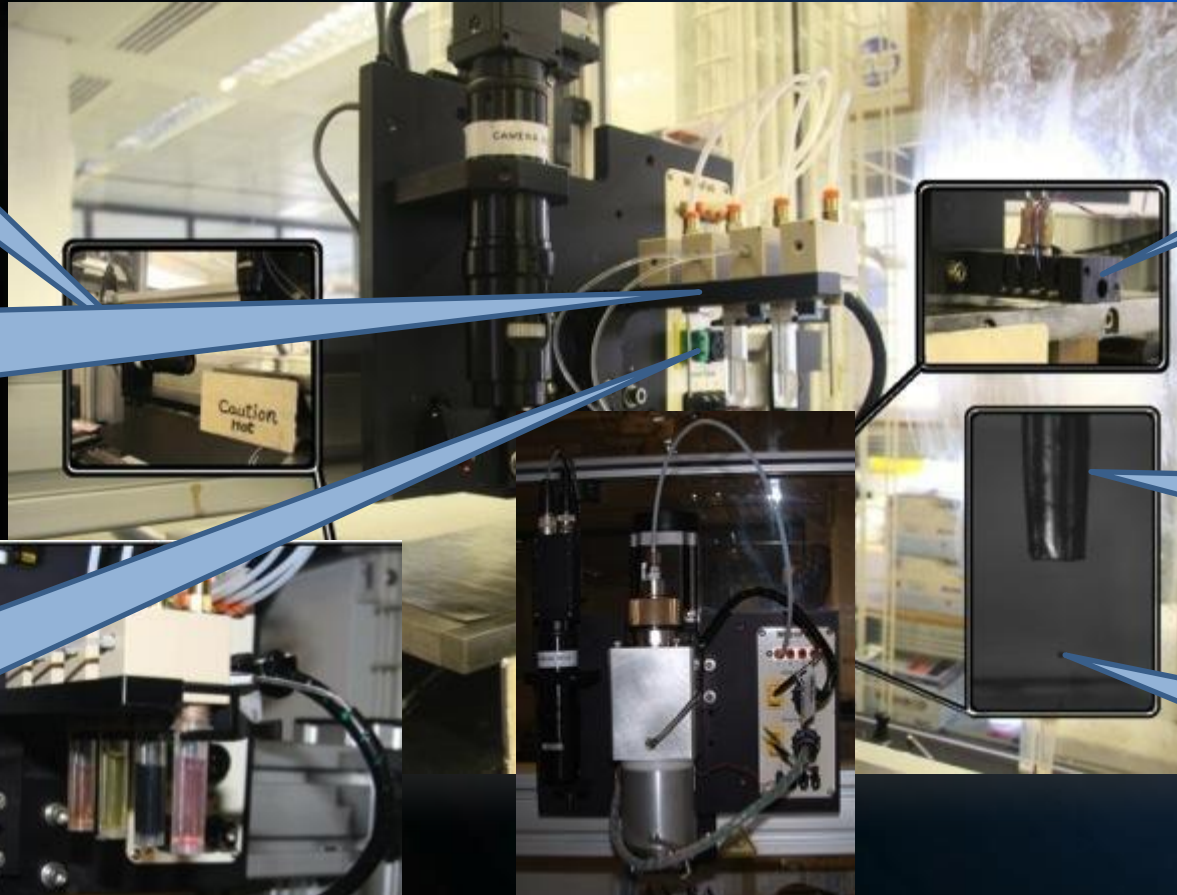
Increased Voltage = Increased Droplet Volume



Aqueous PEDOT solution



Inkjet printer in Sheffield (MicroFab 4, piezoelectric DOD)



Camera

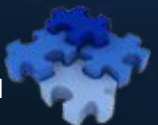
Up to
four
different
inks!

Or one
ink at
high
temp'!

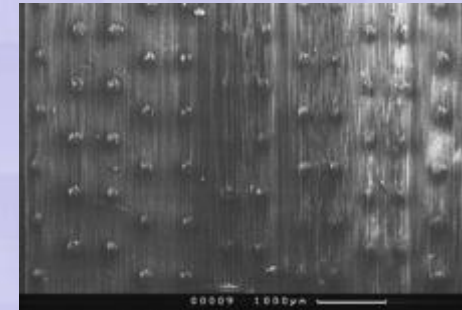
Printhead
holder

Printhead

Droplet



Accuracy & repeatability



Materials & method

Group	Composition of ink			Diameter of printhead / μm	Pattern	Parameters of pattern		Substrate	
	Solute	wt %	Solvent			dx / μm	dy / μm		
PU	Ink 1	PEG ¹	50	DMF	60	Hexagon	0.4	0.2	977-2
	Ink 2	IPDI/BiNeo	74/1	DMF					
PEG		PEG ²	5	Pure Ethanol	60	Hexagon	0.4	0.2	977-2
PMMA		PMMA	5	DMF	60	Hexagon	0.4	0.2	977-2

PU: polyurethane

PEG¹: poly(ethylene glycol) Mn = 400

PEG²: poly(ethylene glycol) Mn = 20,000

IPDI: Isophorone diisocyanate

DMF: N,N-Dimethylformamide

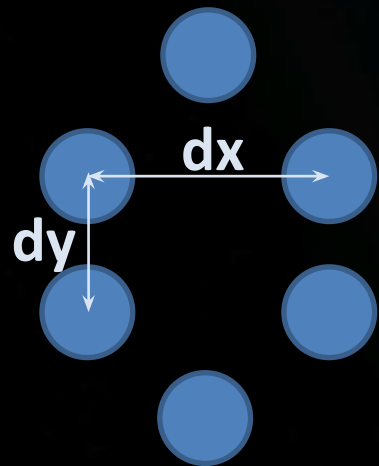
BiNeo: Bismuth neodecanoate

PMMA: poly(methyl methacrylate)

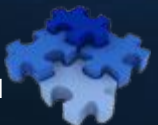
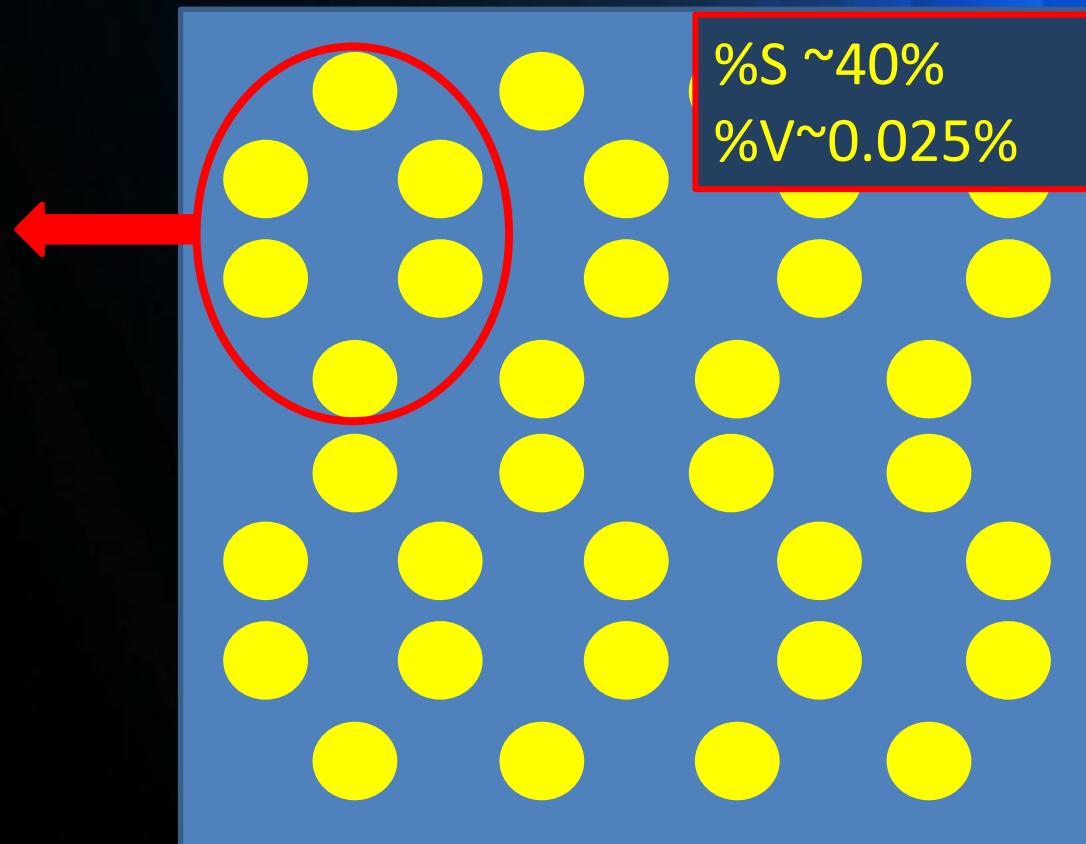
Substrate: Carbon fibre pre-impregnated with resin (prepreg) was obtained from Cytec (CYCOM 977-2-35-12KHTS-268-300, Cytec Industries Inc., New Jersey, USA)



