Oxide CMC Components Manufactured via PIP Processing Based on Polysiloxanes

Martin Frieß, Sandrine Denis

German Aerospace Center (DLR),
Institute of Structures and Design, Stuttgart (Germany)

ECerS XII, 12th Conference of the European Ceramic Society,
Stockholm (Sweden), June 19-23 2011

Content

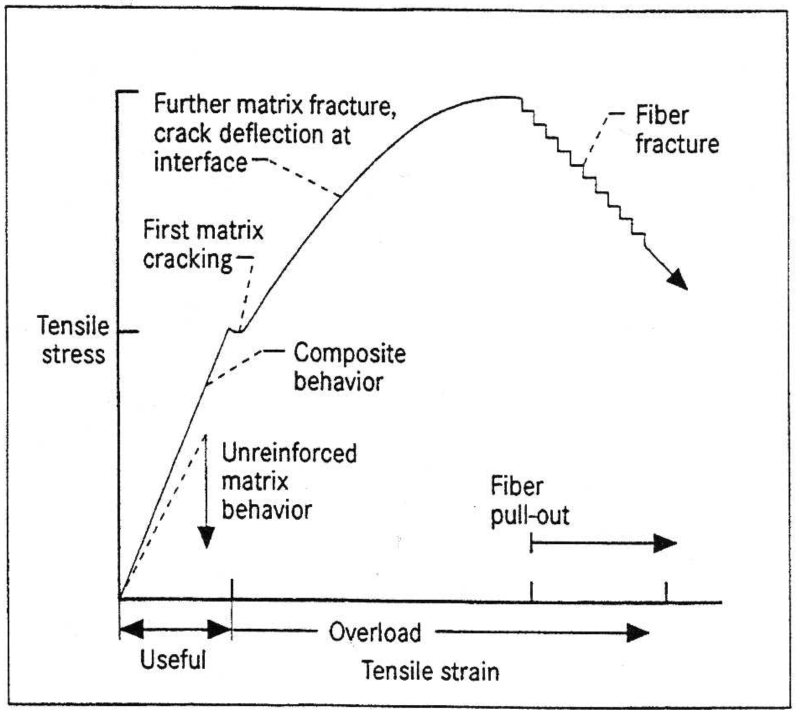
- Introduction and Motivation
- Manufacture of OXIPOL
    - Resin transfer moulding (RTM)
    - Wet filament winding
    - Warm pressing
- Properties of OXIPOL
- Summary and outlook

Why Ceramic Matrix Composites (CMC)?

- Design of fibre/matrix interface
- Composite properties defined by fibre/matrix interface
  - Pseudo-ductile fracture behaviour
  - Weak bond between fibre and matrix
  - Formation of energy absorbing mechanisms
  - Dissipation of tension in fibre/matrix interface

