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TEXTIL+FASERFORSCHUNG

Manufacturing of fiber reinforced phosphate geopolymer composites

S. K. Selvarayan, M.U. Witt, A. Shaik, F. Wollenhaupt, B. Heidenreich (DLR)

12.07.2023 Geopolymer Camp 2023, Saint-Quentin

DITF - Europe‘s Largest Textile Research Center

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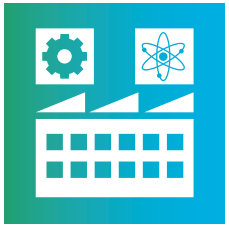
Key Figures 2022

Employees

approx. **220**

Turnover

12 m € public
13 m € industry



Area

25,000 m²

Research

189 public
572 industry

Partners

1158 enterprises
67 % SME

Services

approx. **100**
test customers
5 small batch series

Textile Vertical Integration

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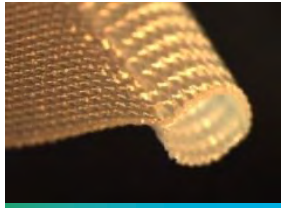
FROM MOLECULES TO PRODUCTS

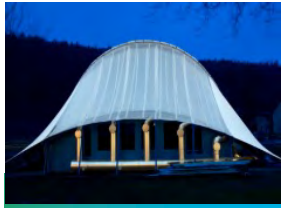
Polymer

Fibers and Yarns



Fabrics

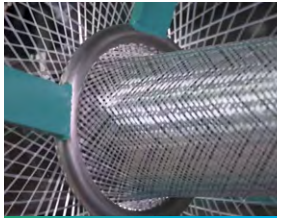
Functionality



Product

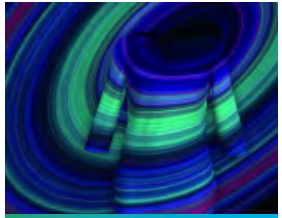
RESEARCH FIELDS

New Materials

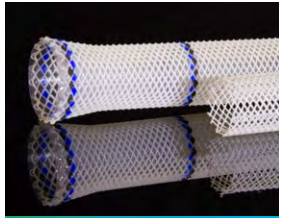


Lightweight Construction

Sustainability

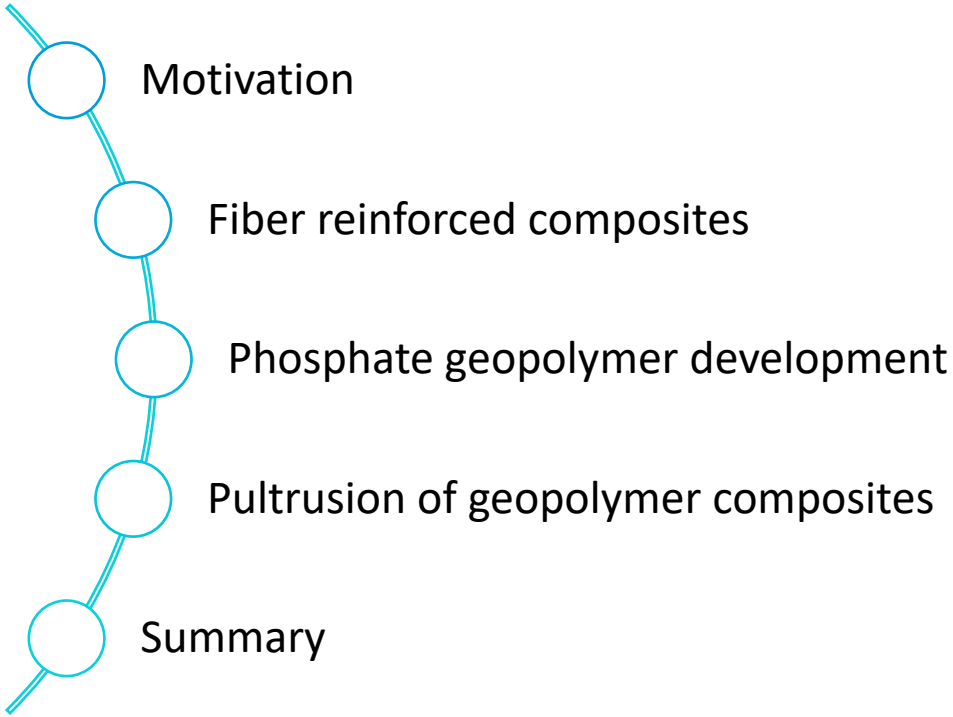


Digitalization

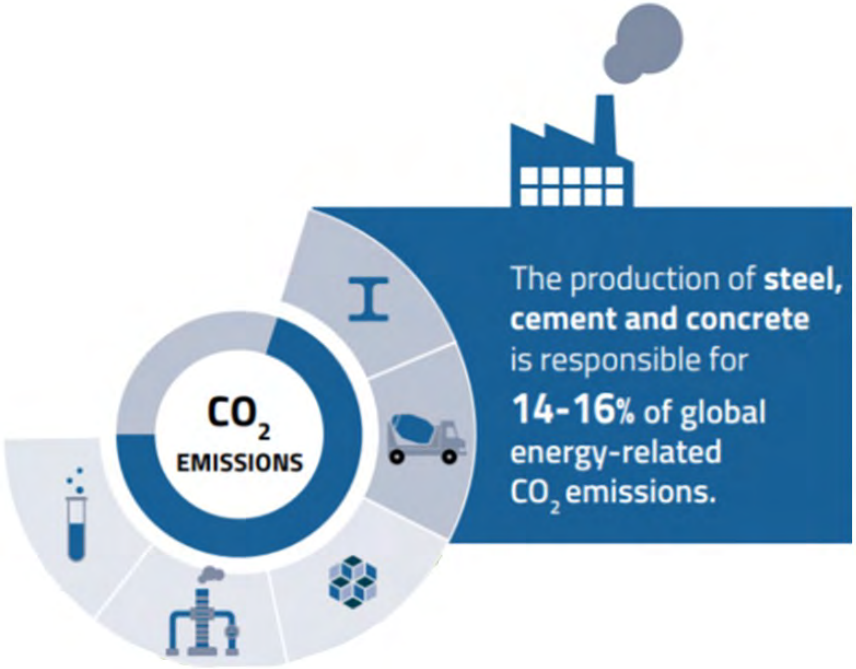


Health

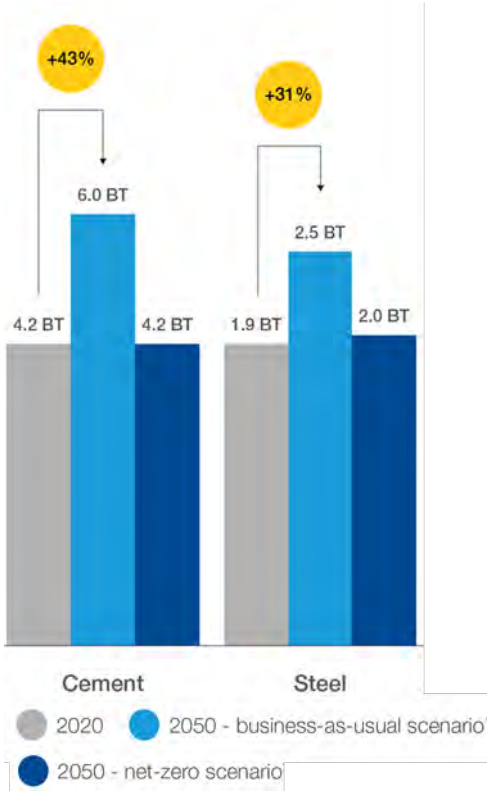
Agenda


Motivation
Fiber reinforced composites
Phosphate geopolymer development
Pultrusion of geopolymer composites
Summary