Pattern – Hexagon

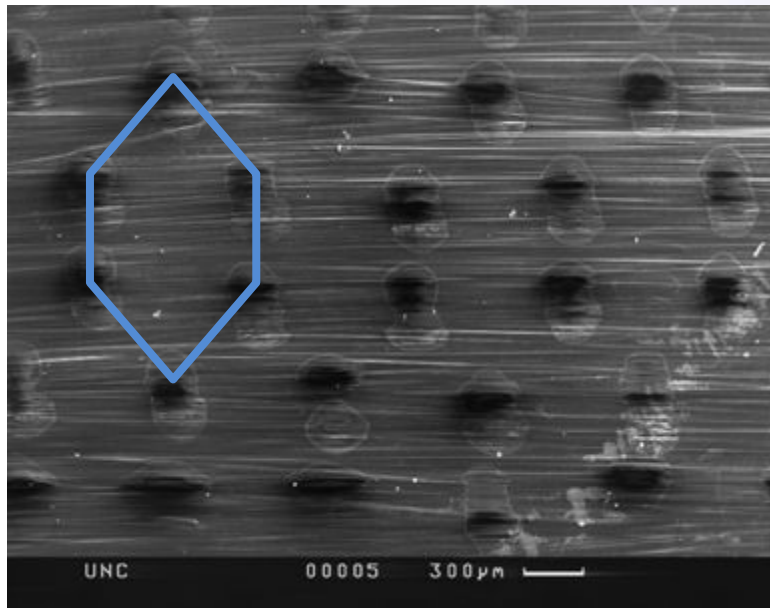


hexagon

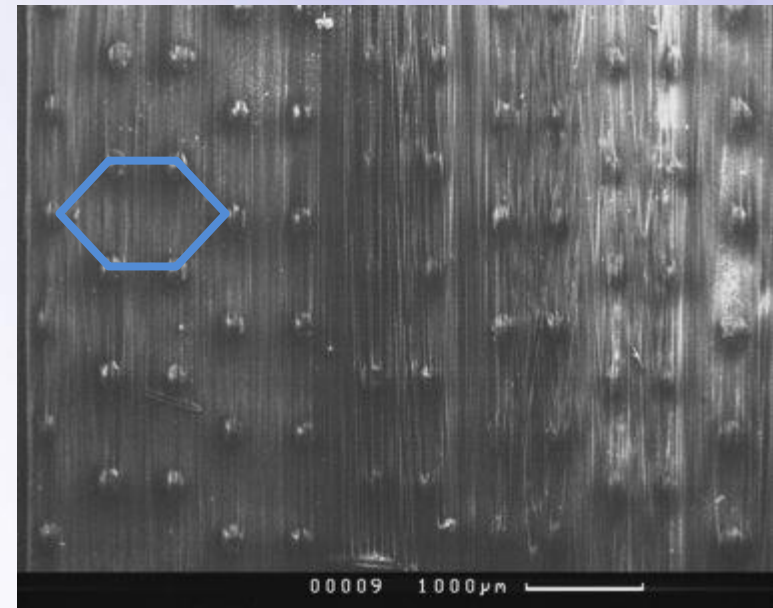


Morphological analysis

PU dots on 977-2 pre-preg



a. Before curing



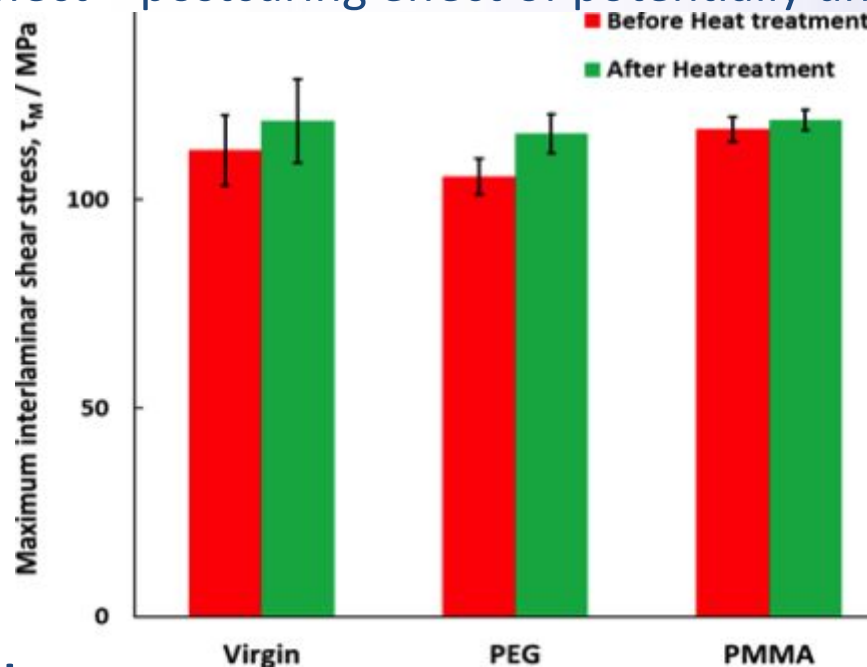
b. After curing

PU droplets are double-printed and polymerised in situ on pre-preg, and keep the printed hexagon pattern after curing cycle. (PU not subject to IP due to limited results – here used only for demonstration of printing accuracy. Synthesised in-situ from two polymer parts.)



Short beam shear test

- Maximum interlaminar shear stress (τ_M), each group contained 5 samples
- No damage introduced, investigation of undamaged parameters and placebo effect – postcuring effect of potentially un-crosslinked groups



Healing cycle: 177°C for 2 hours, harshest conditions

Purpose: to investigate any potential reduction of the shear strength, due to the presence of printed surface. Surprisingly, the structural integrity was improved with PMMA.

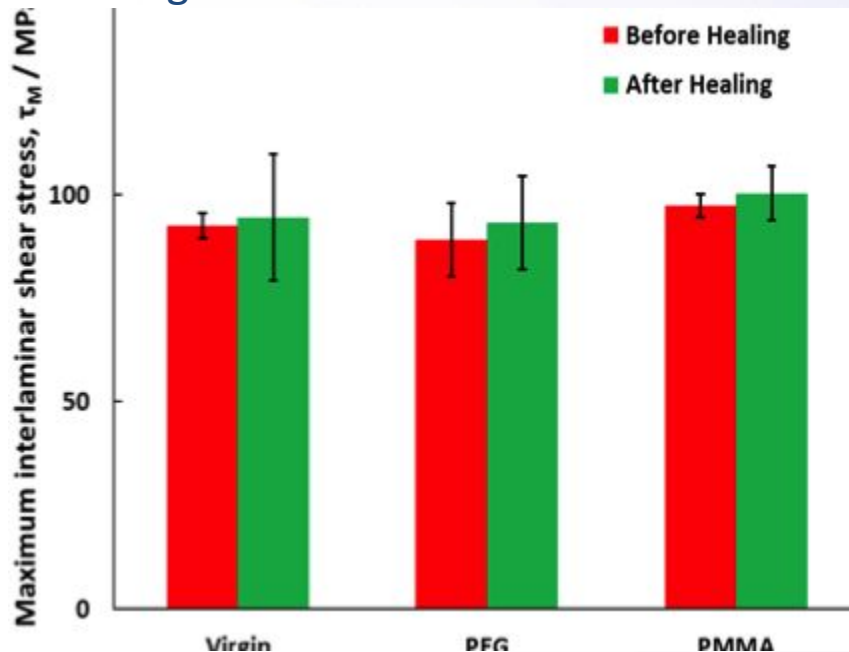
τ_M values of all groups are enhanced after healing cycle.