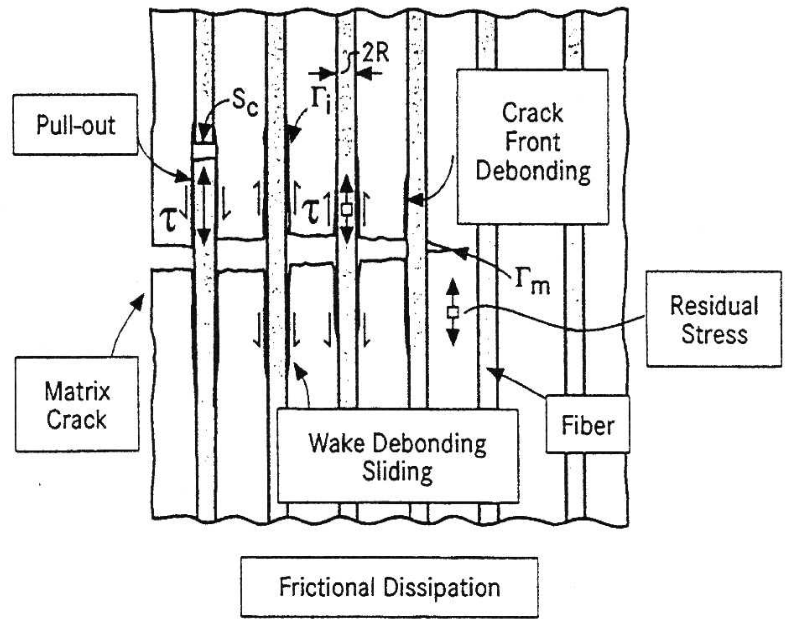
Ceramic Matrix Composites

- Increase in fracture toughness



Ideal stress strain behavior

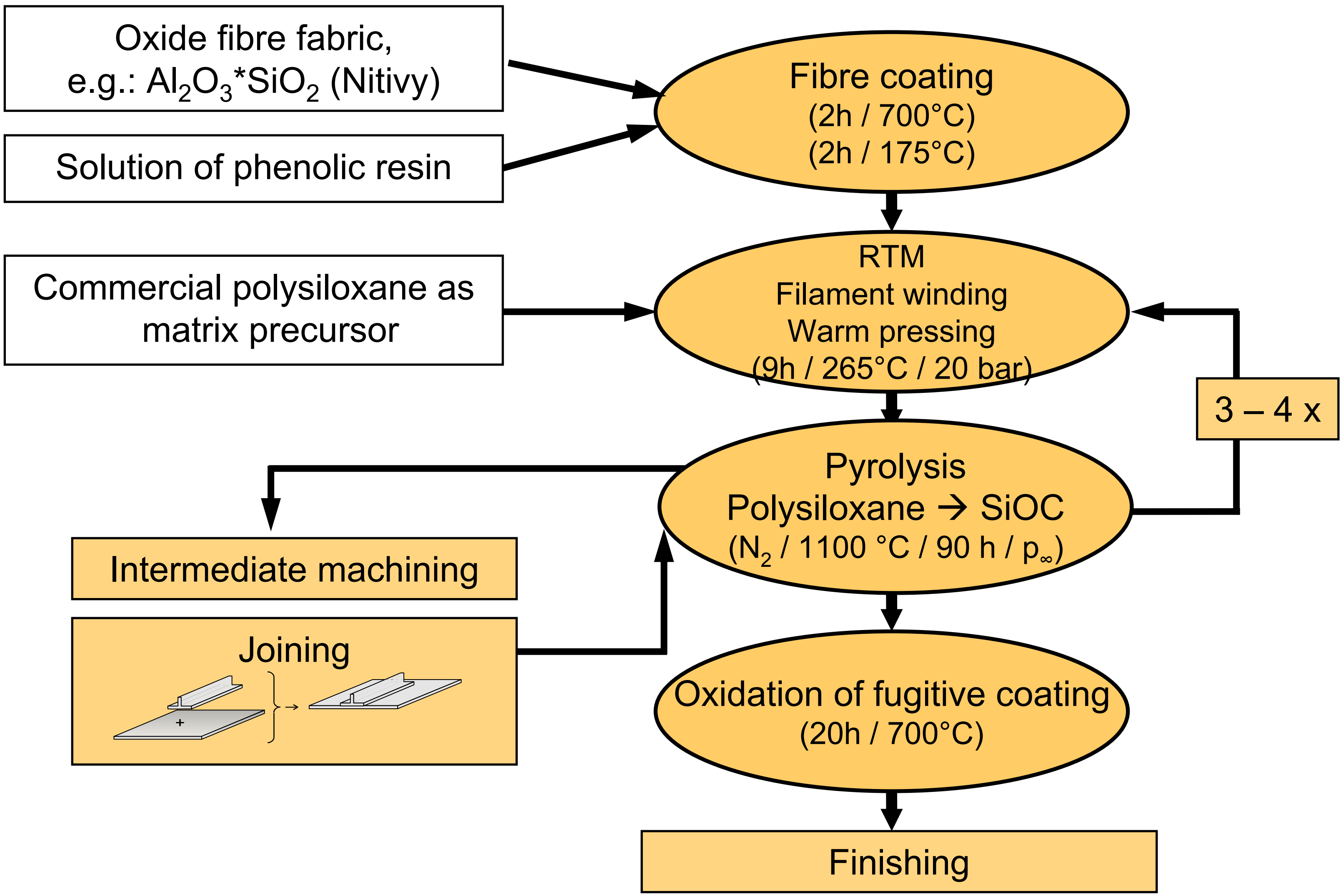
Reference: DiCarlo and Dutta (1995)



Fracture mechanisms

Reference: Zok, Evans and Mackin (1995)

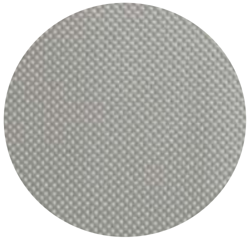
Manufacture of OXIPOL Using LPI-Processing