Motivation



Source: UNIDO



Source: World Economic Forum

Sustainable development goals

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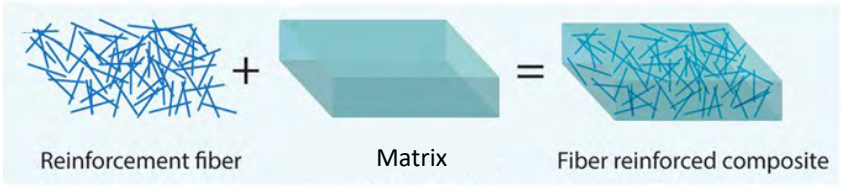
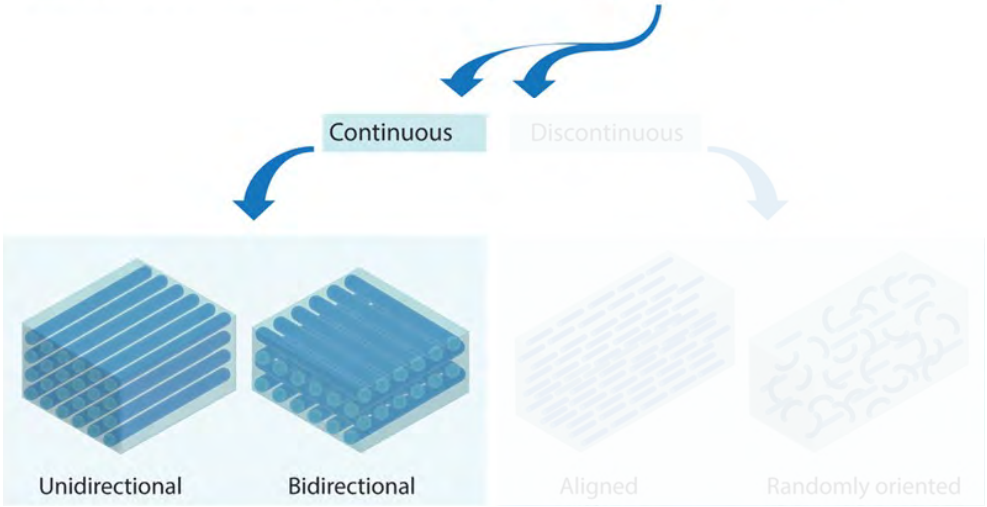
Fiber reinforced composites

Fiber reinforced composites





Source: Advanced Sustainable Systems, Volume: 6, Issue: 11, 2022, DOI: (10.1002/adsu.202200258)

Lightweight construction with FRPs

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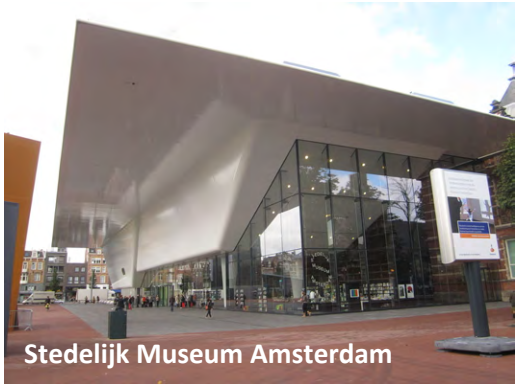
Advantages of FRPs

- Weight saving (lightweight)
- Corrosion resistance
- Free formability
- Tailor-made solutions
- Cost-effective due to high specific properties

Fire safety ??

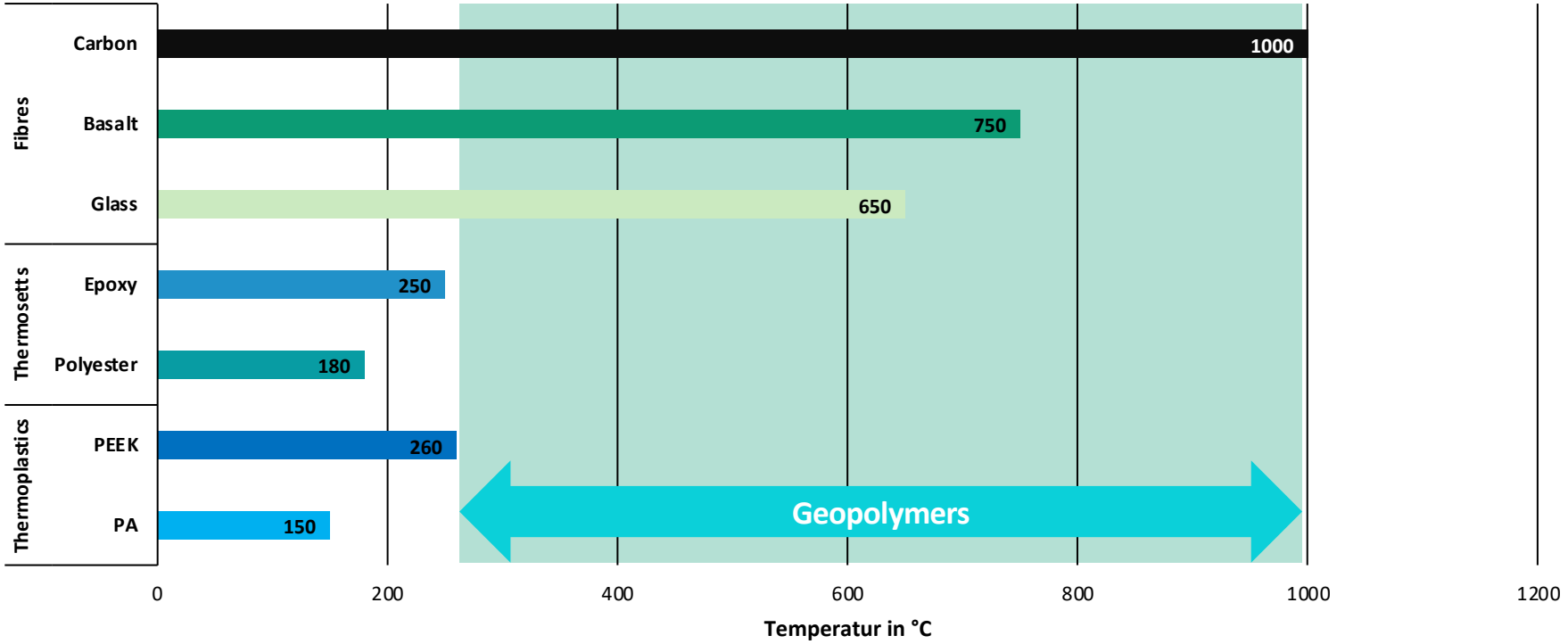

EDEM, Escuela de Empresarios


Stedelijk Museum Amsterdam

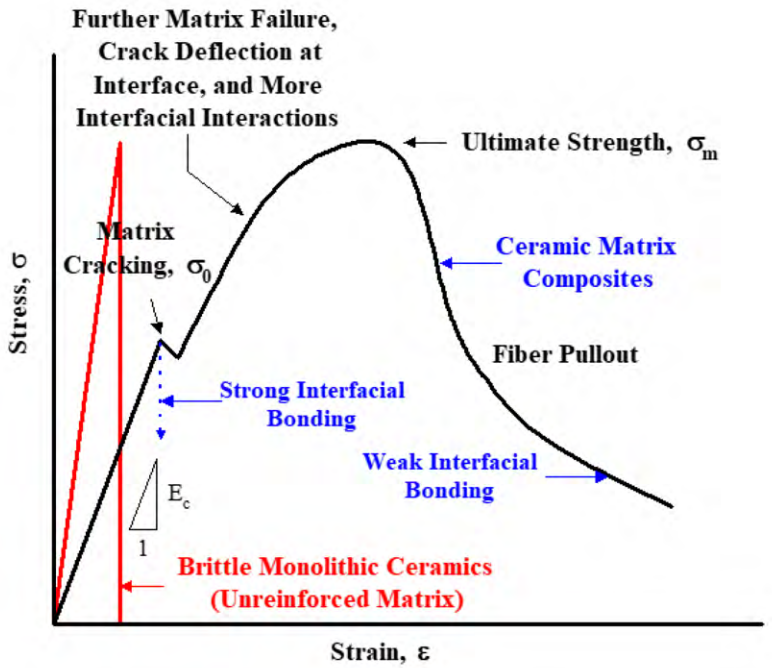

FRP Rebars

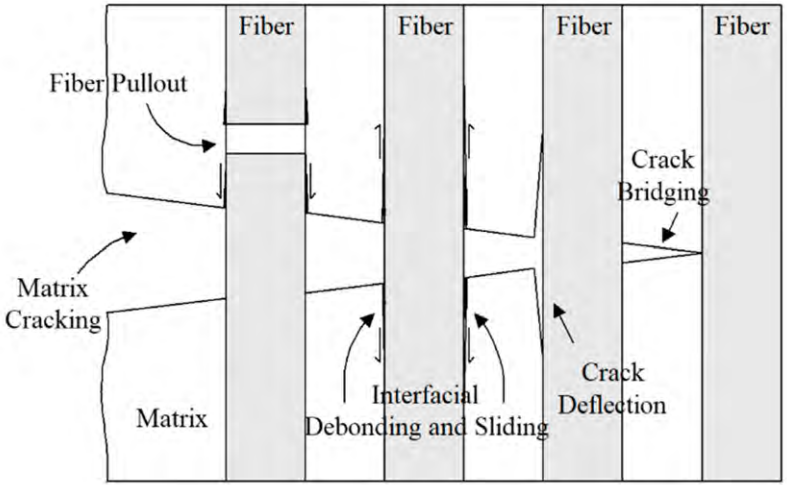

Fiberline Bridge in Kolding, Denmark

Application temperatures of typical Fiber Reinforced Plastics



Why to reinforce with fibers?