Note: error bar represents standard deviation, n = 5
www.sheffieldcomposites.co.uk



Interlaminar shear strength

- Maximum interlaminar shear stress (τ_M) investigation
- Damage has been introduced this time in printed and virgin samples, before self-healing



Healing cycle: 177°C for 2 hours, harshest conditions

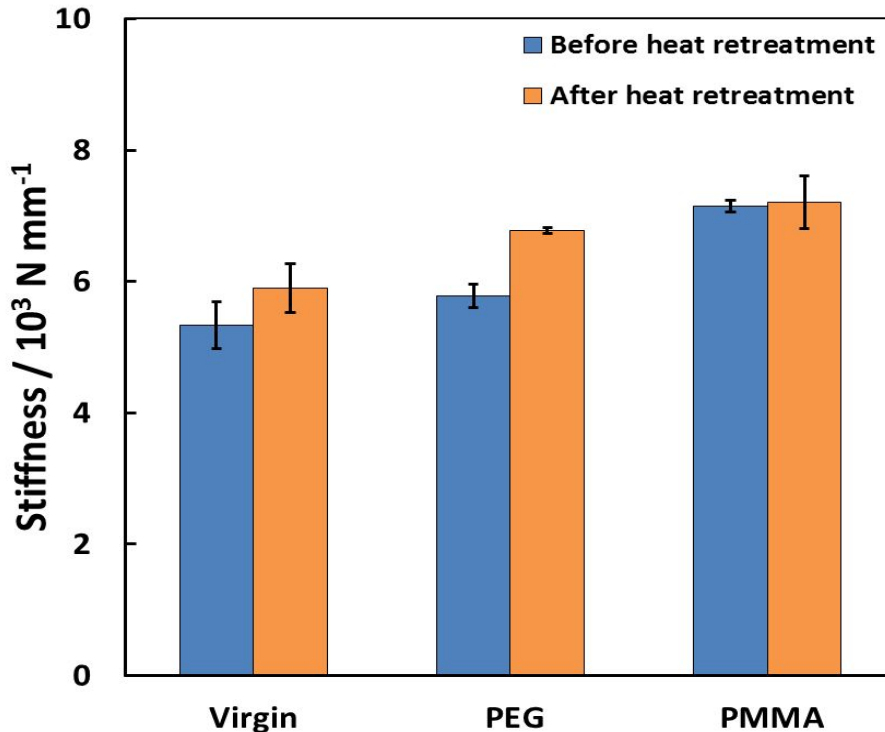
Purpose: to investigate the total reduction in shear strength due to the introduced damage and to look for the effect of self-healing. PMMA again showed improvement in properties, where reduction was initially expected due to the severe damage.

τ_M values are reduced after damage. Enhancement in τ_M can be seen after healing cycle, and the printed M15P specimens showed the highest τ_M results.

Note: error bar represents standard deviation, n = 5
www.sheffieldcomposites.co.uk



SBS test continued



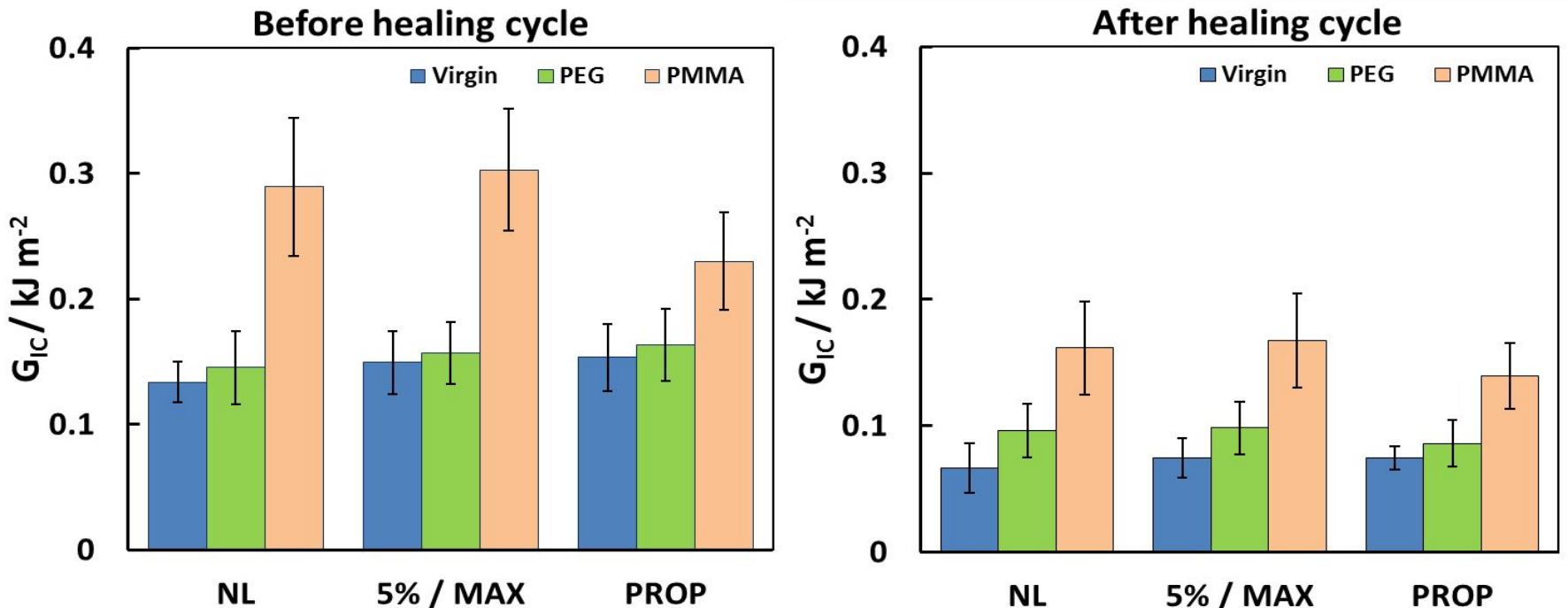
Healing cycle: 177°C for 2 hours, harshest conditions

Purpose: to investigate effect of self-healing on the material's stiffness. The effect achieved successfully. The printed surface noticeably increased the stiffness of the material both before and after the heat treatment.

With printed self-ameliorating agents, unidirectional fibre-reinforced plastic composite has higher stiffness than that of the virgin system.



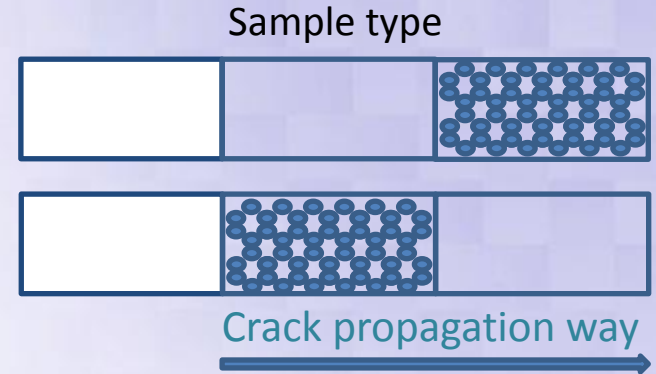
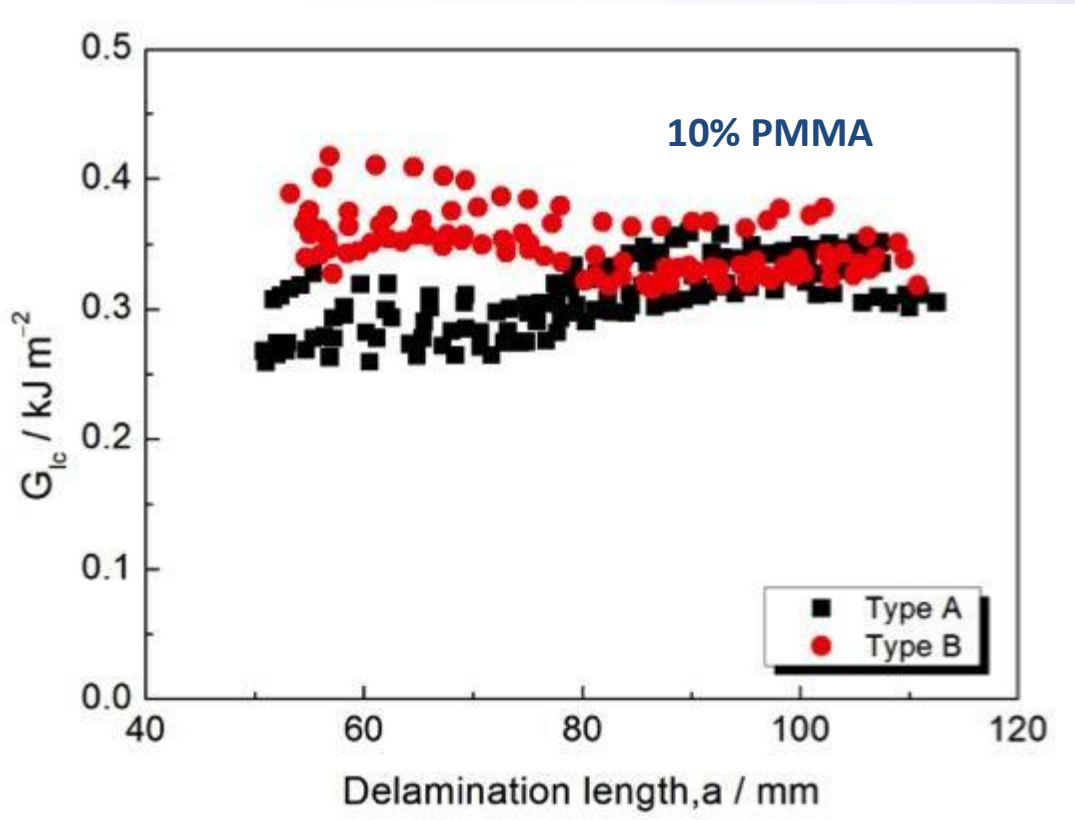
Mode I interlaminar fracture toughness (G_{IC}) test



The fracture toughness, obtained by the most destructive interlaminar test, showed approximately the double increase in value both before and after self-healing for printed PMMA material. To arrest crack propagation at this level implies even stronger capability to arrest the crack in normal service levels.