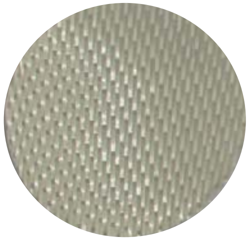
Fabrics Variation

Manufacturer's data

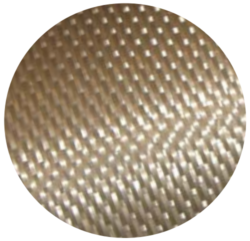
Ref.	Fibres	Fabrics	Manufacturer	Weave	Mass per unit area [g/m²]	Filament tensile strength [MPa]
Nitivy	Nitivy 72/28	2626P	Nitivy Co. LTD	Plain	280	1800
N610	Nextel 610	DF19	3M	8 harness satin	654	3100
N720	Nextel 720	XN625	3M	8 harness satin	637	2100



Nitivy



N610



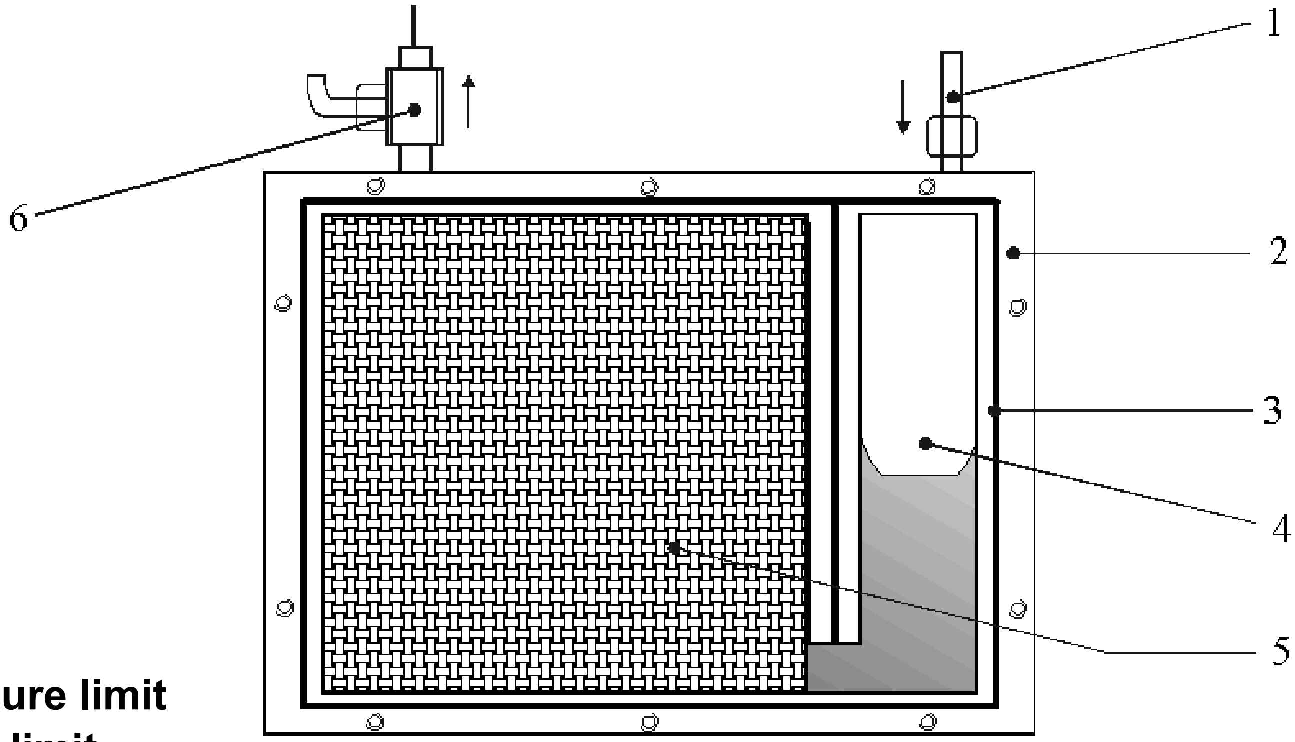
N720

Coating of Fabrics with Phenolic Resin via Foulard

Properties of applied Polysiloxanes

Polysiloxane		MSE 100	MK	MSE 100 + 50% MK
Empirical formula		Methoxymethyl polysiloxane	Methyl polysiloxane	-
Density after curing	[g/cm³]	1.14	0.6 (powder)	1.14
Density after pyrolysis (SiOC) [g/cm³]		-	-	2,3
Viskosity at 25 °C	[mPas]	30	solid	solid
Viskosity at 120 °C	[mPas]	30	> 2000	< 100
Curing typ		Polycondensation	Polycondensation	Polycondensation
ceramic yield at 1100 °C [%]		15	82	80
Volume shrinkage at 1100 °C	[%]	93	60	60
Cost	[€/kg]	18	23	21

