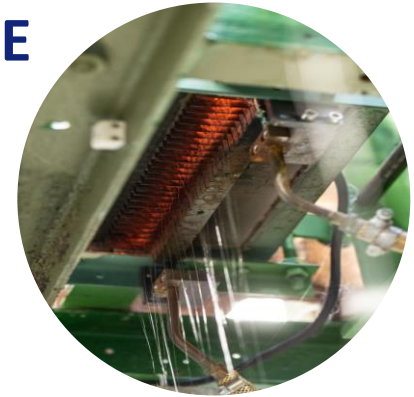
TECHNISCHE UNIVERSITÄT BERGAKADEMIE FREIBERG
Die Ressourcenuniversität. Seit 1765.

Leibniz-Institut für Polymerforschung Dresden

# BIOBASED GLASS FIBER SIZINGS FOR DEGRADABLE COMPOSITES IN MEDICAL AND TECHNICAL APPLICATIONS

**ICCM23, Belfast, 30.07.-04.08.2023**

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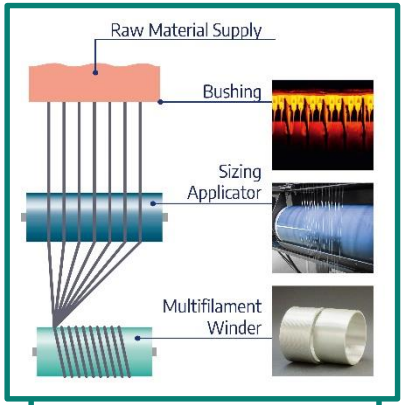
# Biomaterials and Biocomposites

- Biomaterials:
  Ceramics, Polymers and **Glasses**
- Foundation for novel biocomposites:
  bioactive glasses

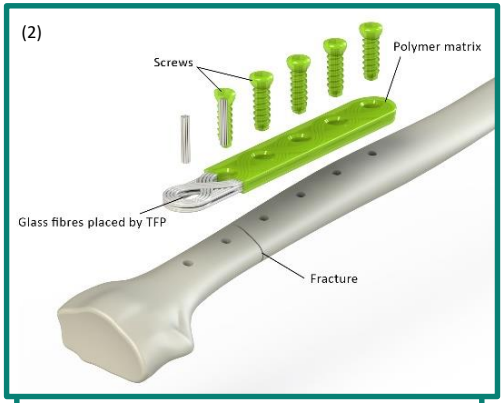
  → osteoinductive, osteoconductive


→ **Our aim: novel biodegradable load-bearing composite materials**


Bioactive glass as granules




Melt spinning process

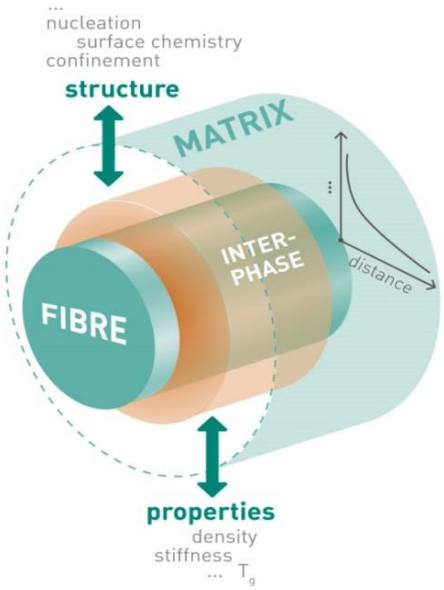



Bioactive glass as endless fiber



(1) www.medeco-ch.com, BonAlive®
(2) Plyusnin, A. et al. ESB2019, 9-13 Sept 2019, Dresden, Germany