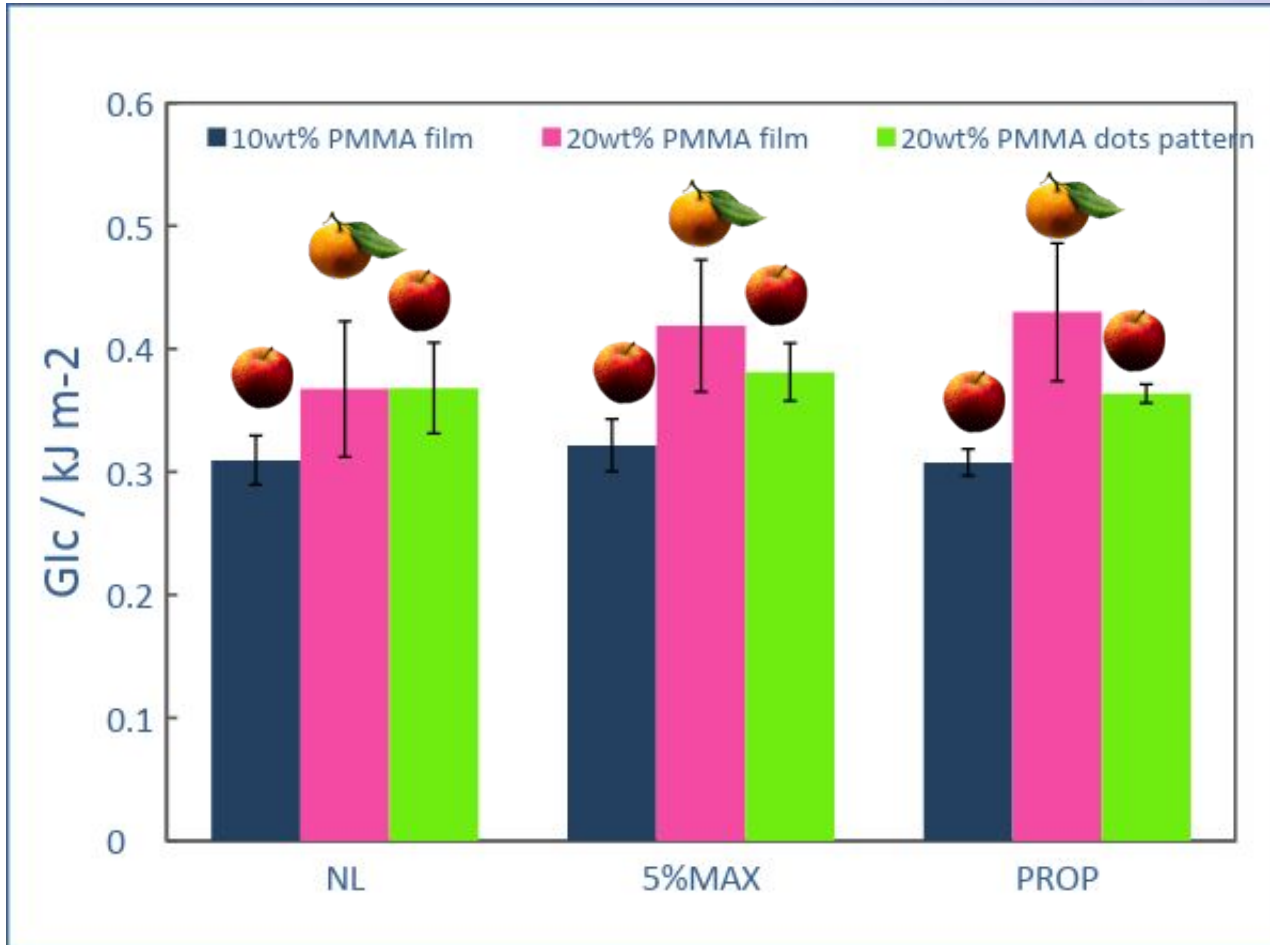
Functional gradation of properties



G_{Ic} (fracture toughness) values of polymer printed areas are comparatively higher than unprinted areas, which means inkjet printing can be applied to delicate material design work, and manufacture property graded multifunctional materials.



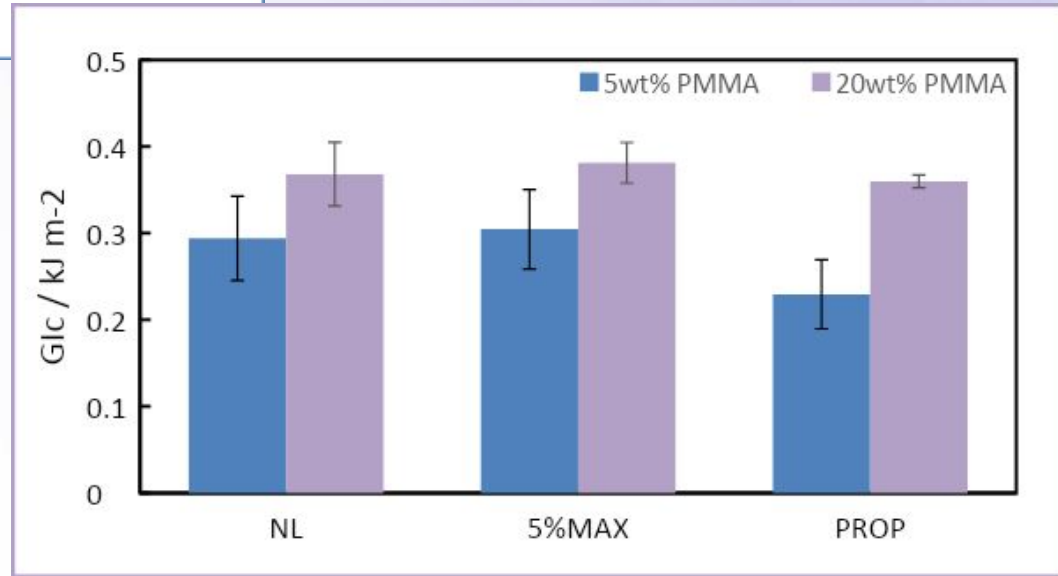
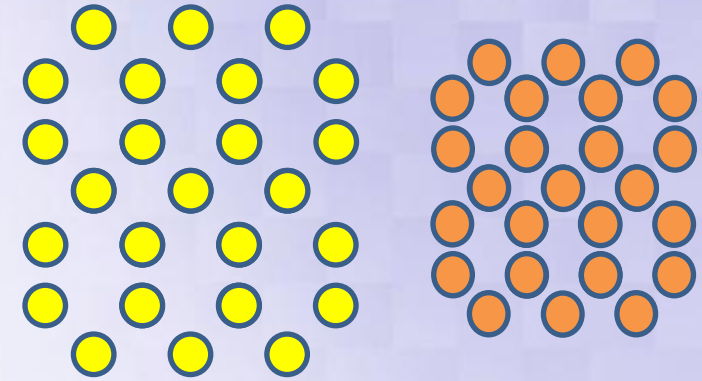
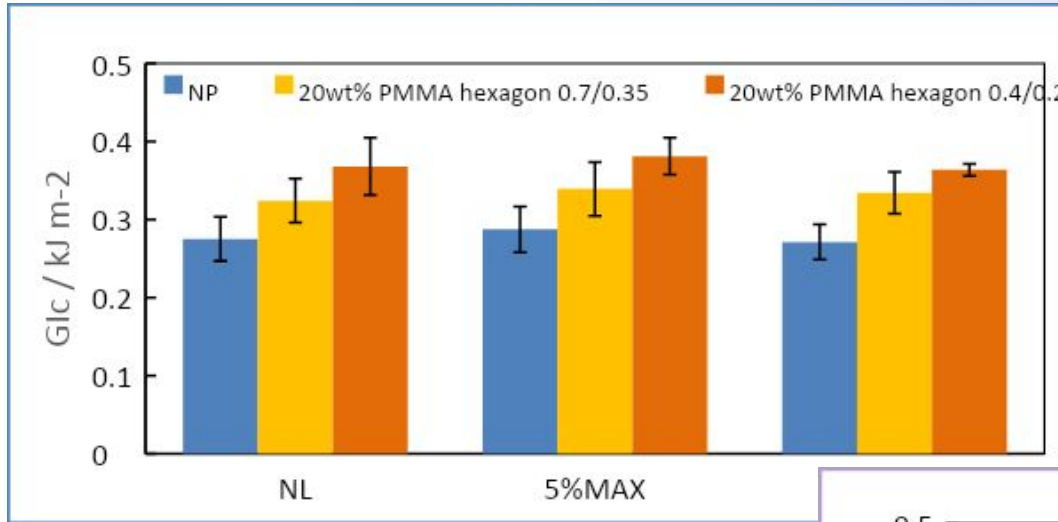
Discrete and film patterns



G_{Ic} (fracture toughness) values of discretely printed areas have comparatively higher fracture toughness values and higher predictability than fully printed surfaces with the same amount of PMMA (20% dots = 10% film by V_f). Adding more polymer to film (20% film equivalent to 40% dots) resulted in the loss of engineering predictability.