J. Kim, Materials 2019, 12(18), 2997; https://doi.org/10.3390/ma12182997

Geopolymer composites in use

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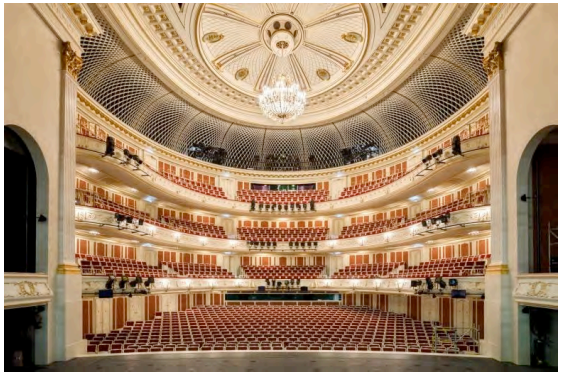
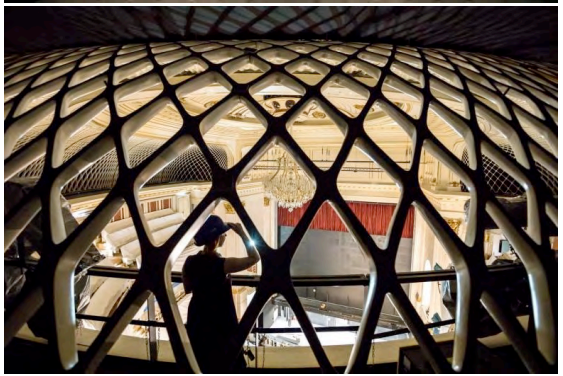
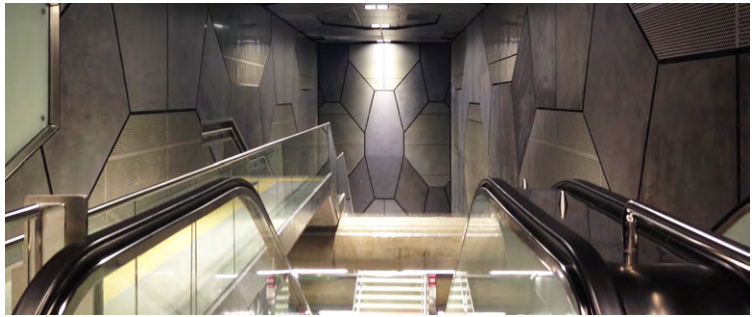




Vubonite (CBPC)



Keraguss

Keraguss and Vubonite systems

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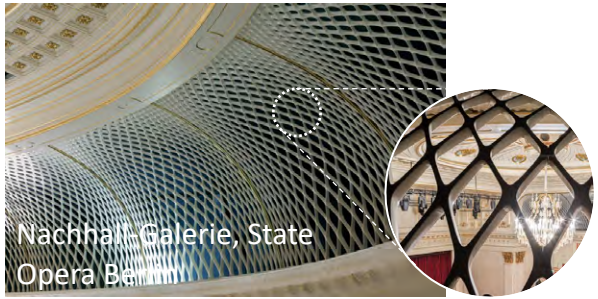
Keraguss

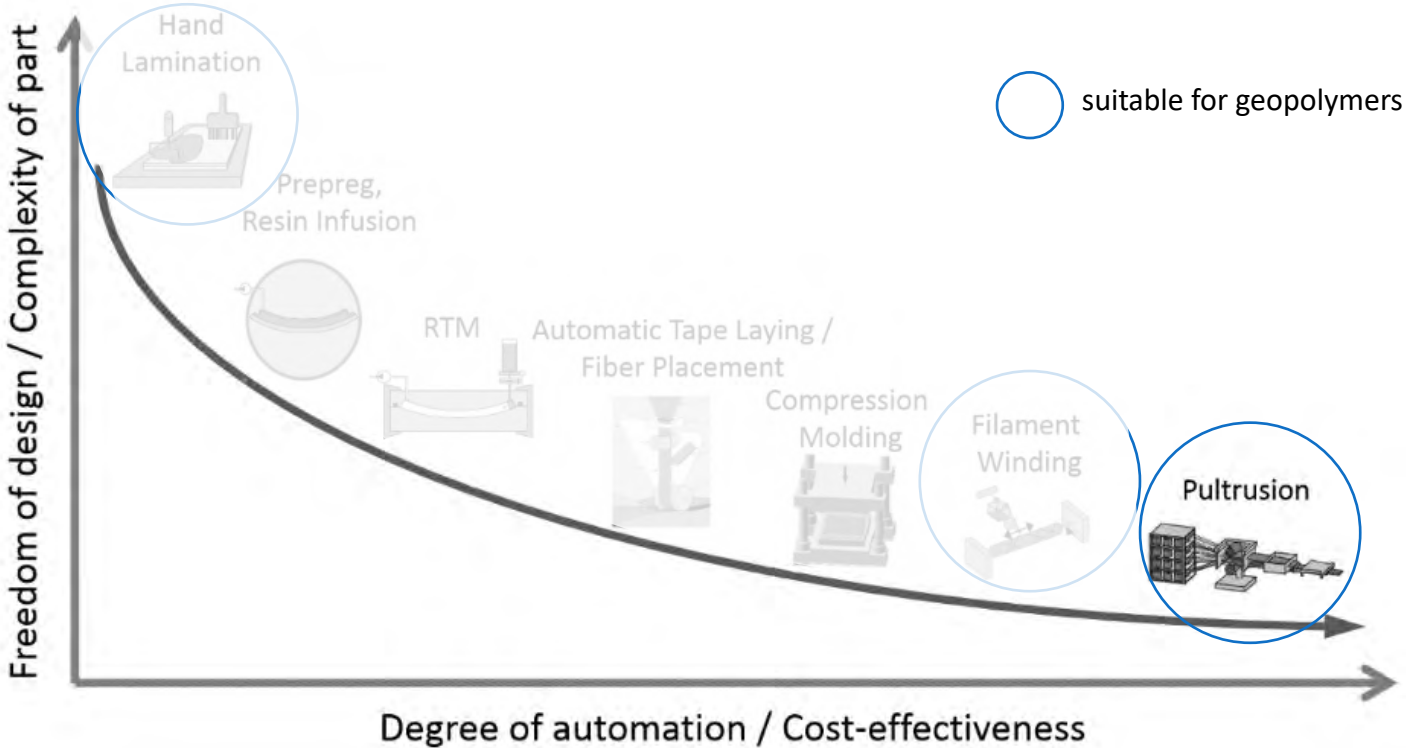
- Liquid: Water glass
- Alkaline → only Carbon or AR-Glass fibers
- Solid: Aluminosilicate
- Fire protection class: A1
- Pot life: approx. 1-2 h
- Solvent: Water

