

Programme

12:30 - 13:00 FibData Background and Concept

• Pasi Kallio and Mikko Kanerva

13:00-15:00 Short presentations

- Pekka Laurkainen: Why microbond? Thoughts on scale "hierarchy" and knowledge gaps
- Sarianna Palola: One method, many possibilities; an introduction to material combinations
- Olli Orell: Extending the microbond testing possibilities aging tests
- Olli Orell: Towards automated finite element analysis
- Jarno Jokinen: finite element modelling
- Royson DSouza: Future Microbond test Towards local strain measurements
- Markus Kakkonen: Automation: How to remove the human
- **Dhanesh Kattipparambil:** Microbond testing machine vision -based movement tracking for enhanced automation





Revolution in Data-based Fibre Material Science using Microrobotics and Computational Modelling



Open Seminar 26.1.2021 Prof. Pasi Kallio





Outline

- Project Information
- FibData Approach
- About Interfacial Properties
- Microbond Test
- Seminar Overview



28.1.2021 | 4



Project Information

- Funded
 - Technology Industries of Finland Centennial Foundation and
 - Jane and Aatos Erkko Foundation
- Duration:
 - 1.1.2019-31.12.2021
- Objectives
 - To demonstrate characterization of mechanical properties of **fibre-matrix interfaces** at high throughput,
 - To automate experimental interface characterization and numerical interface modelling methods
 - To produce large data sets with selected industrially relevant material combinations



FIBdata







FibData Approach





Interfacial Properties

- Interfacial properties of the fibre-matrix system play crucial role in the mechanical properties of composite products
 - Adequate stress transfer between the fibres and the matrix is needed
 - Influenced by the sizing applied on the fibre surface
- Common measure for the stress transfer capabilities of the interface is the interfacial shear strength (IFSS)
- IFSS is measured with single fibre methods, such as
 - single fibre pull-out test, Figure (a)
 - microbond test, Figure (b)





Microbond Test?

- Microbond developed by Miller et al. in 1987.
- The method in brief
 - i. First applying a resin drop onto the surface of a single fiber
 - ii. Curing the fiber-resin system to form the droplet.
 - iii. Applying a shearing force to pull the fiber out of the droplet or vice-versa.
 - iv. Measuring the adhesion force.
 - v. Calculating the IFSS from the measured force and the embedded area of the droplet





Slide provided by Royson D'Souza, Tampere University



Motivation for Microrobotics

- The measurements typically require tedious manual work and the produced data often has really high scatter
- Our goal has been to develop automated high-throughput IFSS tester



Sample Preparation





- The method enables deposition of tens of droplets on a single fibre filament
 - Typically 40-60 droplets / fibre
 - Droplet size can be controlled
- Samples are placed on U-shape sample holder



Measurement Principle 1/2

- Measurement is performed by pulling the droplets out of the fibre
- Sample holder is connected to force sensor
- Microblades exert force on the droplet
 - Gap between the plates can be controlled with submicron resolution







Measurement Principle 2/2

- From each droplet, we estimate
 - Adhesion area (microscope image)
 - Debonding force (max)
- We determine IFSS through a line fit



Microscope image with embedded length determined







Possible Research Questions

- How to modify the interphase by modifying the sizing?
- How does the storage time influence the sizing and thus the resulting interphase?
- How does the matrix material influence the interfacial properties?
- How to maximize the viability of recycled fibres?
- What is the role of ageing in modifying the interfacial properties? (e.g. UV, hygrothermal, etc.)



Programme

12:30 - 13:00 FibData Background and Concept

- Pasi Kallio and Mikko Kanerva
- 13:00-15:00 Short presentations
- Pekka Laurikainen: Why microbond? Thoughts on scale "hierarchy" and knowledge gaps
- Sarianna Palola: One method, many possibilities; an introduction to material combinations
- Olli Orell: Extending the microbond testing possibilities aging tests
- Olli Orell: Towards automated finite element analysis
- Jarno Jokinen: finite element modelling
- Royson DSouza: Future Microbond test Towards local strain measurements
- Markus Kakkonen: Automation: How to remove the human
- **Dhanesh Kattipparambil:** Microbond testing machine vision -based movement tracking for enhanced automation



Thank You for Your Attention







Next Steps?

- Interest in providing samples for testing?
- Interest in any particular aspect presented today?
- Interest in company specific workshops?



Interested in discussing today?