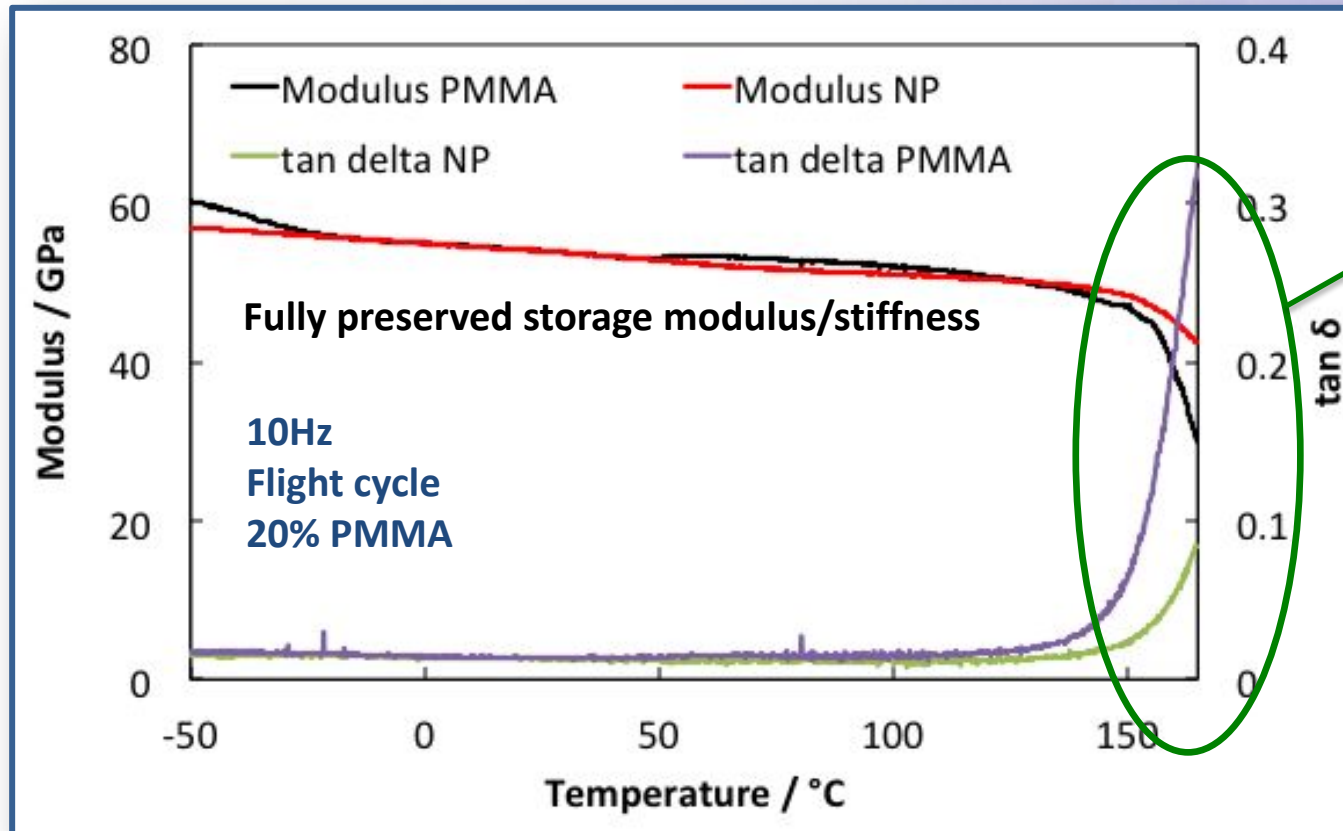


Patterns and polymer loadings



%PMMA ↑
G_{lc} ↑
Repeatability ↑

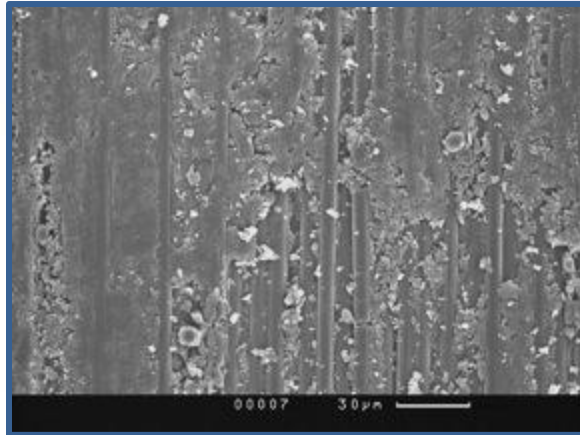
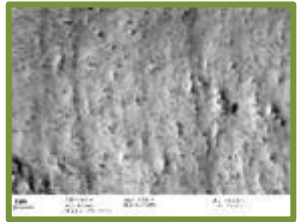
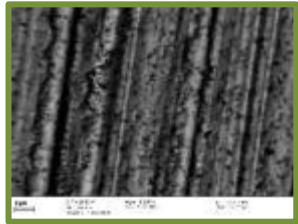
Dynamic mechanical properties preservation



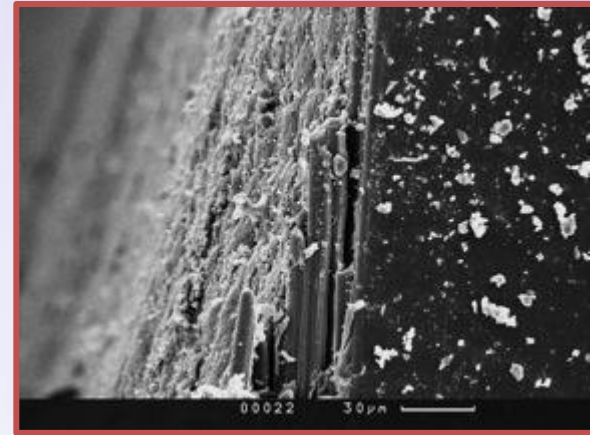
This zone is important in the machining process