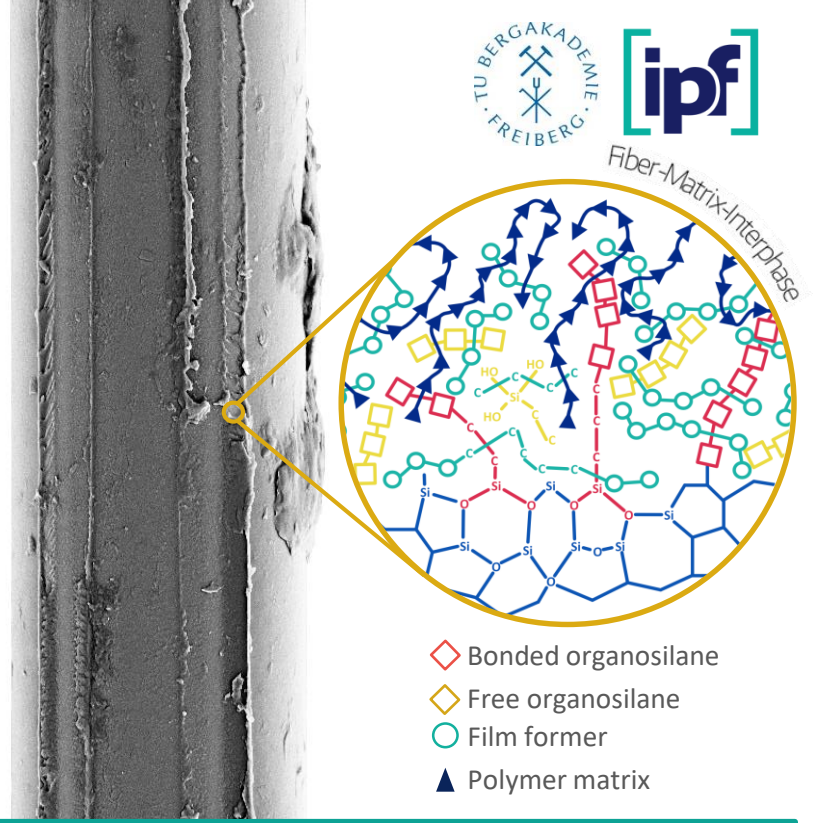
# Novel Biocomposite Materials - Sizing




**Sizing**
**the property-decisive interface**

- Biobased + degradable

1) Increase tensile strength
2) Protection of the fiber
3) Fiber-matrix-adhesion




**My aim**: development and analysis of a suitable biodegradable and biobased sizing for degradable glass fibers



Representation of the fiber-matrix-interphase according to *Petersen. Technical University of Denmark 2017, PhD thesis*

# Materials – Glasses and Sizing Components

## Glasses

| parts | E-Glass M.-% | 45S5 M.-% | 18-06 M.-% |
|---|---|---|---|
| $SiO_2$ | 53 | 45 | 65 |
| $Al_2O_3$ | 15 | - | - |
| $P_2O_5$ | - | 6 | 0 |
| $B_2O_3$ | 5 | - | 1,5 |
| $Na_2O$ | - | 24 | 18,4 |
| $K_2O$ | - | - | - |
| CaO | 19 | 24 | 15,0 |
| MgO | 4 | - | 0,1 |

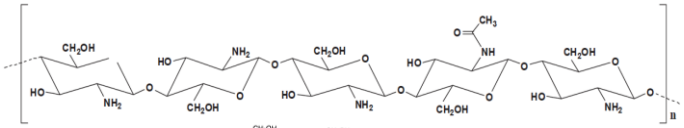
Spinning trials

Dip coating trials

## Sizing: **Water based**

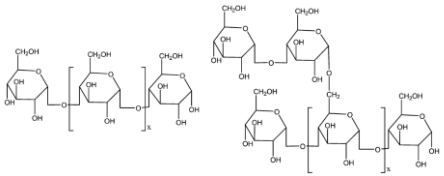
### Bio based film formers

Chitosan



(1)

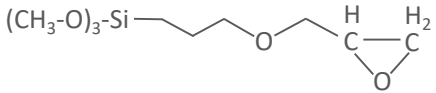
Starch



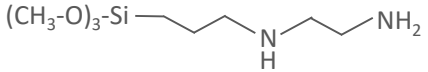
(2)

### Silane coupling agents

GPTMS – Epoxysilane



N-(2-Aminoethyl)-3-APTMS - Diaminosilane





(1) Marks et al., ISSN 0025-5866
(2) Mekonnen, T. et al., J. Mater. Chem. A, 2013, 1, 13379