• raise your "hand"

Or contact us later using email

Contact Information:

- Mikko.kanerva@tuni.fi
- Essi.sarlin@tuni.fi
- Pasi.kallio@tuni.fi
- Markus@fibrobotics.com

Tampereen yliopisto Tampere University

Human Potential Unlimited.





Associate Professor Mikko Kanerva

Plastics and Elastomer Technology

22.1.2021



Fibrous materials

- Allow to apply enormous improvements in material performance
- Lead to anisotropy (on **macro** scale)
- Lead to interfaces on micro scale
- Require multiscale framework



https://technology.nasa.gov/

Anisotropy

- Complex theme when material has strong multiscale relationships
- Allows to <u>tailor and optimize</u> material locally in precise application and operation
- Handling requires exact and reliable material data



https://www.luxturrim5g.com/



Material data

• Fibre properties are dominating – e.g. tensile behaviour, thermal expansion, anisotropy







- Matrix properties are the second important along with adhesion to fibres
- Interface affects many of the composite properties in a complex fashion



Interface

- Due to small size, small test devices
- 'Strength' or adhesion along the interface is not a standard property
- Ideally a 2-D concept no-volume material
- For almost all modelling and design, implicitly affecting performance
- Properties like interfacial shear strength (IFSS) and interface fracture toughness have been introduced currently





Interfaces tested

- Test devices need to record accurately:
 + force
 + displacement or strain
- Test specimens, to form reasonable sample of a material batch, need to be *many* and *fast prepared*
- Statistical analysis of data is important



Models and design

- Typically laminate theory is in use for composites – even when embedded (for FEM)
- For single fibre-matrix analysis, multiscale approaches can be used, e.g. with representative volume elements (RVEs)
- Industry requires fast design and simulation codes



What are INTERFACE material properties ?

The SCIENCE seminar will be organized Tuesday 26.1.2021:

15:30-15:45 Why we need multi-scale modeling of composite materials?



Why microbond? – Thoughts on scale "hierarchy" and knowledge gaps

Pekka Laurikainen Doctoral researcher (M.Sc)

Contents

What does microbond measure?

What are the problems?

What is the point?

What does microbond measure?

Repeatability is key!

Tampereen yliopisto Tampere University

- Representative dataset needs adequate sampling to ensure the range of properties
- Conceptually more similar to fibre testing
- "Traditionally" very labour intensive
- Measurement output force vs. embedded area – only useful when comparing similar systems
 - Analytical and/or numerical (finite element) models needed to expand applicability.



What does microbond measure?

Error source	Possible negative outcomes
Load measurement	Over-/Underestimation of the measured load
Device optics	Inaccurate embedded length and/or fibre diameter
Microvise control	Variation of droplet loading state, high scatter in final results
Embedded area range	Basic assumptions of microbond invalid, fibre breakage
Fibre surface	Variation of the results, always present
Resin curing	Inconsistent results, increased scatter, Resin failure

- The amount of possible variables is massive
 - Due to the scale, seemingly insignificant uncertanties can lead to significant errors in measurement.
- Major issues
 - Scale appropriate equipment
 - Loads commonly in range < 0.5 N
 - Accurate motion in micrometer scale and below
 - Selection of representative sample
 - Characterising a short single filament from a fabric or roving requires 20-40 microbond measurements.
 - Resin cure is well known in the scale of millilitres and above (from DSC samples to macroscale parts)
 - Droplets measured in microbond are in the scale of picolitres (10⁻¹²).

So what's the point?

What is the point?



Tampereen yliopisto Tampere University

- Modern state-of-the-art applications can already have major structural parts from composites
- However, current understanding is already pushed to the limit, within reasonable margins of safety.
- So what is needed for the next generation?





The point is...

 In order to meet the demands of next generations of high-end applications, comprehensive understanding – as currently sort-of exists in macroscale– needs to be expanded through the different scale levels.

"Mesoscale"; Composite as a material

Macro scale; Structural

properties and product design

Polymer / reinforcement morphology and properties [Å - <µm, ps - ns]

Atoms, molecules, chemical reactions [Å - nm, fs - ps] Microscale; interfacial properties [µm - mm, s - min] ... and how to fill the gaps!



In practice

The scales are mostly explored individually. Problems:

- Inherent problems in upscaling specific phenomena
 - E.g., computational cost
 - "Ensemble averages"
- Relevance
- And, of course, the information gaps

Tying the scales together is one of the biggest future challenges for composites.