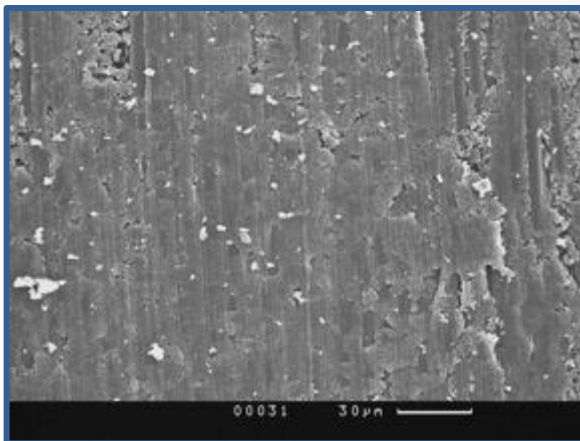
Machining quality improvement



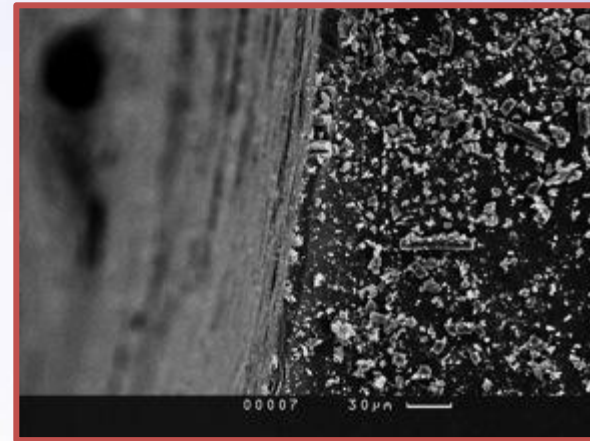
Inside CFRP hole



Edge of CFRP hole



Inside printed CFRP hole



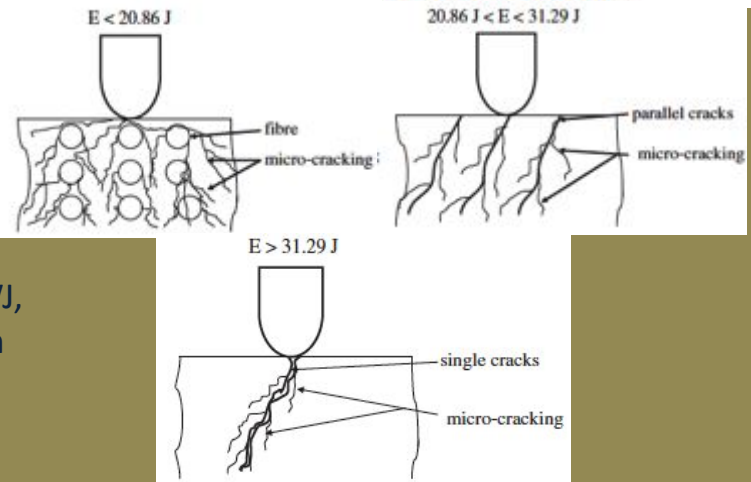
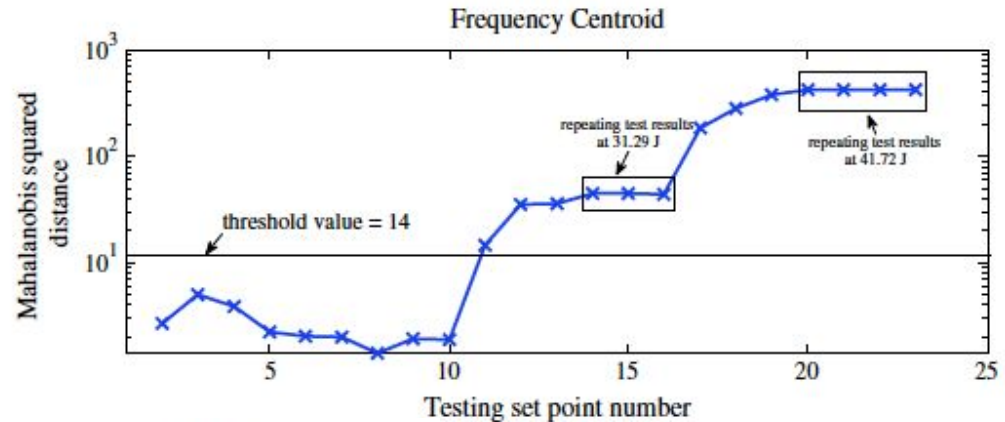
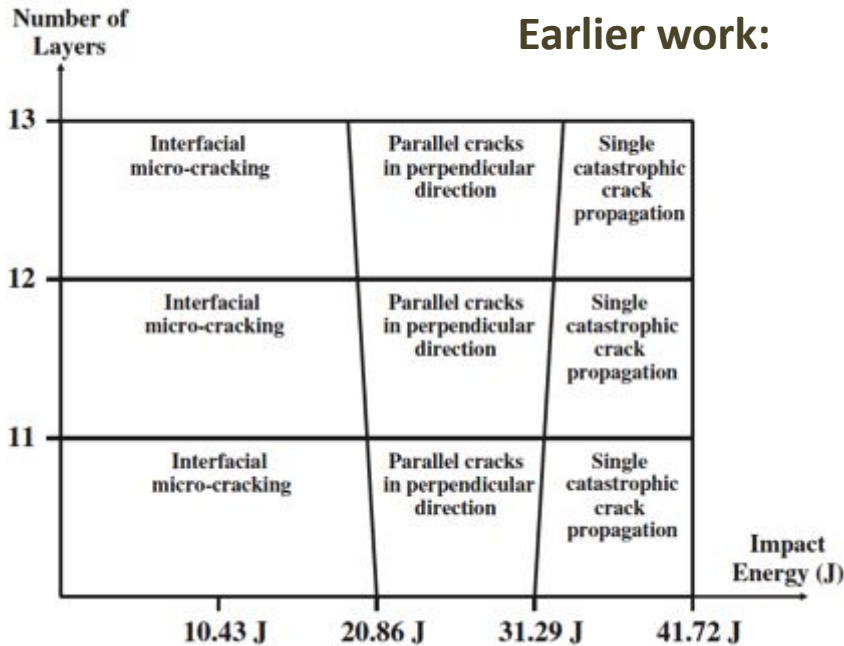
Edge of printed CFRP hole



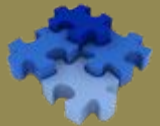
Typical tool wear in CFRPs

A plan to develop BVID detectable by SHM...

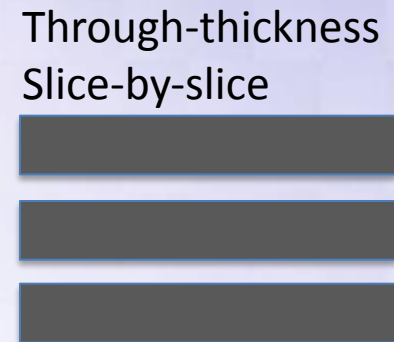
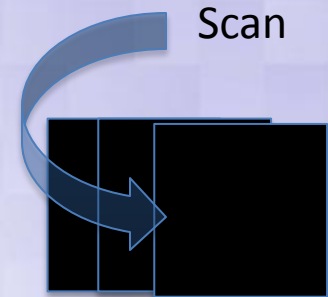
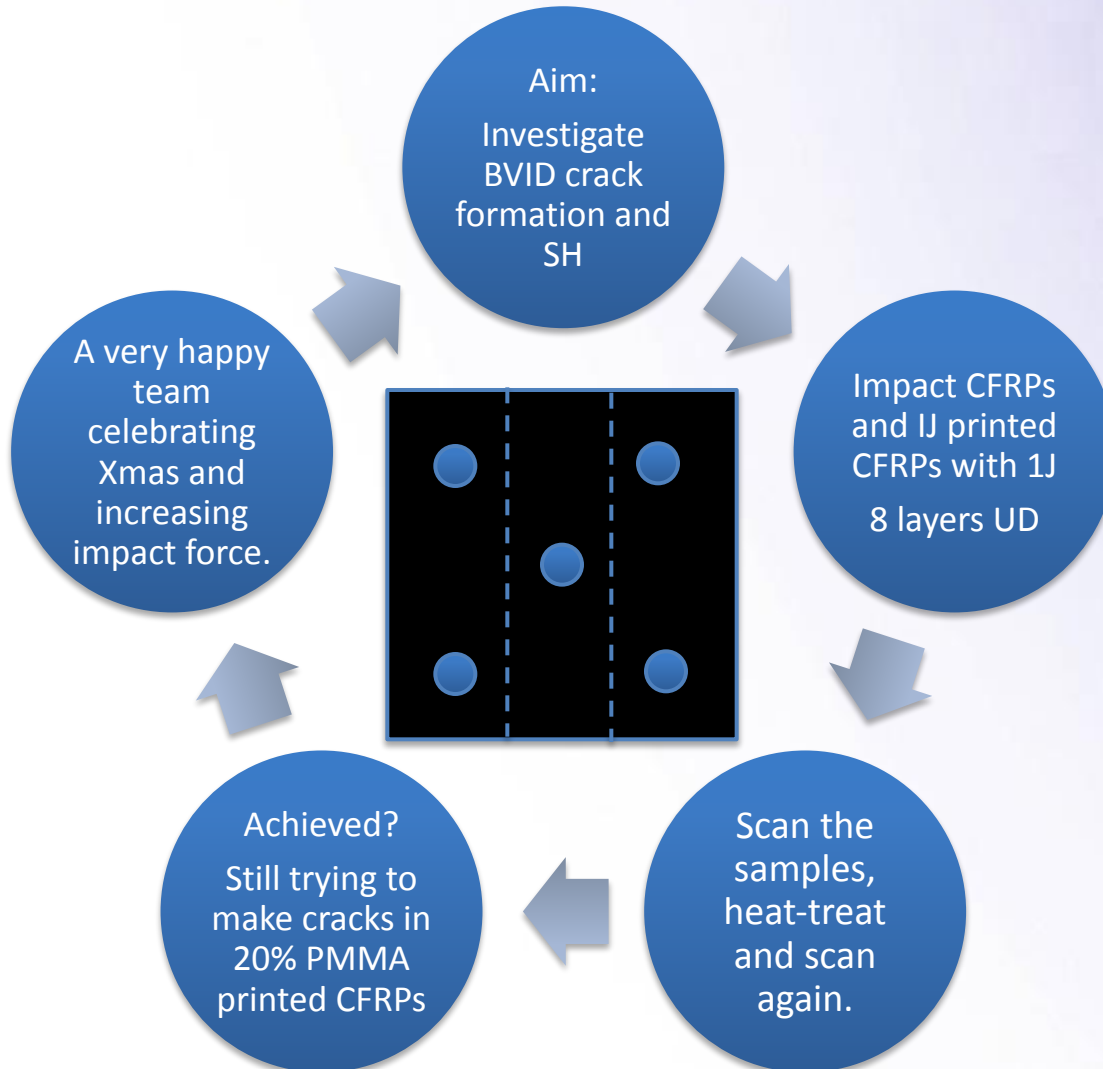
...ended up with 1J impact only in our UD specimens



Sultan MTH, Worden K, Pierce SG, Hickey D, Staszewski WJ, Dulieu-Barton JM, Hodzic A, On impact damage detection and quantification for CFRP laminates using structural response data only, Mechanical Systems and Signal Processing 25(8): 3135-3152, 2011.