Vubonite



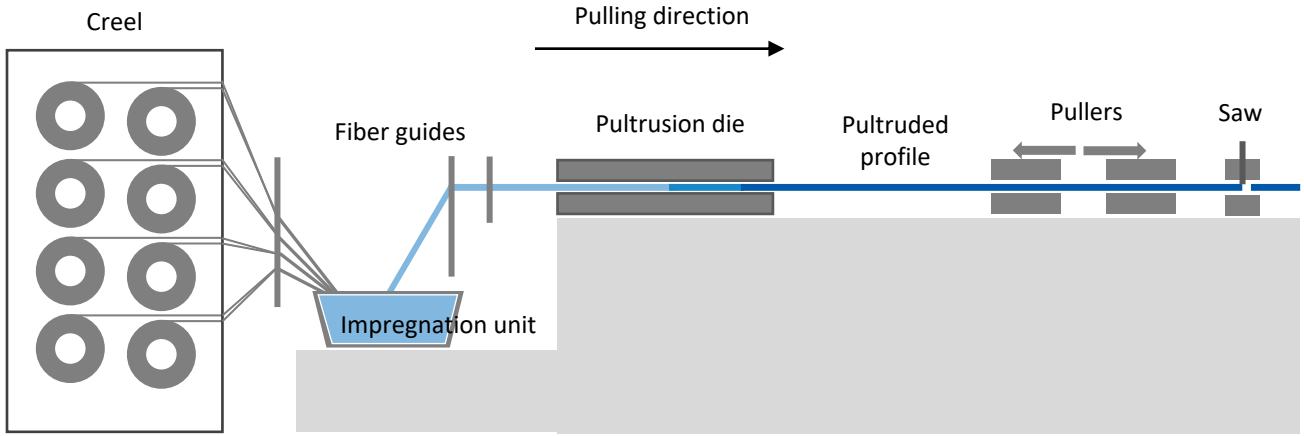
- Liquid: Phosphoric acid
- Acidic → All types of high-performance fibers
- Solid: Calcium silicate
- Fire protection class: A1
- Pot life : approx. 45 min
- Solvent: Water

Composite manufacturing

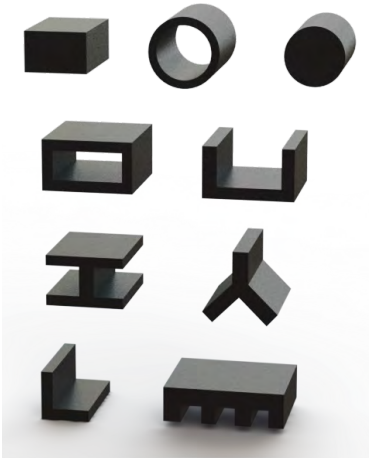


M. S. Renato and M. Bezerra, “Modelling and Simulation of the Closed Injection Pultrusion Process,” 2017

Pultrusion process

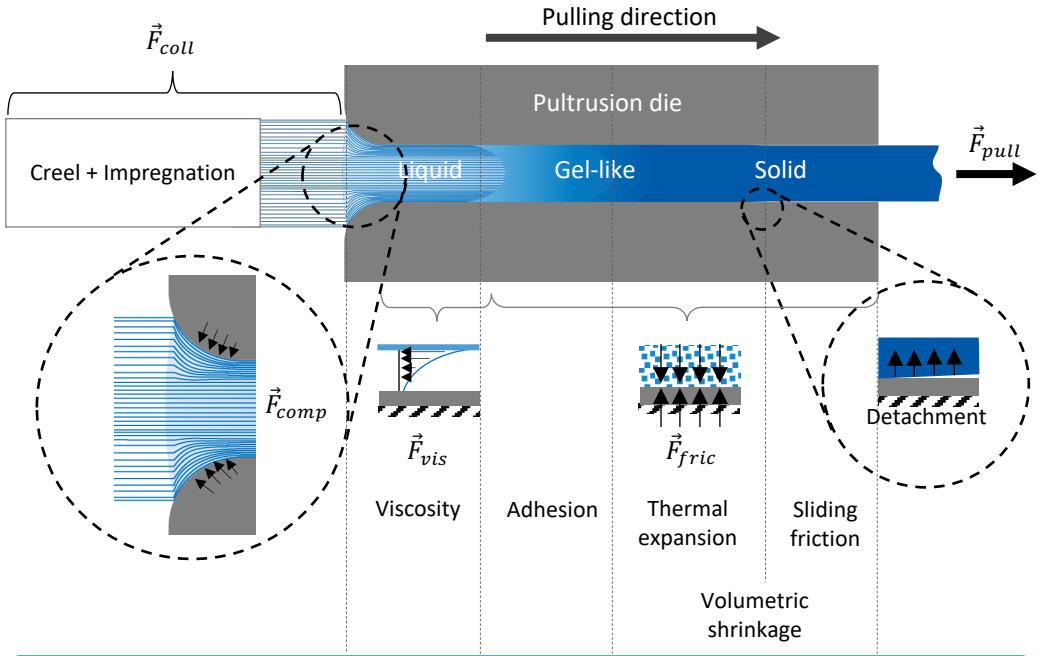


Thermoset pultrusion line



Typical profile cross-sections

Die Dynamics in Pultrusion process



Before the tool

- Reinforcing material pre-tension
- Friction in the guide elements
- Friction in the impregnation unit

In the die

- Compaction of the reinforcing material
- Viscosity resistance
- Thermal expansion
- Adhesion
- Friction

$$\vec{F}_{pull} = \vec{F}_{coll} + \vec{F}_{comp} + \vec{F}_{vis} + \vec{F}_{fric}$$

Matrix requirements

Material properties

- Morphology
- Mechanical properties
  (χ_i, additives, H_20, pH, T, rH….)

Processability

- Fiber wetting (η, γ_{sl}, μm)
- Pot life (K, pH, T,..)
- Corrosiveness (pH,..)
- Filler content (V_f + V_s)
- Curing (K, pH, T, k)

Material development

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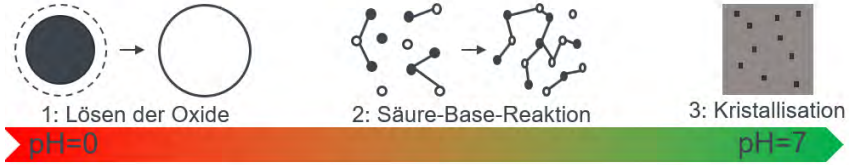
Recipe	Components	Weight [%]
NiBreMa#	Aluminum Dihydrogen Phosphate	21,50
	Dipotassium hydrogen phosphate	10,80
	Water	32,50
	Metakaolin	35,20
Vubonite*	Phosphoric acid	27,8 - 33,3
	Zinc oxide	0,6 - 5,6
	Water	27,2 - 16,7
	Vubonite powder**	44,40
Calcium silicate	Phosphoric acid	29,00
	Zinc oxide	2,80
	Water	23,80
	Calcium silicate	44,40

First recipe

*Proportions only approximate according to data sheet

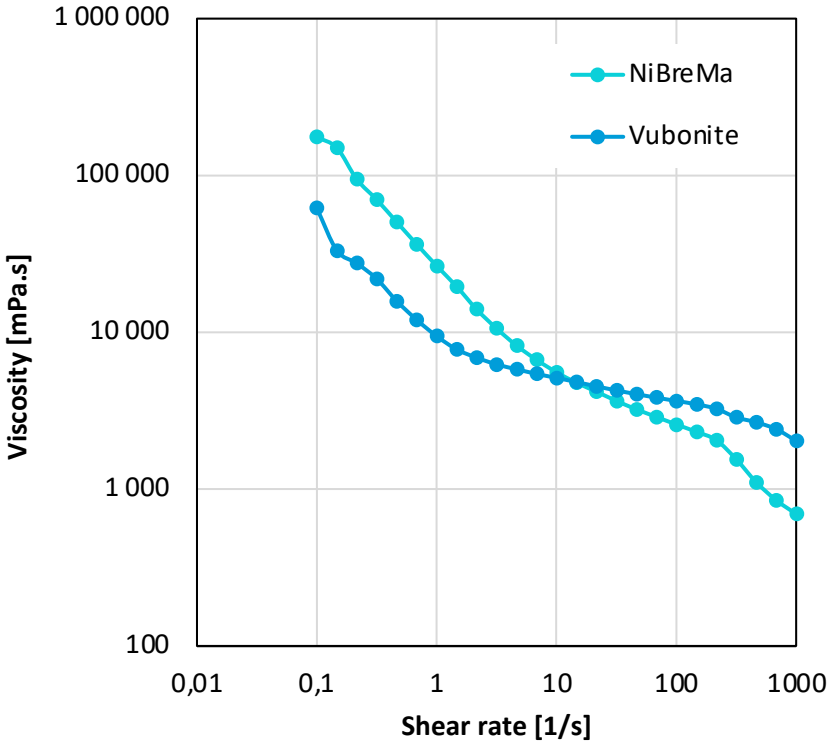
**Vubonite : Calcium silicate + ?