And the gaps might just be the largest around interfacial testing methods!







Thank you! Any questions?



One method, many possibilities; An introduction to material combinations FibData seminar 26.1.2021 Sarianna Palola



- What?
 - A versatile method to study fiber – matrix interphase
- Why?
 - Interfacial properties impact greatly composite performance
- How?
 - "The sky is the limit" with possible material combinations
 - Fiber: synthetic vs natural
 - Matrix: thermoset vs thermoplastic





From microscale to macroscale








Fibers

- Synthetic fibers tested so far:
 - Carbon fiber (CF)
 - Recycled carbon fiber (rCF)
 - Glass fiber (GF)
 - Recycled glass fiber (rGF)
 - Aramid fiber
 - Polyethene fiber
- Natural fibers tested so far:
 - Flax
- With or without surface treatements





Fibers; case example

Effect of surface treatment on IFSS in CF/PMMA





2 um



Thermoset matrix

- Tested so far:
 - Epoxies
 - Polyvinylesters
 - Polyester
 - Polyurethane
 - Silicone
- Pot life of 10 minutes adequate but longer is better
- Protective N2 atmosphere can be used during
 - Sample manufacturing
 - Curing





Thermoplastic matrix

- Tested so far:
 - Polypropene (homo- and copolymer grades)
 - Polyamide
 - Polycarbonate
 - Polyester
 - PMMA (poly(methyl methacrylate))
 - PEEK (polyether ether ketone)
 - PEI (polyetherimide)
- Protective N2 atmosphere can be used to prevent oxidation during sample preparation
- Low melt viscosity materials work the best



Conclusions

- Multiple material combinations are possible
 - From synthetic to natural fibers and thermoset to thermoplastic matrix
 - With or without protective atmosphere
- Fast and efficient method to evaluate coposite properties in microscale
 - Represents macroscopic behaviour
 - Reliable results regardless of material combinations

Thank you!

Any questions?

Tampere University **Extending the** microbond testing possibilities aging tests

Olli Orell, Jesse Savolainen



Aging tests for composites

- Mechanical properties of FRPs degrade due to adverse environmental conditions
- Laminate scale aging tests are carried out routineously
 - realistic in-service environments or accelerated by temperature
- Microbond testing using the current technology offers high throughput method with minimal amount of materials and simple manufacturing



Several benefits to study the aging phenomena of the 'purely' interfacial properties of composites





Microbonds and aging

- Sample preparation can be carried out normally
- Aging possibilites:
- a) Full immersion in liquid
 - The acid resistant sample holders allow use of even corrosive media
- b) In controlled atmosphere
 - The impermeable sample holder can be controlled to desired atmosphere by flushing with gases
 - Saturated salt solutions in the closed box can be used to achieve desired relative humidity
- c) Thermal/moisture cycling
- d) UV exposing







Example case

Matrix: Vinylester Fiber: ECR glass

Aging media: DI water (*and 75% RH*) Temperatures: RT and 60 C (and *40 C*) Aging time: 0h, 24h, 50h, (68h)





Test observations

- Change in the behaviour scatter increases
- Some droplets remain visully almost original, but some show radical changes
- Apparent average interfacial shear strength shows decreasing trend for aging in 60 C







Reliability analysis with large datasets

- Large datasets will enable various analysis methods –statistics or propabilities
- Eg. 2-P Weibull analysis can reveal differences in the fracture behaviour

Tampere University





Lessons learned

- Suitable for comparison studies between different materials or aging parameters
- Quick tests compared to normal aging tests with coupons
- High scatter result analysis needs rethinking
- Consistent specimen fabrication is essential laborious screening for specimen fabrication often needed
- How to 'age the interface', not the fiber (or matrix)



Thank you for attention!



Towards automated finite element analysis

Olli Orell, Jarno Jokinen



Contents

- Motivation Current state of microbond analysis
- Input for analysis
- Approach for an automatic finite element analysis
- FE model features and results





Motivation – Current state of microbond analysis

- The comparison of microbond tests are typically based on 'Apparent Interfacial shear stress (IFSS)
 - Using the force maxima, fiber thickness and droplet length
 - Provides average stress value
- IFSS is easy and quick method for post-processing, but do not take account:
 - geometric shapes,
 - residual stresses
 - nonlinear materials,
 - contacts
- Need for more accurate analysis method, applicable for automated post-processing









Input for model – Available raw data

- More experimental data available for modelling (than maximum force and diameter):
 - Force-time curves of the tests
 - Images of the droplets
 - Fabrication parameters (curing) of the matrix
 - Droplet position in the test fixture
 - Displacement







Input for model -Database based data storage

• Amount of test data is extensive

Tampere University

- 15-30 droplets for each fiber specimen (CSV result files and images)
- Several paraller specimens
- All the results are collected into database (MongoDB) instead of separate files allowing simple querying of the data
 - Force data, droplet images, material properties, sample manufacturing parameters, material bathes, etc.