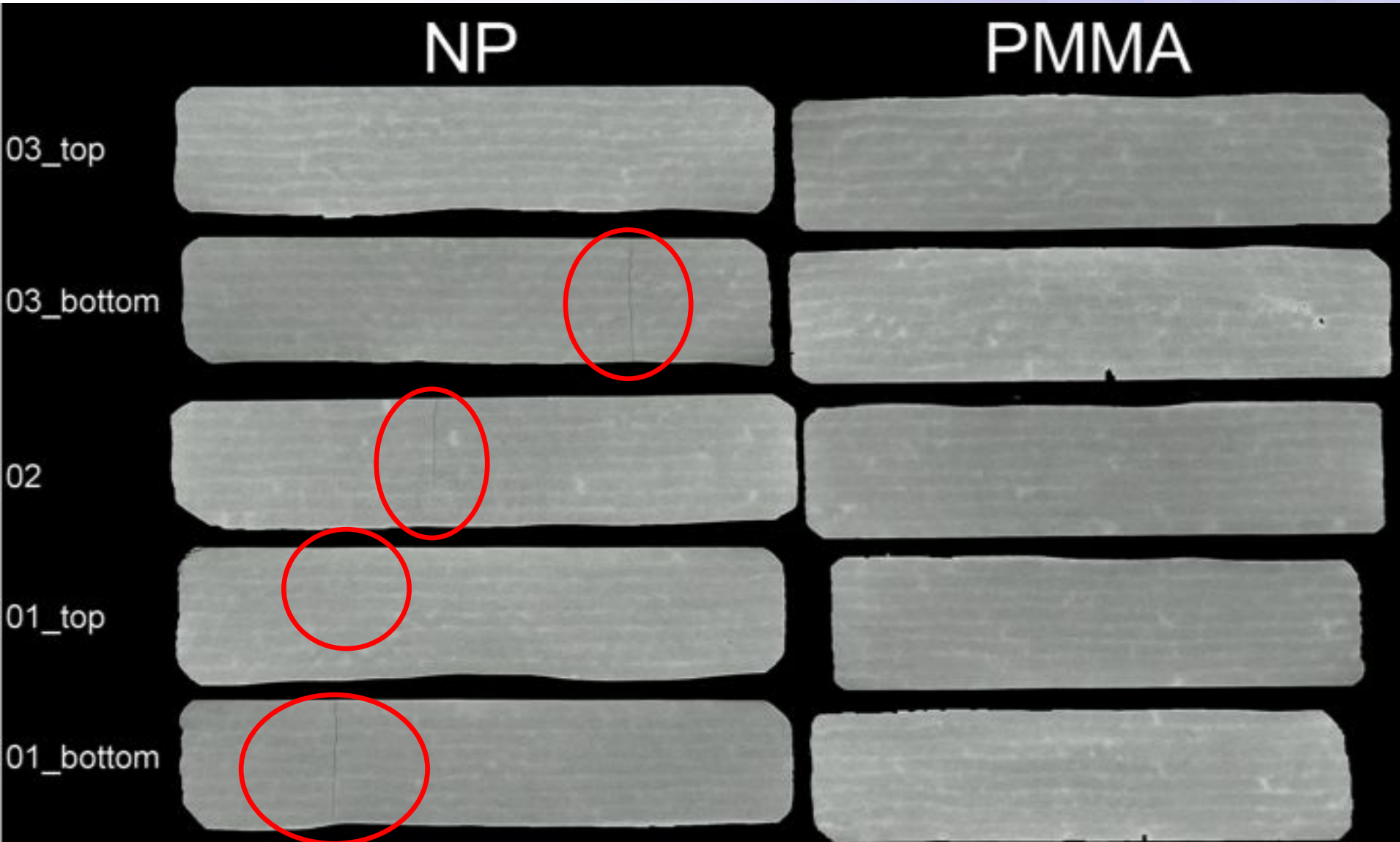
X-ray tomography @ Southampton



X-ray tomography @ Southampton

- System: Custom design Nikon/Metris dual source high energy micro-focus walk-in room system
- This scan used the 225kV source with and 1621 PerkinElmer cesium-iodide detector
- To enhance contrast a Mo target was used and peak voltage was set at 55kV, with no pre-filtration
- The current was set at 157uA (8.6W) and the panel brought forwards so that the source-imaging distance was ~ 700 mm. At this power, the focal spot is spread slightly to prevent melting of the target - however, since the voxel size at this magnification was 7.6microns, we could afford to gain flux at the expense of focal spot size, without affecting the resolution of the reconstruction.
- 3142 projections were taken over the 360 degree rotation, with 4 frames per projection being averaged in order to improve signal to noise
- Exposure time of each projection was 354ms and the gain set to 30dB
- To reduce the effect of ring artefacts, shuttling was used with a maximum displacement of 5 pixels

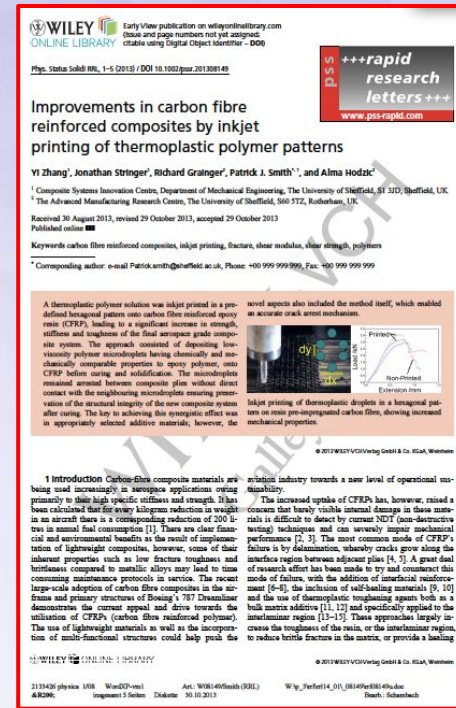




In nuce *(In pursuing the original task: to quantify the SH effect)*

- Can we accurately print thermoplastics in AE accredited CFRPs?
- Are there compatible SH polymers in the incompatible families?
- Are structural static and dynamic properties preserved?
- Is damage tolerance improved?
- Are discrete patterns more desirable?
- Are shear properties improved?
- Is there improvement after 2nd thermal treatment?
- Is machining qualitatively improved?
- **Did we manage to avoid adding any parasitic weight?**
- Did we conform to the existing supply chain?
- Did we increase the value of the product?
- Did we pioneer a new improved system?

With massive thanks to

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Phys. Status Solidi B, 1-5 (2013) / DOI 10.1002/pssb.201308149

Improvements in carbon fibre reinforced composites by inkjet printing of thermoplastic polymer patterns

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Keywords: carbon fibre reinforced composites, inkjet printing, fracture, shear modulus, shear strength, polymers

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A thermoplastic polymer solution was inkjet printed as a predefined hexagonal pattern onto carbon fibre reinforced epoxy resin (CFRP), leading to a significant increase in strength, stiffness and toughness of the final aerospace grade composite system. The approach consisted of depositing low-viscosity polymer microdroplets having chemically and mechanically compatible properties to epoxy polymer, onto CFRP before curing and solidification. The microdroplets remained around composite plies without direct contact with the neighbouring microdroplets ensuring preservation of the structural integrity of the new composite system after curing. The key to achieving this synergistic effect was an appropriately selected additive materials; however, the novel aspects also included the method itself, which enabled an accurate (small) printed mechanism.