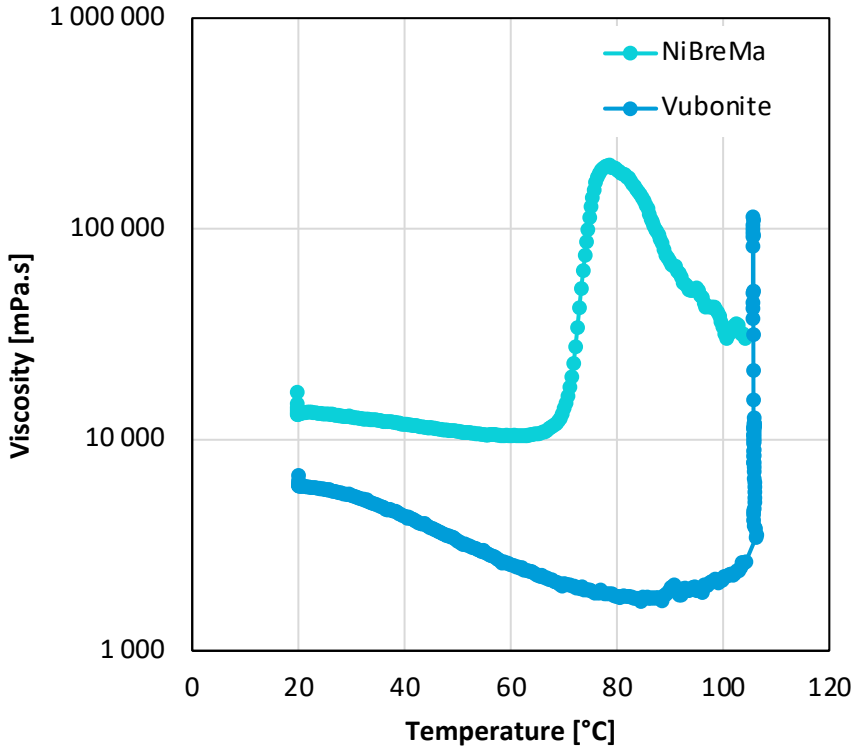
- Reaktionsmechanismus läuft in drei Schritten ab:



Property	NiBreMa	Vubonite	Calcium silicate
Compressive strength [MPa]	11,8	123	20,9
Compression modulus [GPa]	1,2	14,5	2,8
Density [g/cm^3]	1,3	1,6	1,4
Shrinkage [%]	2,7	0,2	-
Porosity [%]	46	35	39,4

Rheological properties of the slurry





Pultrusion line for manufacturing phosphate composites

Pultrusion line for manufacturing phosphate composites

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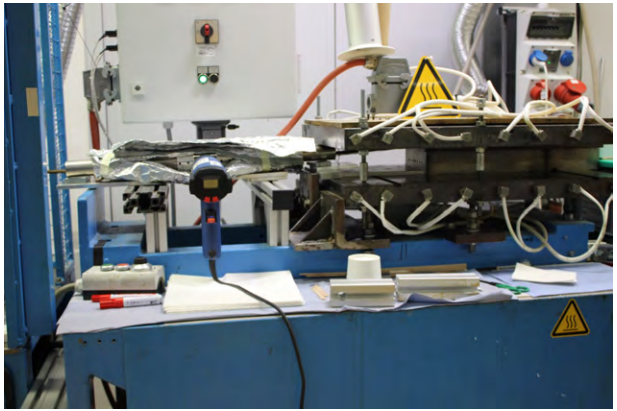

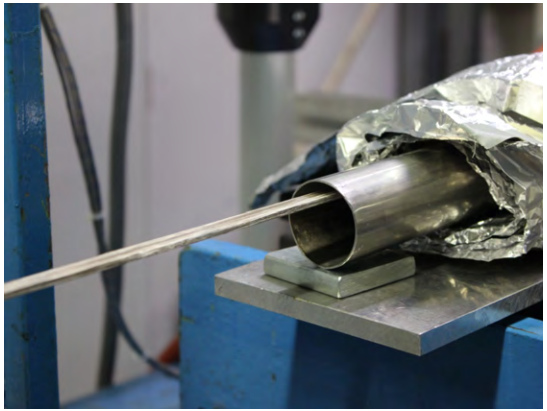

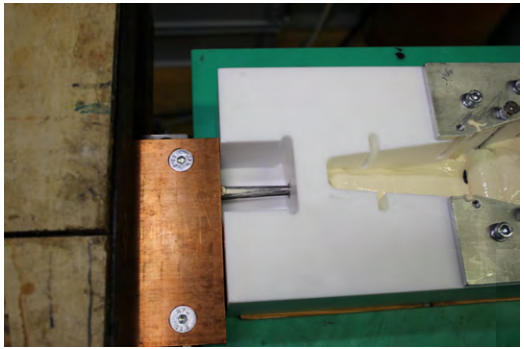

Pultrusion parameters

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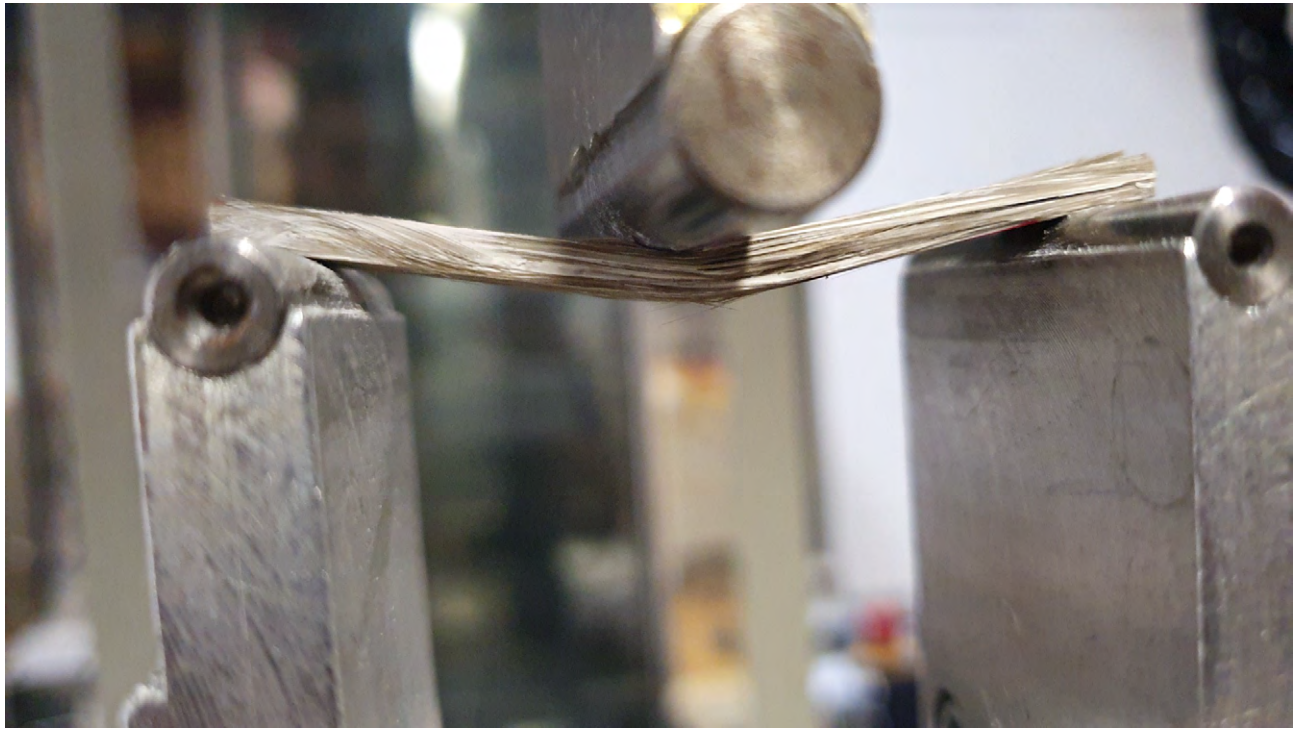
Basalt fiber: 2400 tex (24 x 100 tex) → 2 different fibers with different sizing