Approach for an automatic FEM analysis





From images to mesh - examples





Ready FE problem in text format to be send to FE solver with no efforts required by the user!







15652	10161,
15653	10162,
15654	10163,
15655	10164
15656	*Node
15657	1,43.0,220.0,0
15658	*Nset, nset=rp
15659	1.
15660	** Constraint: Constraint-1
15661	*Tie, name=Constraint-1, adjust=ves
15662	dropletInterfaceSurface, fiberInterfaceSurface
15663	** Constraint: Constraint-2
15664	*Rigid Body, ref node=rp.pin nset=bladeNodes
15665	*End Assembly
15666	**
15667	** MATERIALS
15668	**
15669	*Material, name=Fiber
15670	*Elastic
15671	70000, 0.2
15672	*Material, name=Droplet
15673	*Elastic
15674	2500, 0.35
15675	*Material, name=Blade
15676	*Elastic
15677	210000, 0.33
15678	**
15679	** INTERACTION PROPERTIES
15680	**
15681	*Surface Interaction, name=Kitkaton
15682	1
15683	*Friction
15684	0.,
15685	*Surface Behavior, pressure-overclosure=HARD
15686	**
15687	**BOUNDARY CONDITIONS
15688	**
15689	** Name: RightFiberEnd Type: Displacement/Rotation
15690	*Boundary
15691	rightFiberEnd, 1, 1
15692	rightFiberEnd, 2, 2
15693	** Name: refPoint Type: Displacement/Rotation
15694	*Boundary
15605	rp.2.2
13033	
15696	rp, 6, 6



FE features

Advantages

- Based on real geometry
- Complicated material models both droplet, fiber and interface can be included
- Contact included between blade and droplet
- Realistic load value

FEA enables taking account the various factors in order to analyse of the interface



Current model advantages

- Efficient analysis
- No need for manual modifications of the input Limitation
- Axisymmetric model (visualization shown in figure)



Results

- Stress analysis
 - Interface (average vs distribution)
 - Plasticity
- Deformation
 - Strain
 - Displacement

- FEA will be likely carried as batch runs after tests
 - Fluent data handing will be necessary
 - Future development: automatization of postprocessing







Thank you for your attention!

Future Microbond tests – Towards local strain measurements

Royson Donate D'Souza Doctoral Researcher Tampere University, Finland.



Motivation towards strain measurements

Numerical model with complex damage at interface with just one output parameter ????

400

The current measures of fibre matrix Fmax • Force interfacial adhesion rely only on single parameter i.e., Force. 0.8 Force (N) 0.2 Knife-Edge 100 150 200 250 300 350 Blade displacement (μm) Free Fiber lens Embedded Lengt

Current interface test methods for microbond can result in force and blade displacement data.

Cross-head displacement is basically never used for standard fracture testing of interfaces.

Fracture process in a laminate or ply cannot be described well without parameters giving allowables to interfacial dissipation.

9

D

Investigation on strain measurements





a. Force Cell		b. Sample Holder	c. Adhesive	d. Optical fibre with FBG e		e. Blades	
Constituent	Optical FBG fibre		Droplets		Sample ho	lder Knive	es
Material	Glass fibre (GF1, Nufern)		Araldite® 5052-resin Aradur® 5052-hardener (Huntsman)		Acrylic	Stainless	s steel

Finite Element Model

FibreNet



Features revealed by force-strain data

Local strain measurement using optical fibres not only enhance the microbond test but also provides additional parameter for solving interfacial fracture phenomenon.

Peak strain, peak force and first derivative of force strain profile are crucial to understand fracture process.

Derivatives of force strain curves provide accurate estimation of Gc and τ .

Fracture process at the interface

Four stages of fracture:

Stage 1: Fibre elongation and deformation of the droplet.

Stage 2: Damage progresses circumferentially.