# Workflow – Fiber Processing and Investigation

Single fiber dip coating

Glass 18-06 — unsized

coupling agents | Film formers | Blends

Fiber drawing in house

E-Glass — unsized

coupling agents | Film formers

GPTMS | APTMS | Chitosan | Starch

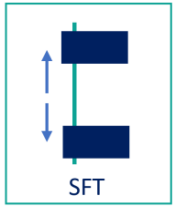
Chitosan + GPTMS | Starch + APTMS

Fibre drawing

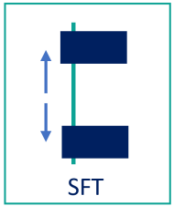
Hydrolytically active glass 15-06

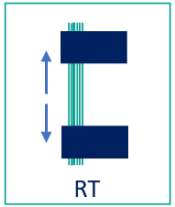
Successful trial in pilot plant

- Thermal properties of films
- SEM/EDX
- Single Fiber Tensile test (SFT)



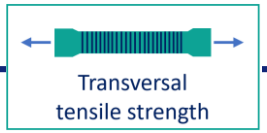

- Sizing Content
- SEM/EDX
- Single Fiber Tensile test (SFT)
- Roving Tensile test (RT)



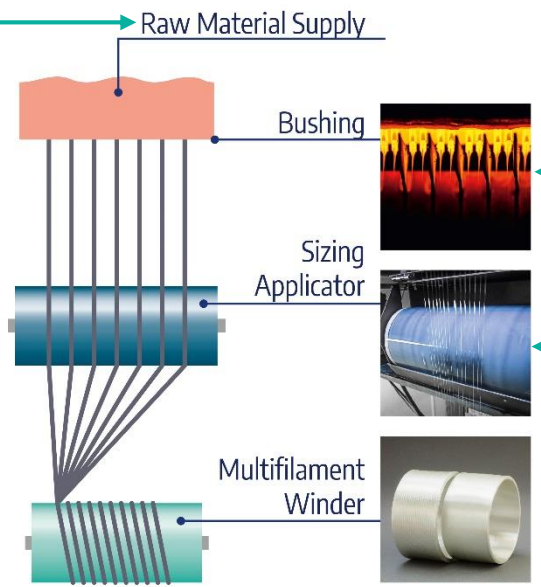



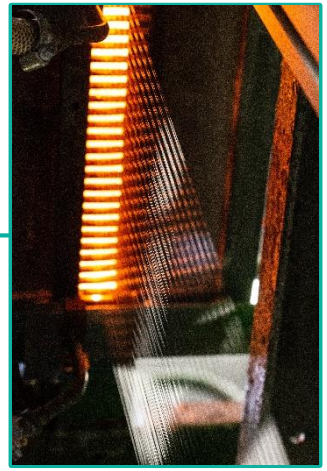

**Composite manufacturing** of unidirectional plates by winding **with PLA filaments**

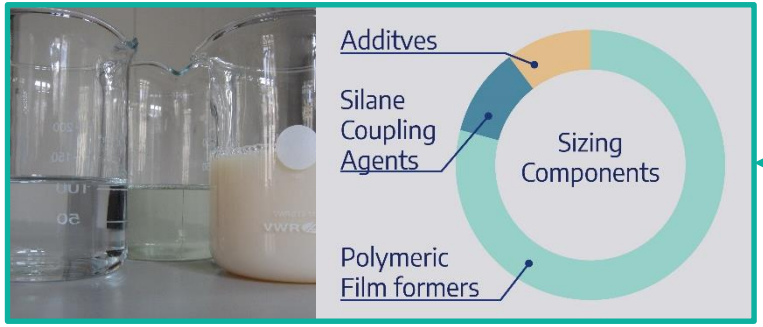
→ Transverse and longitudinal strength

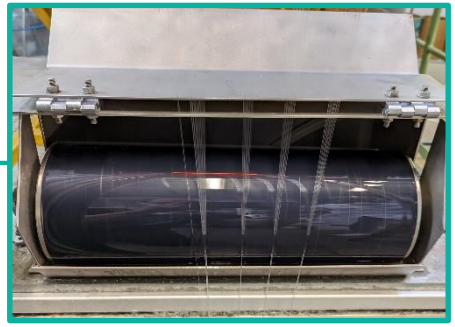



# Melt Spinning of Fibers

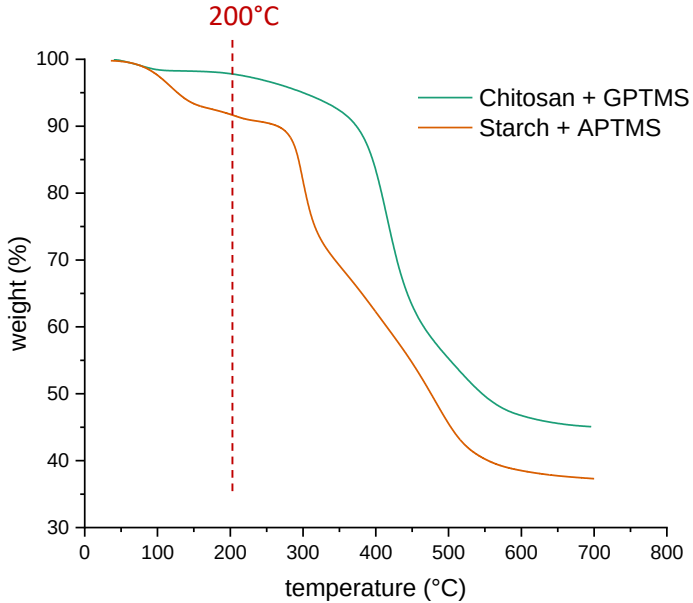


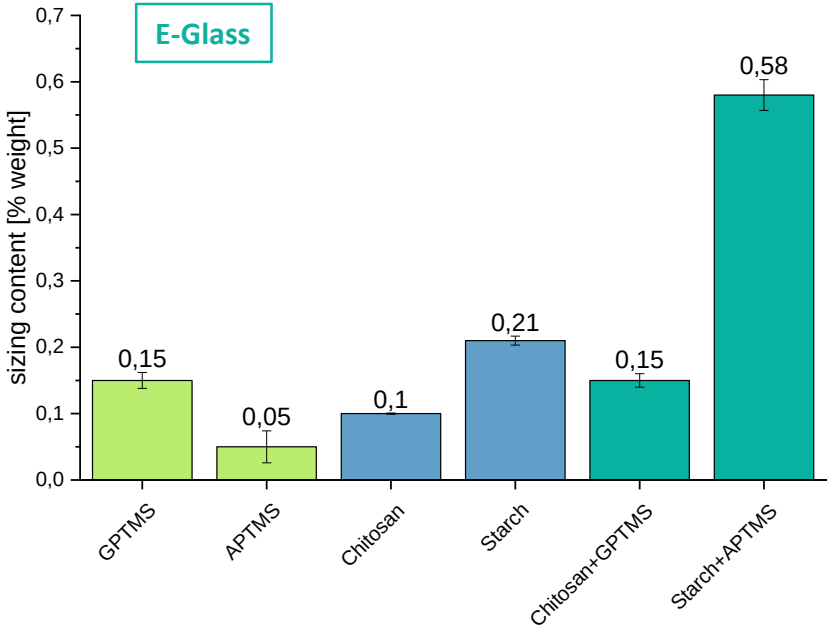





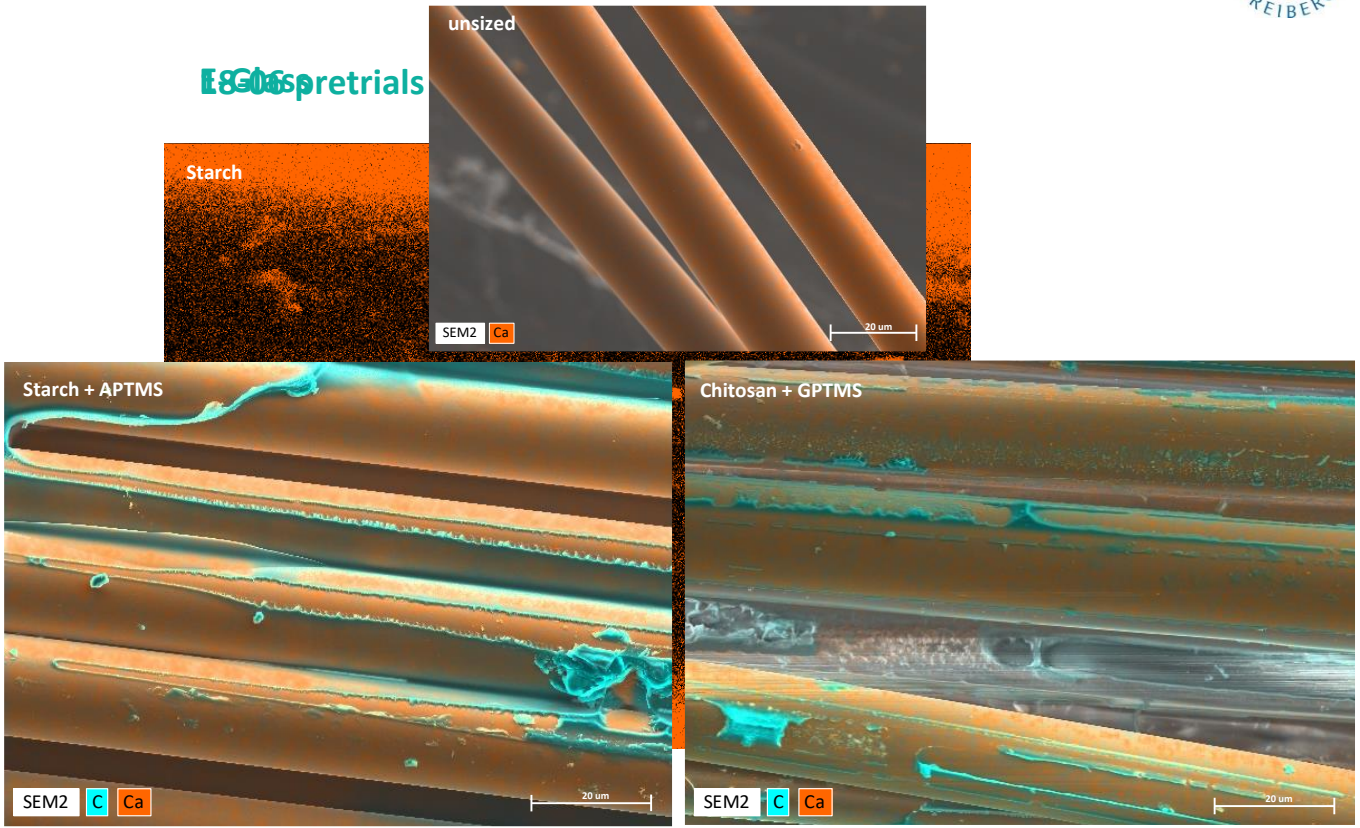
# Results – Thermal Properties and Sizing Content



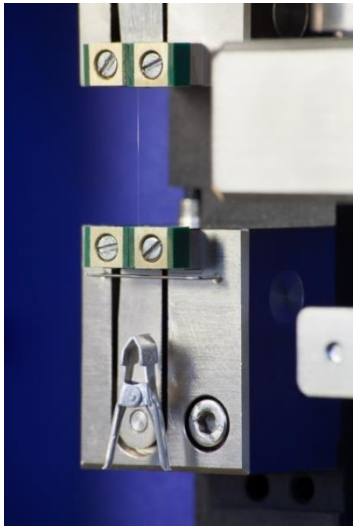

- Macroscopic films:
  - Homogeneous film forming observed
  - Thermal stability over 200°C for all film formers
- Sizing content was determined gravimetrically

# Results – Sizing Characterization - SEM/EDX



# Results – Single Fiber Tensile Tests

**Objective: increase of single fiber tensile strength through the sizing**

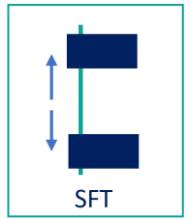


Weibull-distribution:

- 50 samples measured
- Probability of failure (P) is assigned to every fracture strength (*σ)*

$$P = 1 - e^{-(\frac{\sigma}{\sigma_0})^m}$$

- **Weibull modulus** *m*
  = homogeneity of distribution of flaws in the glass fibre network
- **Weibull characteristic strength** $\sigma_0$
  = 63,2% of fibres break at this value