DBF GmbH (Natural basalt)

Isomatex (Synthetic basalt)

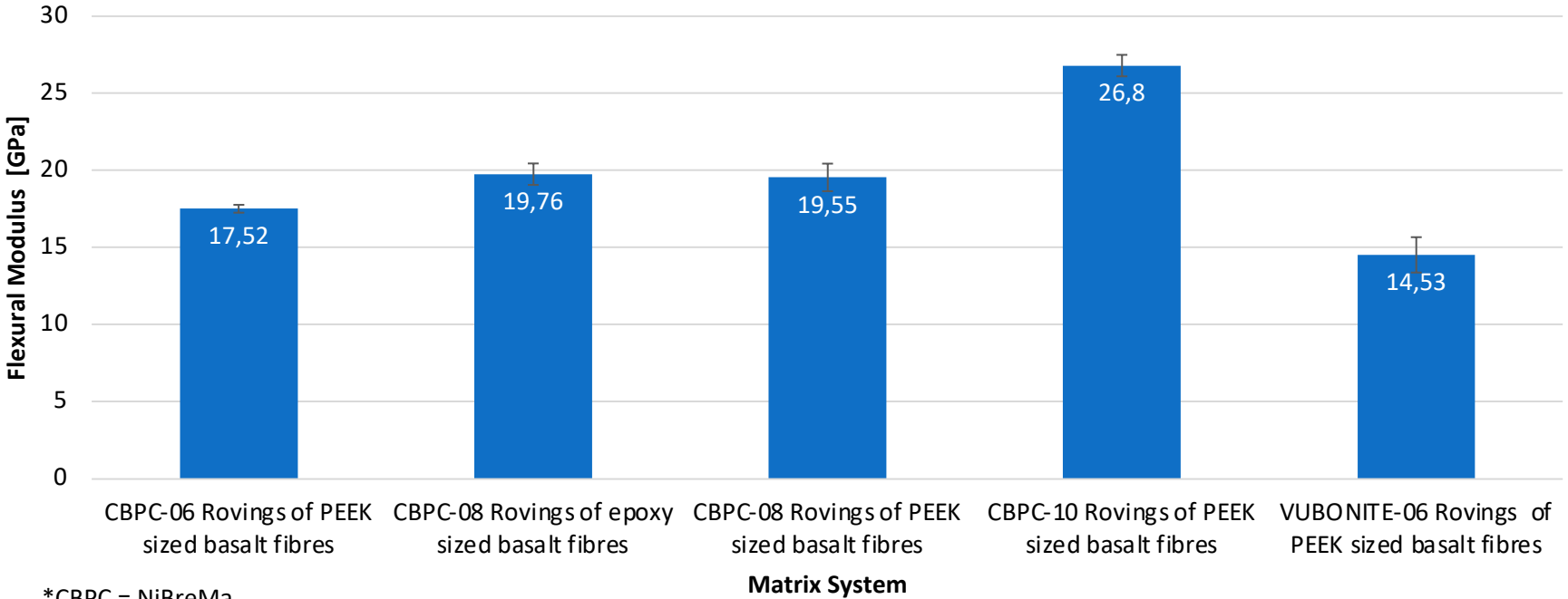
Parameter		Value				
Profile geometry	Rectangular	10 x 3 mm^2				
	Circular	4 mm ∅				
	Number of rovings	6	8	10	12	14
Fiber volume content (%)		18	24	30	36	42
Pulling speed (cm/min)		25, 35, 50				
Pultrusion die temperature (°C)		120				
Post curing temperature (°C)		200, 250, 300, 350				