Stage 3: The interfacial damage progresses and extends spatially in the fibre's longitudinal direction.

Stage 4: There is an abrupt rise in the consumption of interfacial damage energy

Acknowledgements

- Dr. Mikko Kanerva
- Dr. Jarno Jokinen
- Dr. Pasi Kallio
- Dr. Essi Sarlin
- Dr. Paulo Antunes
- Markus Kakkonen
- Pekka Laurikainen
- Farzin Javanshour
- Olli Tanhuanpää
- Dr. David Seveno

This project has received funding from the European Union's Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant agreement No 764713.

Automation: How to Remove the human

Markus Kakkonen Markus@fibrobotics.com

Start-up from Tampere University of Technology

- Project FIBRobotics (2015-2017) developed micromechanical characterization of fibrous and fiber reinforced materials
 - Funded by Finnish Agency for Innovations

BROBOTICS

- Technological goal: develop a high-throughput interfacial shear strength (IFSS) tester
- Start-up company established Q2/2019
- Market launch 2021

FIBRObond and FIBROdrop

- Currently we can perform 40 measurement events / hour
 - A measurement event measures the force that is required to debond a polymer droplet from a fiber

 Efficient sample manufacturing for thermoset and thermoplastic samples


FIBROBOTICS

FIBRObond

- Time spend on measuring is dependent of material properties
- Even if total time of measurement is lower.
 - General user can not do more than 150 measurement events per day. Equal to 5 samples
 - Limiting factor is human
 - Next step is to automatize the measurement



FIBROBOTICS

Automation

- All actuators in Fibrobond has encoder
 Positions are known
- Requires user to input a filter for measurable droplets
- Based on information for computer vision



Automated measurement of 23 droplets (video is sped up 4x)







Results from automated measurement



Conclusion



- FIBRObond automation is proof of concept what the device is capable
- To unleash the full potential of the device automation is the key
 - Sample manufacturing is fast enough to support this
 - Releases user to do other tasks



Any Questions?

More information at www.fibrobotics.com



Microbond testing – Machine vision-based movement tracking for enhanced automation



Dhanesh Rajan PD Research Fellow

26.1.2021



Microbond testing device

- Single droplets are pulled apart from the filament using microtome blades and the force required for this is recorded
 - Enhanced automation of microtome blade 'vertical' movements

✓ Detect the blade-to-fibre contact reliably.





- Single droplets are pulled apart from the filament using microtome blades and the force required for this is recorded
 - Enhanced automation of microtome blade 'vertical' movements

✓ Detect the blade-to-fibre contact reliably.

Tampere University



Microbond testing device- enhanced automation

• Optical methods?



Requires

- additional instrumentation &
- implementation can be complex!



- Image based methods? especially when we have integrated cameras in the system?
 - ✓ Machine vision <u>optical-flow</u> methods



In macroworld (E.g. Traffic surveillance)

Tampere University



In microworld (E.g. Our expertise)





Automation: How to Remove the human

Markus Kakkonen Markus@fibrobotics.com Automation

- All actuators in Fibrobond has encoder
 - Positions are known
- Requires user to input a filter for measurable droplets
- Based on information for computer vision



Why do we study about image-based methods?

- Automatic droplet removal based on actuator encoder information- K. Markus
- \circ To improve the robustness and reliability
 - Image-based measurements as an additional means for investigating the measurement
 - Fibre types:
 - glass fibres, carbon fibres,...
 - Vibrations, Intensity non-uniformities..
 - An active and suitable measurement method

Optical-flow; How does it work?

• Track the velocity vectors (motion) from frame to frame.



Optical-flow; How does it work?

o Track the velocity vectors (motion) from frame to frame.



Tampere University









Testing a few optical-flow methods



Method development; current status!

Condition for movement detection

• Thresh=1.29*(mean+std)

Tampere University

	Success % [X/45]		
Method	1s	2s	3s
im1=im2	63.6363636	72.72727	81.81818
im1=avg (4s)	66.6666667	72.72727	75.75758
im1=avg_BackGroundRemoved	80	84.4444	88.8889
im1=avg_BackGroundRemoved old	75.7575758	81.81818	90.90909
im1=avg_1234	69.6969697	72.72727	75.75758
im1=avg_1234_BackGroundRemoved	69.6969697	72.72727	75.75758





What is next?

- Real time image processing
 - Decision making optimization and trigger signal for microbond testing





Thank you! Questions?