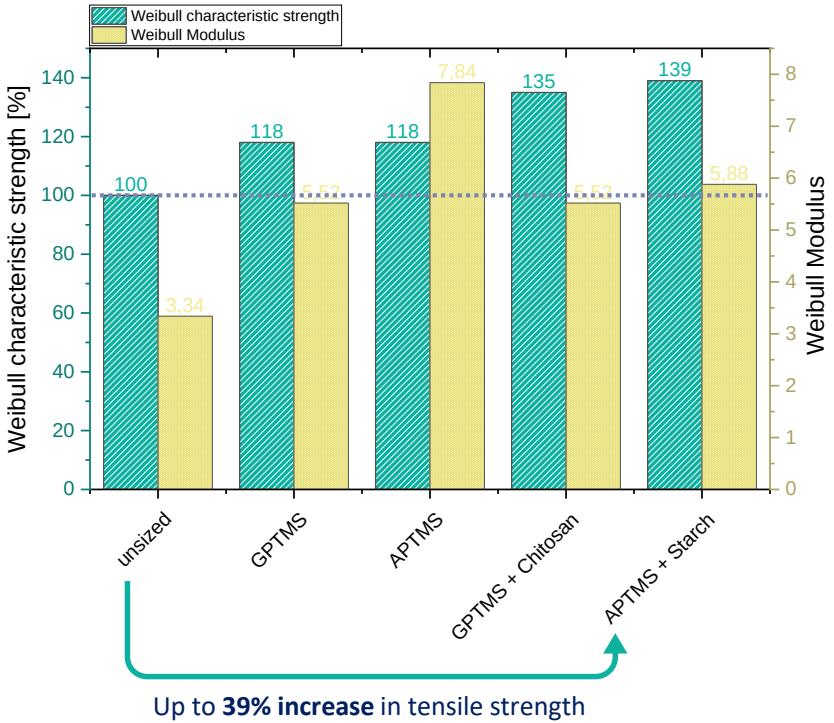



Weibull characteristic strength: A statistical strength value with a probability of failure at 63.2 % of the fibres

# Results – Single Fiber Tensile Tests

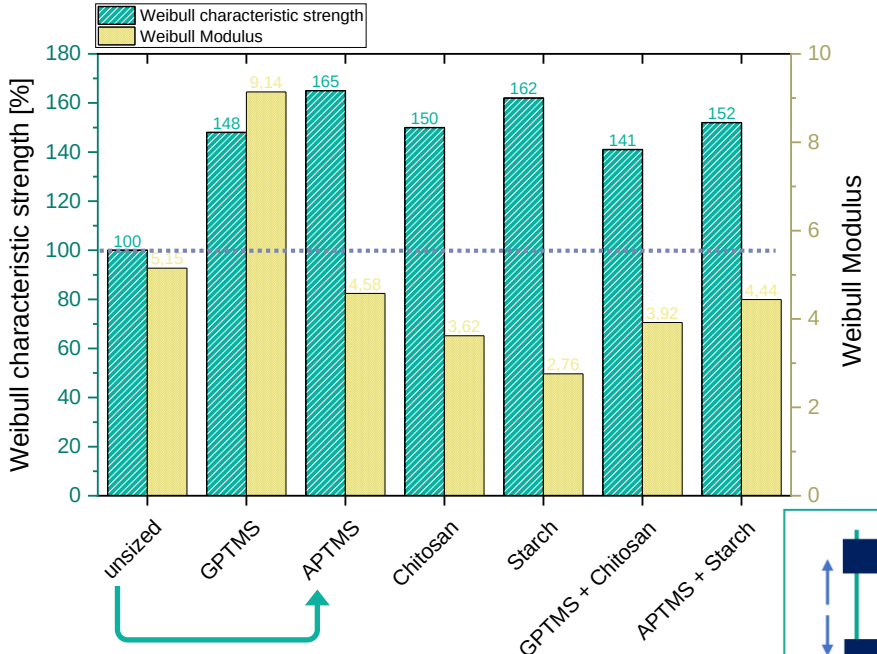
**Objective: increase of single fiber tensile strength through the sizing**