The increased uptake of CFRPs has, however, raised a concern that barely visible internal damage in these materials is difficult to detect by current NDT (non-destructive testing) techniques and can severely impact mechanical performance [2, 3]. The most common mode of CFRP failure is by delamination, whereby cracks grow along the interface region between adjacent plies [4, 5]. A great deal of research effort has been made to try and construct this mode of failure, with the addition of interfacial reinforcement [6-8], the inclusion of self-healing materials [9, 10] and the use of thermoplastic toughening agents both as a bulk matrix additive [11, 12] and specifically applied to the interlaminar region [13-15]. These approaches largely increase the toughness of the resin, or the interlaminar region, to reduce brittle fracture in the matrix, or provide a healing

1 Introduction Carbon-fibre composite materials are being used increasingly in aerospace applications owing primarily to their high specific stiffness and strength. It has been calculated that for every kilogram reduction in weight in an aircraft there is a corresponding reduction of 200 L of in annual fuel consumption [1]. There are clear financial and environmental benefits as the result of implementation of lightweight composites, however, some of their inherent properties such as low fracture toughness and brittleness compared to metallic alloys may lead to time consuming maintenance protocols in service. The recent large-scale adoption of carbon fibre composites in the airframe and primary structures of Boeing's 787 Dreamliner demonstrates the current appeal and drive towards the utilisation of CFRPs (carbon fibre reinforced polymer). The use of lightweight materials as well as the incorporation of multi-functional structures could help push the aviation industry towards a new level of operational sustainability.

The increased uptake of CFRPs has, however, raised a concern that barely visible internal damage in these materials is difficult to detect by current NDT (non-destructive testing) techniques and can severely impact mechanical performance [2, 3]. The most common mode of CFRP failure is by delamination, whereby cracks grow along the interface region between adjacent plies [4, 5]. A great deal of research effort has been made to try and construct this mode of failure, with the addition of interfacial reinforcement [6-8], the inclusion of self-healing materials [9, 10] and the use of thermoplastic toughening agents both as a bulk matrix additive [11, 12] and specifically applied to the interlaminar region [13-15]. These approaches largely increase the toughness of the resin, or the interlaminar region, to reduce brittle fracture in the matrix, or provide a healing

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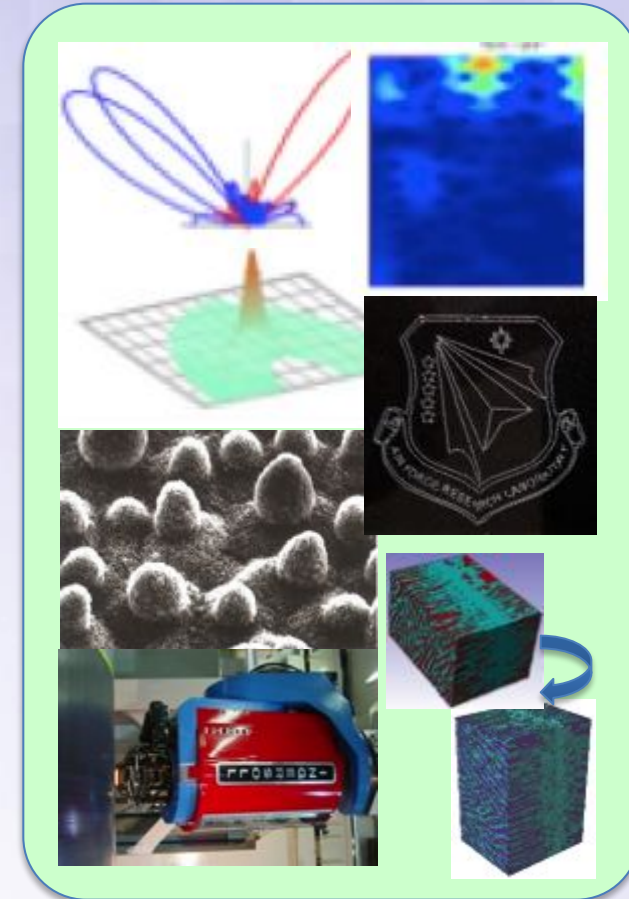
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International roadmaps for IJPCs

Sheffield, Bristol, South Carolina (McNair) and Clemson:

- **R1: manufacturing of novel IJPCs**
 - (Smith, Hodzic, Scaife, Tarbutton, van Tooren)
- **R2: embedding novel sensors in IJPCs**
 - (Giurgiutiu, Tarbutton, Smith, Hodzic)
- **R3: grafting novel polymers for IJPCs**
 - (Luzinov, Kornev, Smith)
- **R4: watermark composites**
 - (Smith, van Tooren, Majumdar)
- **R5: multiscale ultrasonic inspection in woven IJPCs**
 - (Banerjee, Giurgiutiu, Smith, Hodzic, van Tooren)
- **R6: developing FEA from x-ray tomography of IJPCs**
 - (Pinna, Deng, Majumdar, Smith, Hodzic, van Tooren)
- **R7: validation of damage models in IJPCs using SHM and 3D NDT**
 - CSIC (Hodzic, Smith, Pinna), DRG (Worden, Manson) from Sheffield and NDT (R. Smith) from Bristol – white paper submitted to AFOSR
- **R8: machining of IJPCs, influence on durability**
 - (Hodzic, Scaife, Pinna, Smith)
- **R9: integration of R1-8**



Innovation and Research

Manufacture/Characterization/Certification

Research

➤ Advanced Materials and Structures

- Design
- Development
- Testing
- Commercialization
- Response to
 - extreme conditions
 - lightning

➤ Life Cycle

- Monitoring
- Remediation and Maintenance

➤ Systems design

➤ Fuel Cells/Power Sources

➤ Cyber security

➤ Supply Chain

- Sustainability
- Optimization

➤ Global Regulated Business Strategy

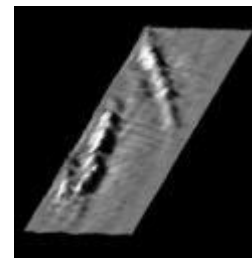
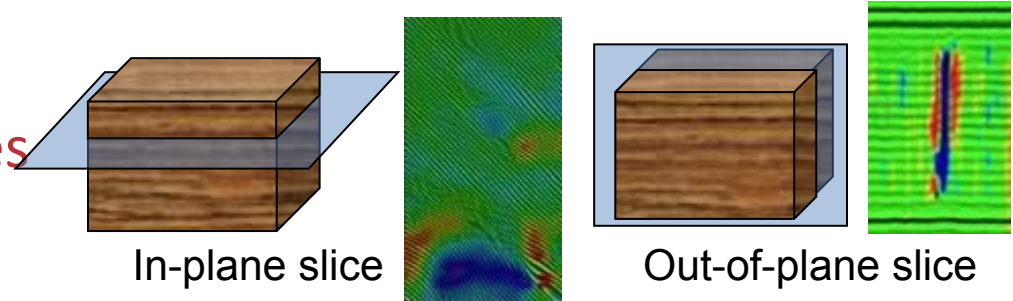
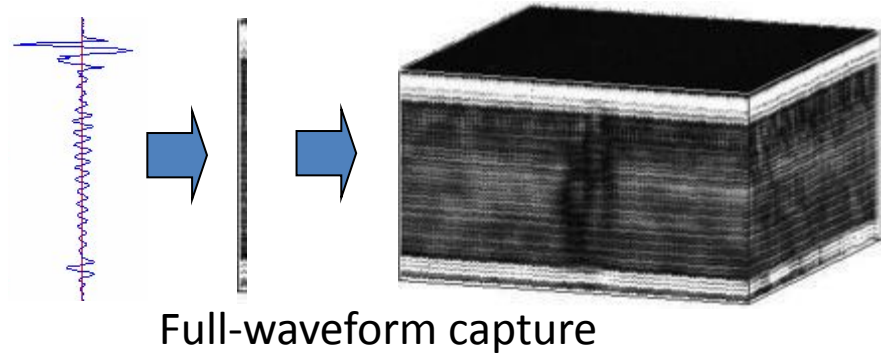
- Center for Mechanics, Materials, and Non-Destructive Evaluation
- Laboratory for Active Materials and Smart Structures
- Center for Friction Stir Processing, NSF/UCRC
- Virtual Test Bed
- Condition-Based Maintenance Research Center
- Lightning Response Laboratory
- HetroFoam Center
- Solid Oxide Fuel Cell Center
- Strategic Approaches to the Generation of Electricity
- May 2014: Advanced Composite Material Research Laboratory



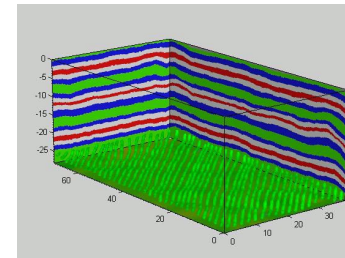
FW: NDT at high frequencies

Prof. Robert Smith

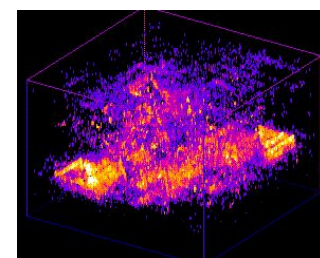
- 3D Characterisation of composite materials
 - Ultrasonic response
 - Inversion methods give actual material properties
- Fibre vector maps
- Fibre volume fraction
- Porosity
 - Frequency response
 - Distinguish between types



Wrinkle



Vector Map



Porosity