3-Point bending test



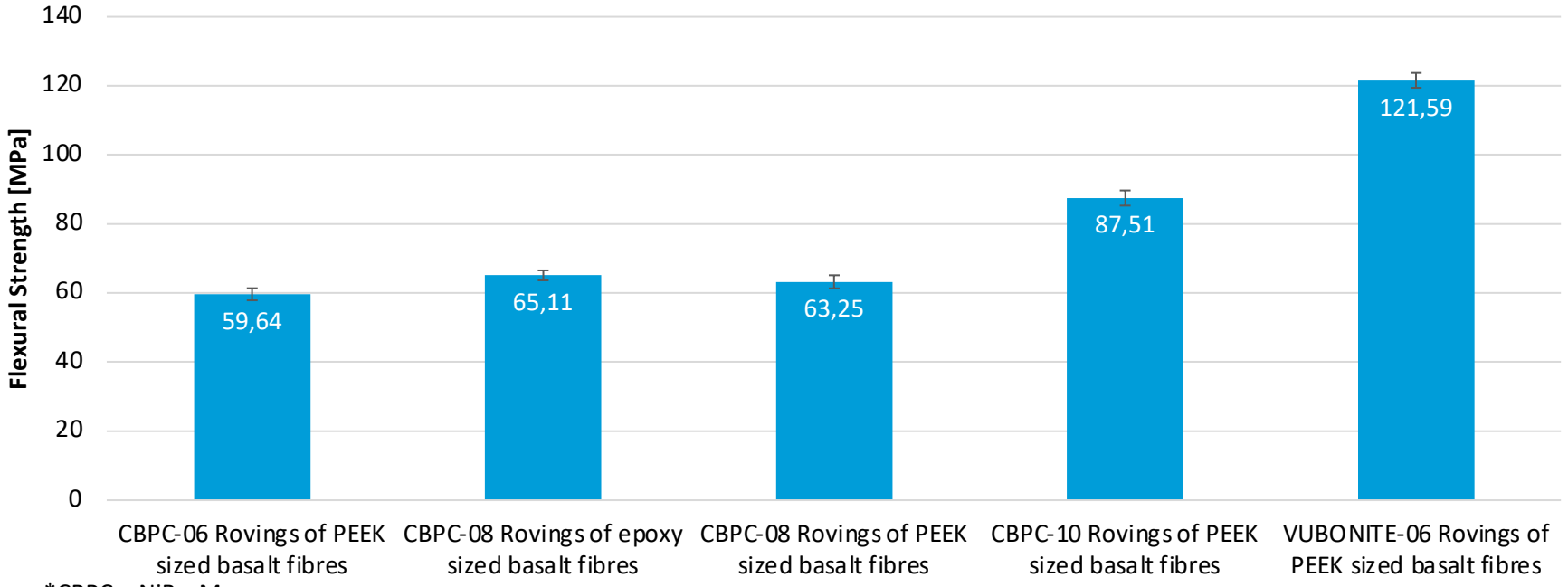
DIN-EN 685-3

Flexural properties of basalt reinforced composite



*CBPC = NiBreMa
25 cm/min
300 °C Post-Cure

Flexural properties of basalt reinforced composite



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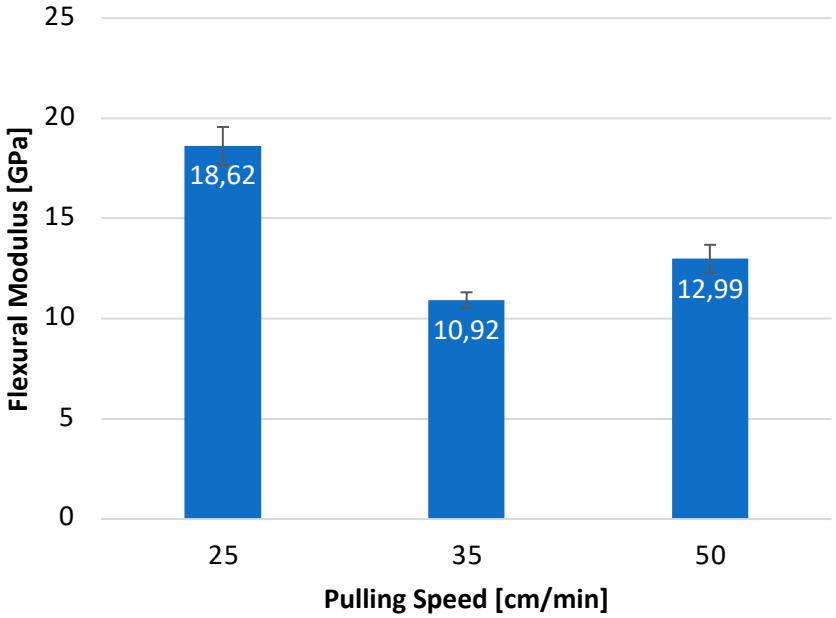
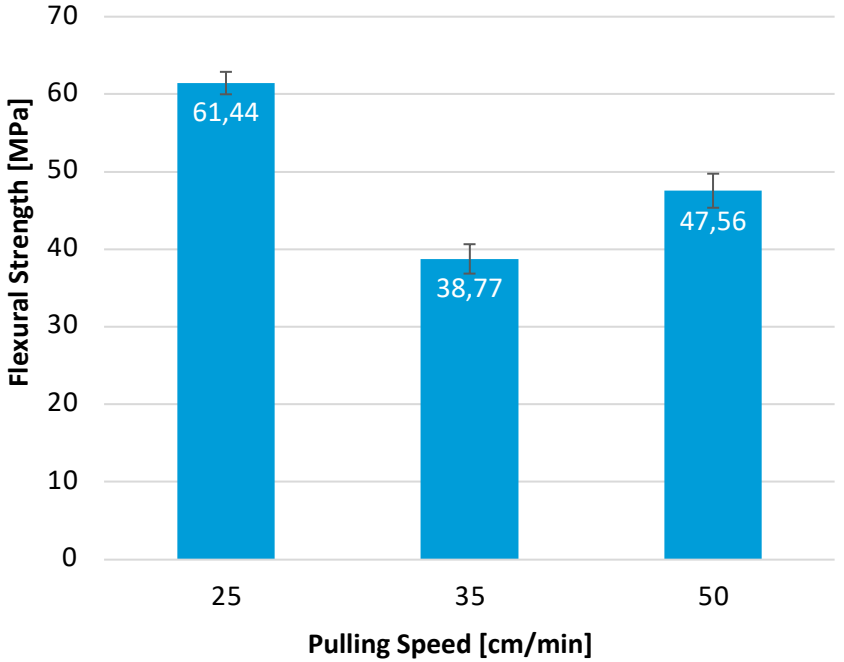
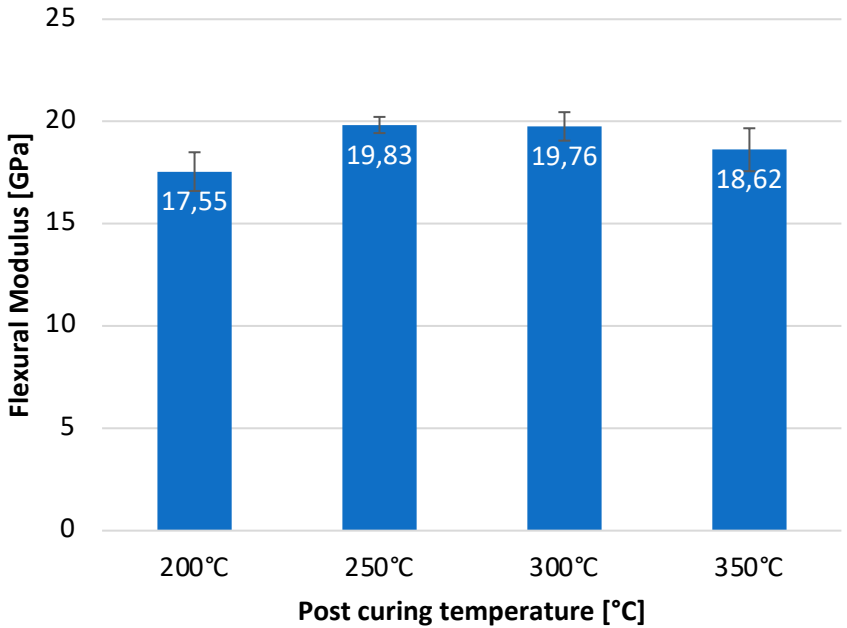
Flexural properties of basalt reinforced composite

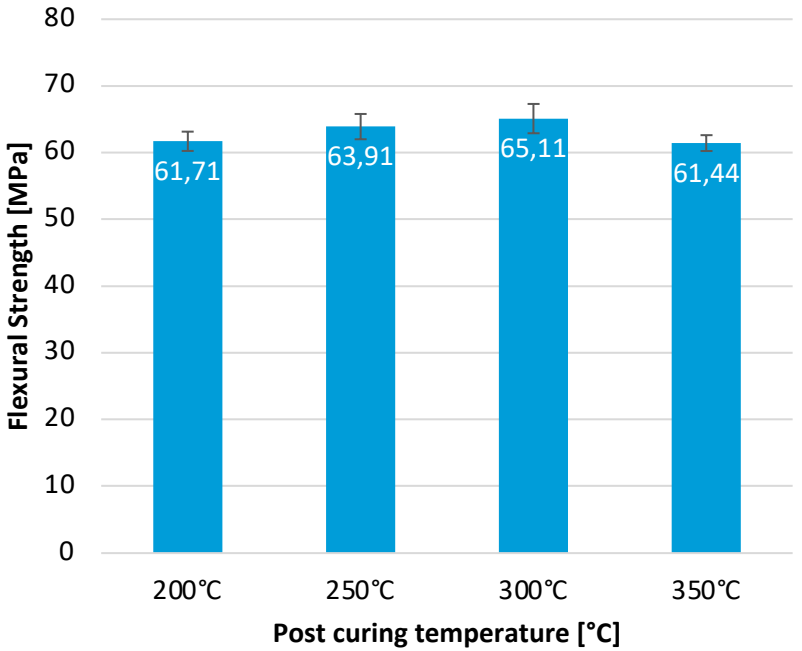




8 Rovings
350 °C Post-Cure

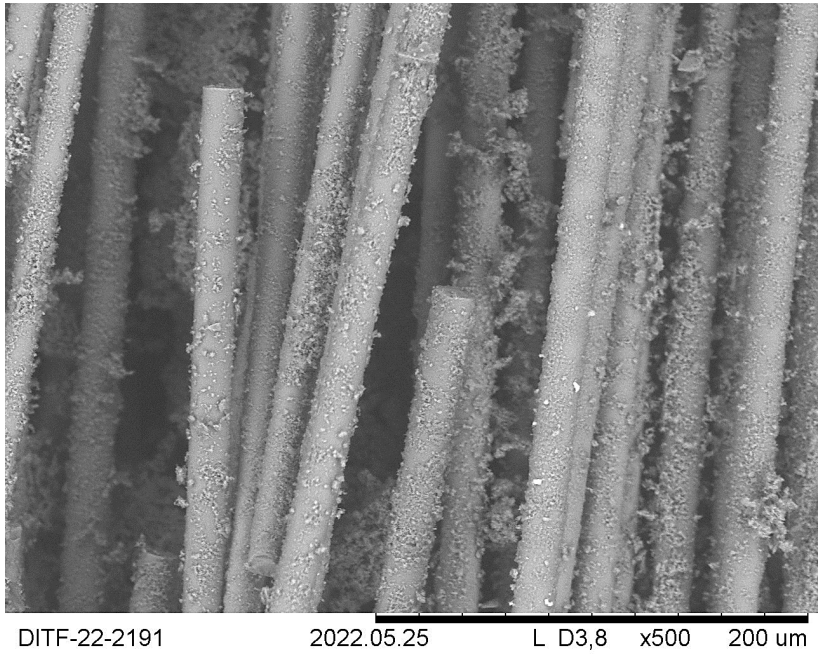
Flexural properties of basalt reinforced composite