## Dip-coating Pre-trial - 18-06

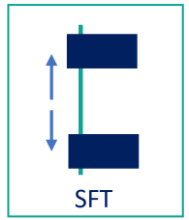



Up to **39% increase** in tensile strength

## Fibre Drawing - E-Glass




Up to **65% increase** in tensile strength

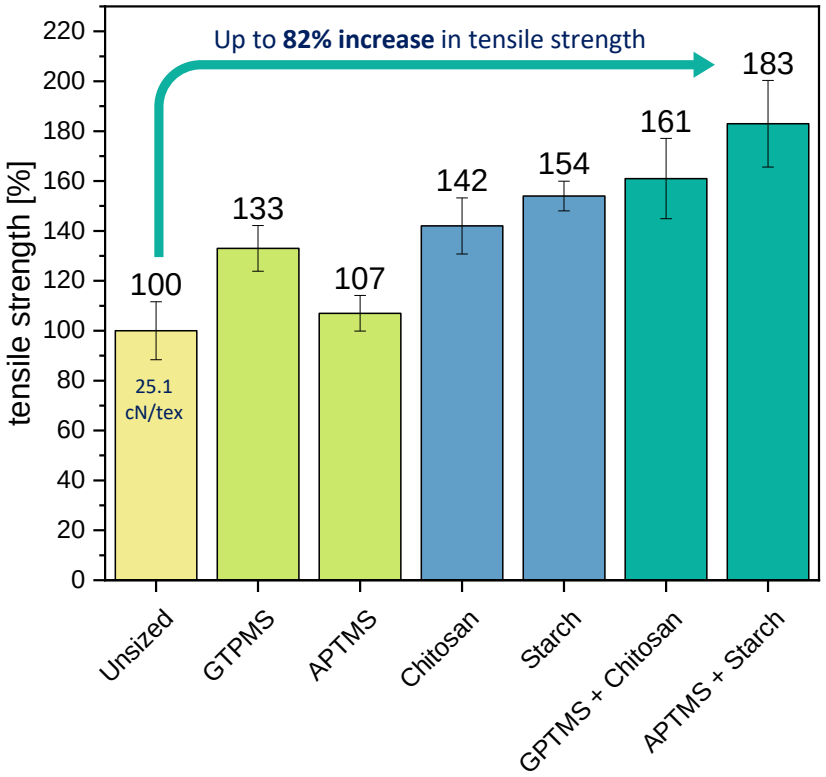





Weibull characteristic strength: A statistical strength value with a probability of failure at 63.2 % of the fibres

# Results – Roving Tensile Tests on E-Glass

**Objectives**: filament-to-filament adhesion, transmission of force




**Two effects strengthen the yarn:**

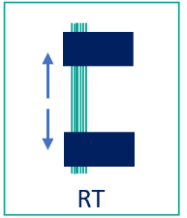
→ *On fiber scale*:
healing of defects by silane

→ *On yarn scale*:
filament-to-filament adhesion by film formers

→ Good filament-to-filament adhesion on the yarn with bio based film formers

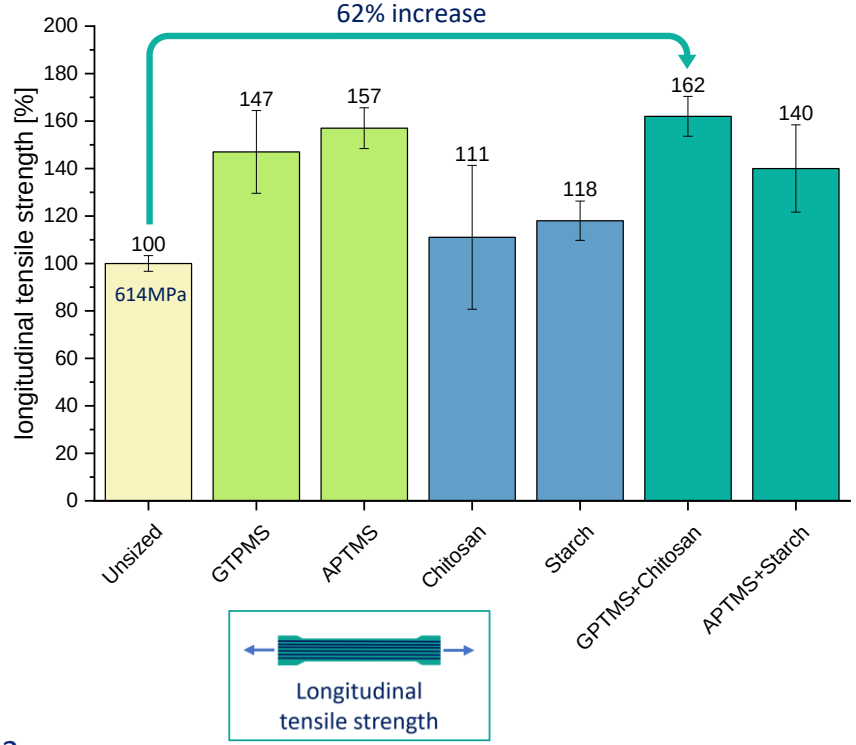
**Feel and condition of the yarn:**
When bio based film formers applied