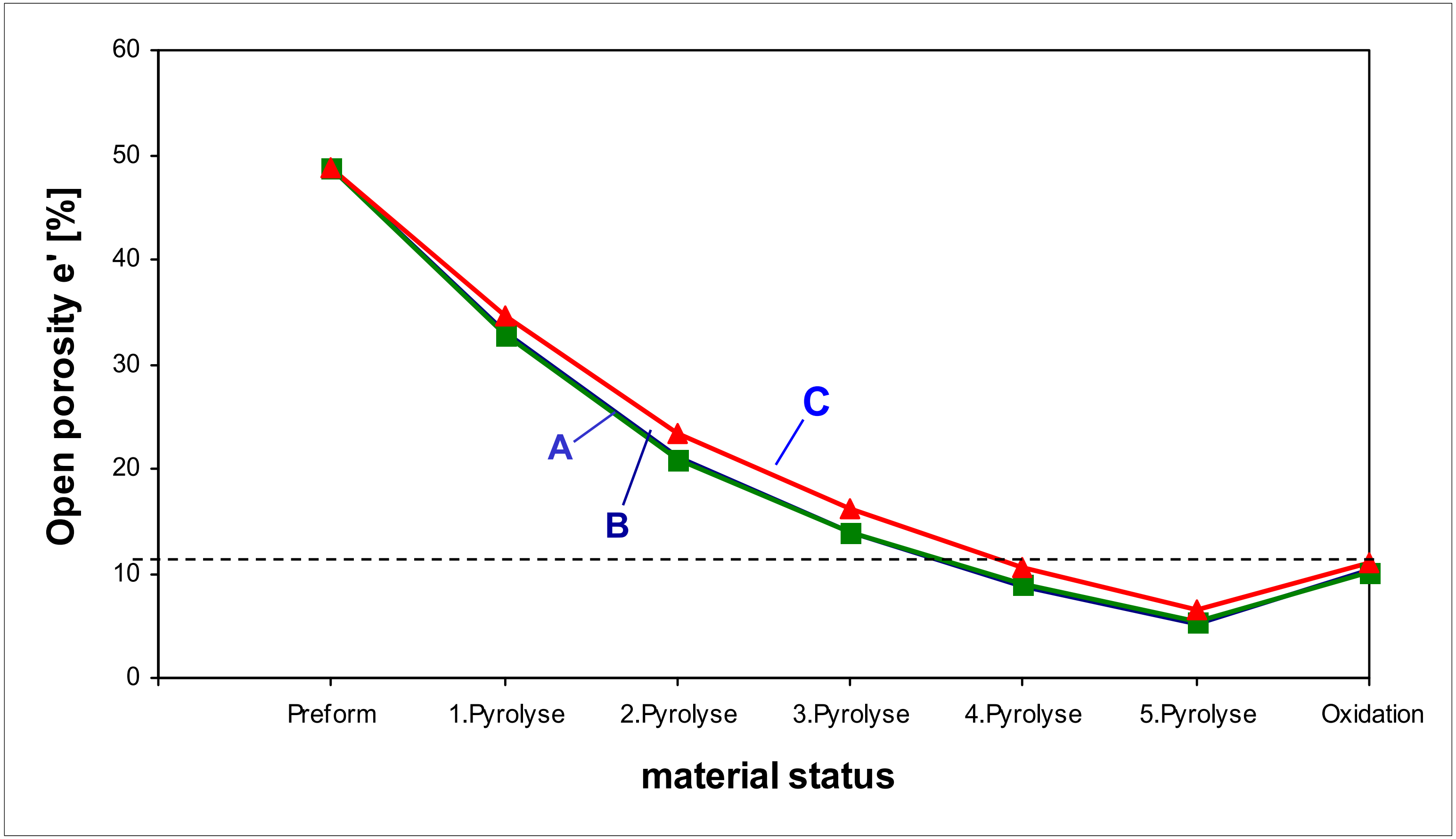
Resin Transfer Moulding (RTM) I



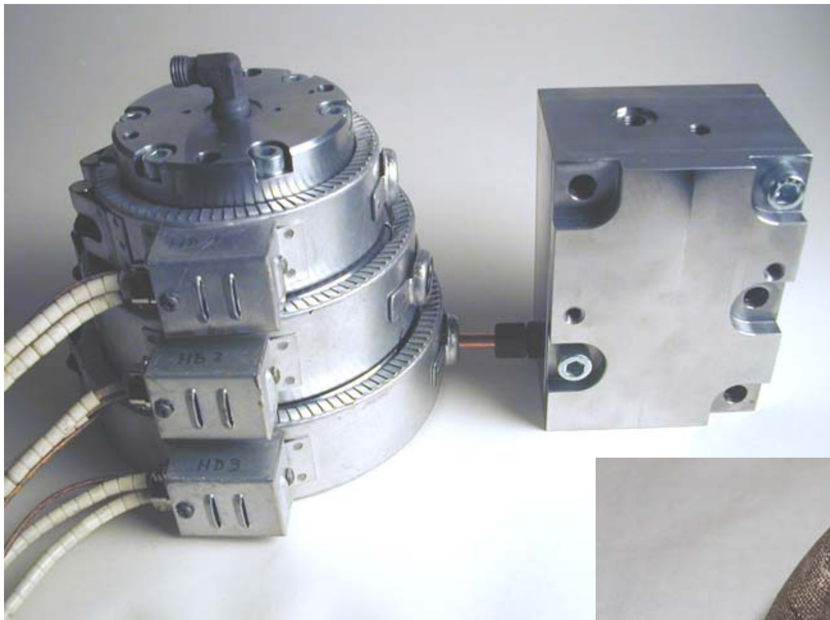
1 Pressure Inlet
2 Mould
3 Seal
4 Resin chamber
5 Fibre Structure
6 Vacuum pump

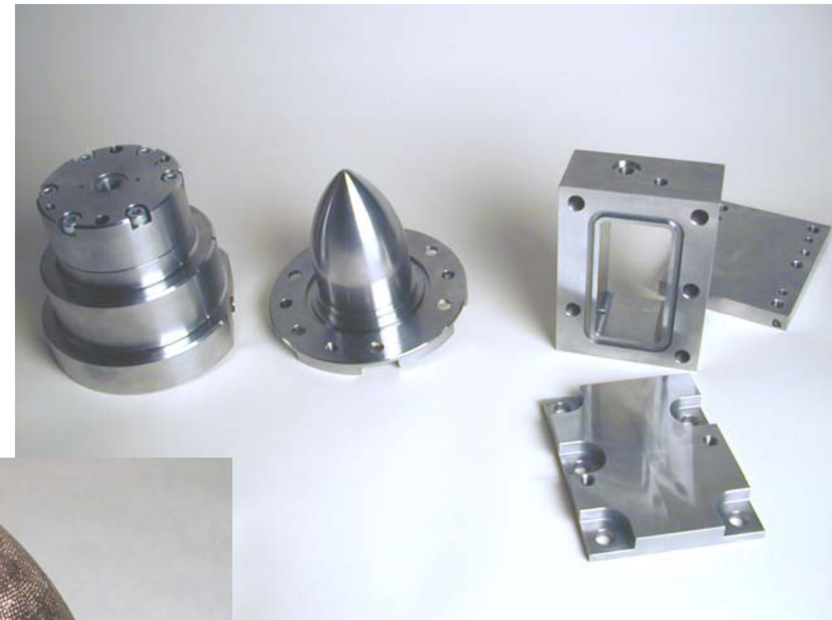
- **250°C Temperature limit**
- **20 bar Pressure limit**
- **Possibility of processing precursors with cross-linking via condensation**
- **Ability of exact adjustment of fibre fraction**
- **Complete infiltration of preform volume**
- **Ability of processing in inert atmosphere**