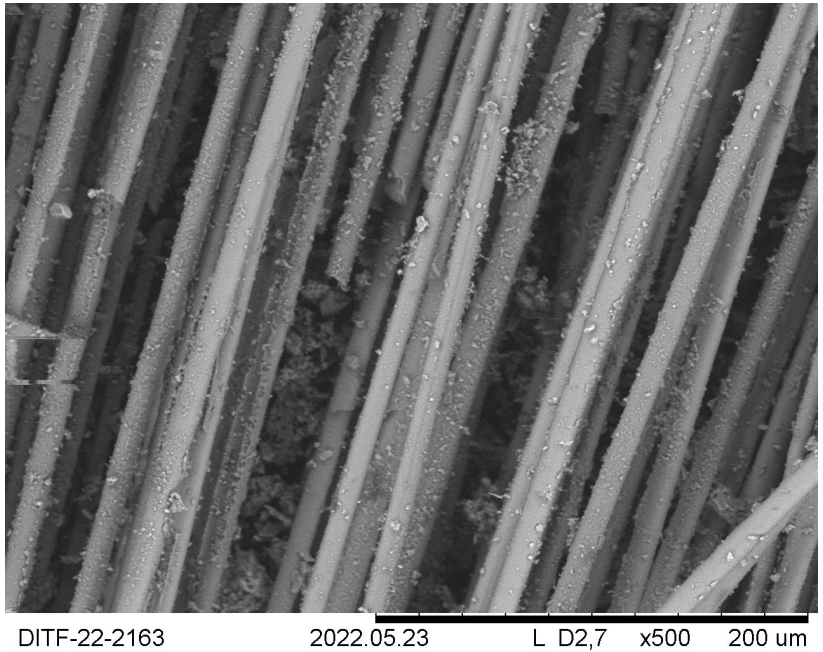




8 Rovings
25 cm/min

SEM Imaging





Weak matrix composite

High-temperature testing

Samples treated at 550 °C

- 36 % increase in strength
- 10% decrease in stiffness
- 3x increase in breaking strain

Summary

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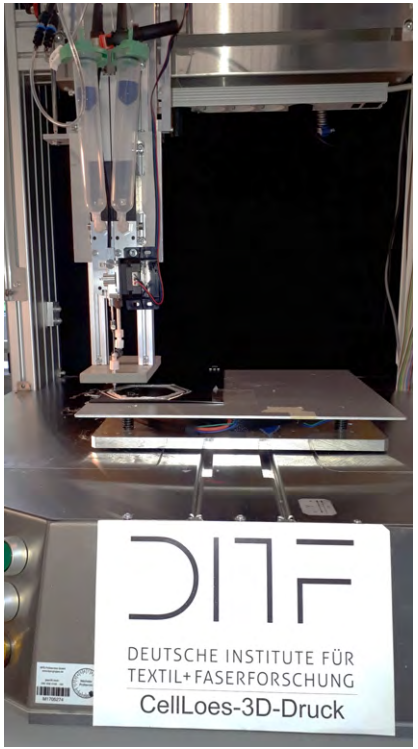
- Formulated phosphate geopolymer recipes suitable for composite manufacturing
- Water content and Molar ratio of the slurry influences both processability and mechanical properties of the cured phosphate geopolymer
- Demonstrated the pultrudability of the CBPC / phosphate geopolymer systems with basalt fiber reinforcements
- Process and material parameters influence the mechanical properties of the composite profiles

Outlook

Transferring the knowledge gained in composite manufacturing to 3D-Printing continuous fiber reinforced geopolymers

Natural fibers → alkali system

Glass & Basalt fibers → acidic system



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Contact:

Dr.-Ing. Sathis Kumar Selvarayan

Email: sathiskumar.selvarayan@ditf.de

Forschungsnetzwerk Mittelstand

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on the basis of a decision by the German Bundestag

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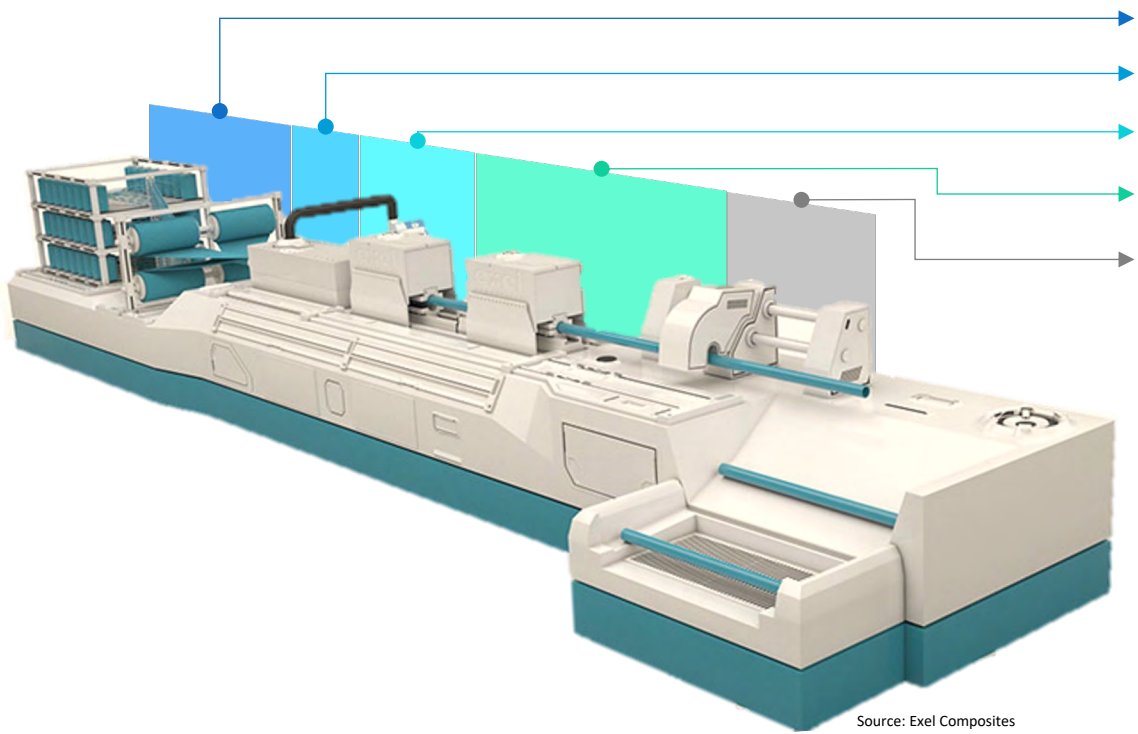
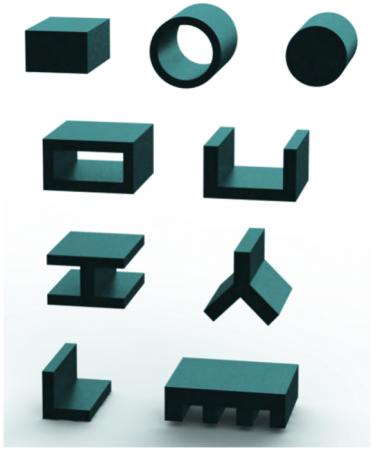
The Future is Textile

Pultrusion



Source: Exel Composites



Basalt fibers

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Thermal properties of Basalt fibers

- Melting temperature: ~1500°C
- Application temperature range: -260°C bis +600°C
- Max. temperature: +890°C

E-Glass vs Basalt:

- approx. 20% higher thermal resistance
- 15% higher mechanical properties
- Higher chemical resistance against acids and solvents
- 10 x better absorption of electro-magnetic radiation

Poor alkali resistance

Faserart	Durchmesser [µm]	Zugfestigkeit [N/mm2]	E-Modul [GPa]	Dichte [g/cm³]	Kosten [€/kg]
Stahl	10 - 200	2.200	210	7,8	1 - 100
(AR) Glas	14 - 20	1.700 - 2.000	72	2,7	2 - 8
Basalt	9 - 13	1.000 - 4.000	75 - 100	2,6 - 2,8	3 - 5
Carbon	7	bis 4.000	240 - 400	1,8	>18
Aramid	12	3.400	60 - 130	1,4	> 30
Polypropylen (PP)	18	300	3,5	0,9	3 - 5
Polyvinylalkohol (PVA)	40	1.600	42	1,3	5 - 10
Polyethylenterephthalat (PET)	8 - 200	35 - 130	4,5	1,4	k.a.
Polytetrafluorethylen (PTFE)	k.a.	170 - 310	0,6	2,1 - 2,3	k.a.

E-Modul Barometer

Source: http://www.bafanet.com/basaltfasern/

Source: www.afbw.eu