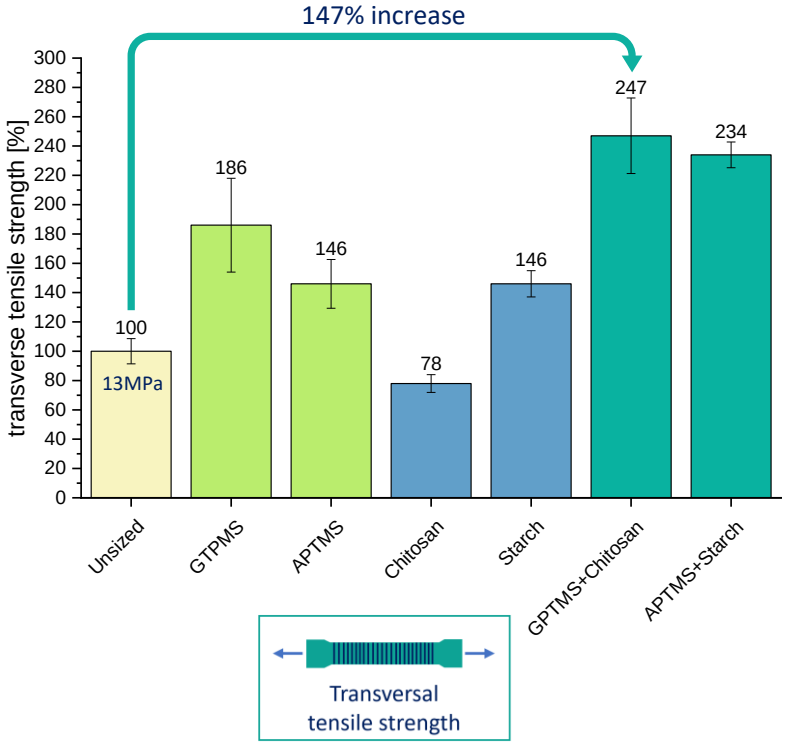
- **yarn closure – no splicing**
- **processability – easy to pull from the spool**



# Results – Composite Manufacturing and Tensile Testing

**Objectives**: filament-to-matrix adhesion, protection of the fiber through sizing, yarn closure/processability

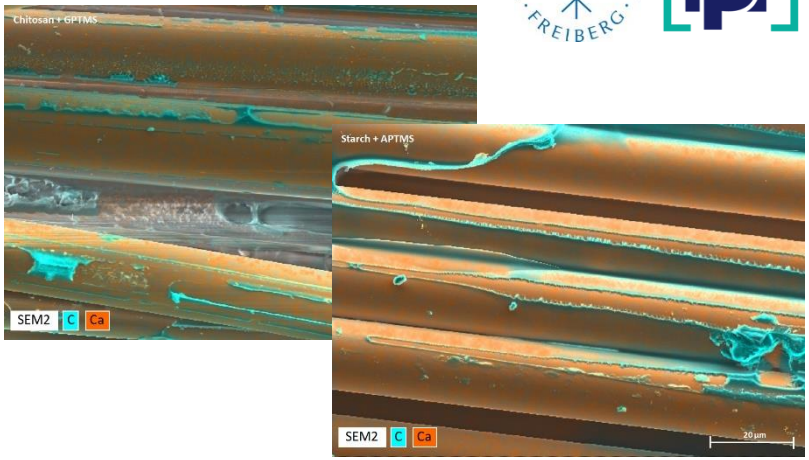


# Summary and Outlook

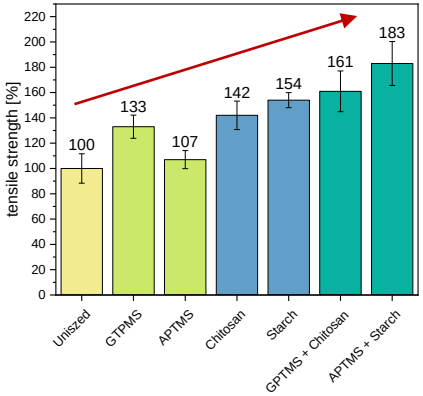
- Improvements in tensile strength, fiber matrix adhesion and processability
- Simple and biobased sizing formula

→ Publication in progress
including spinning of bioactive glass fibres




## Next steps:

- Optimize sizing (further film formers, tailor amount of all components) and spool geometry for better processability
- Go into application (medical or technical)



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# Thank you!


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