Open Porosity of OXIPOL versus PIP cycle

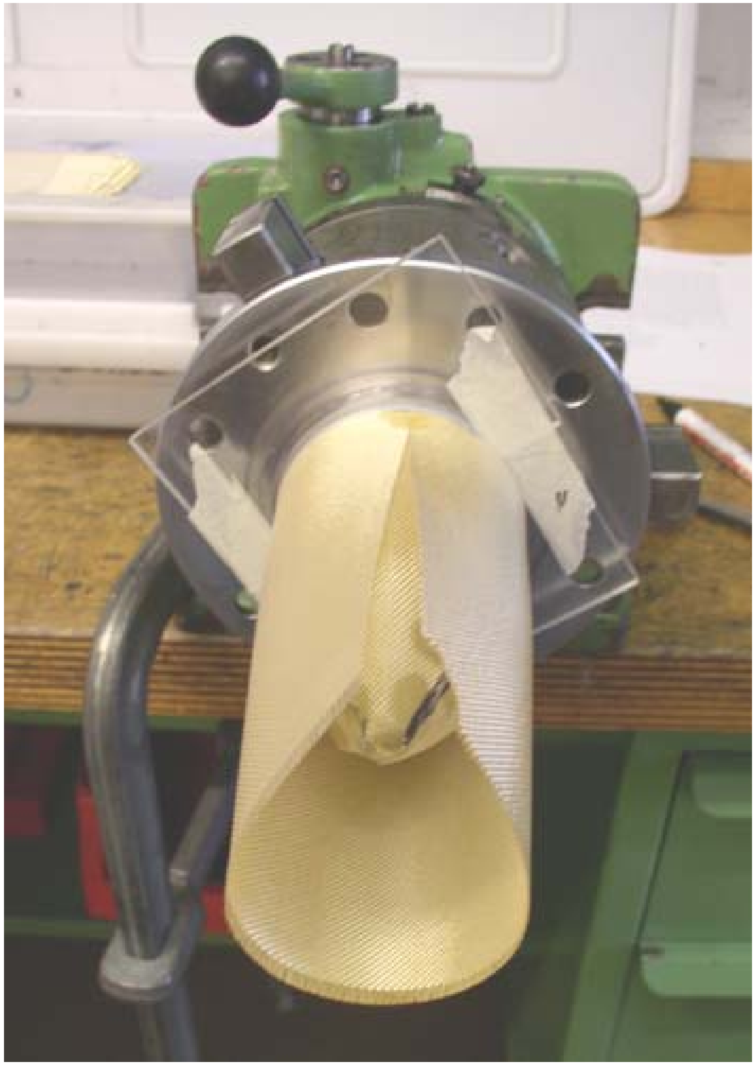


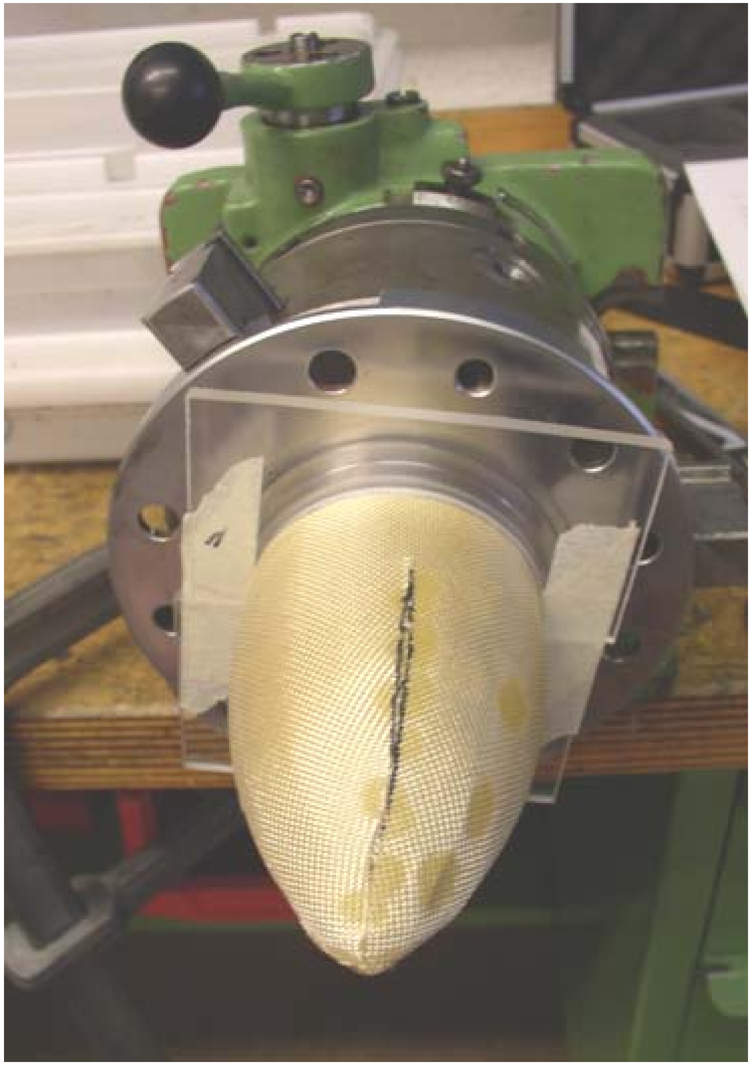
RTM-mould for OXIPOL Manufacture



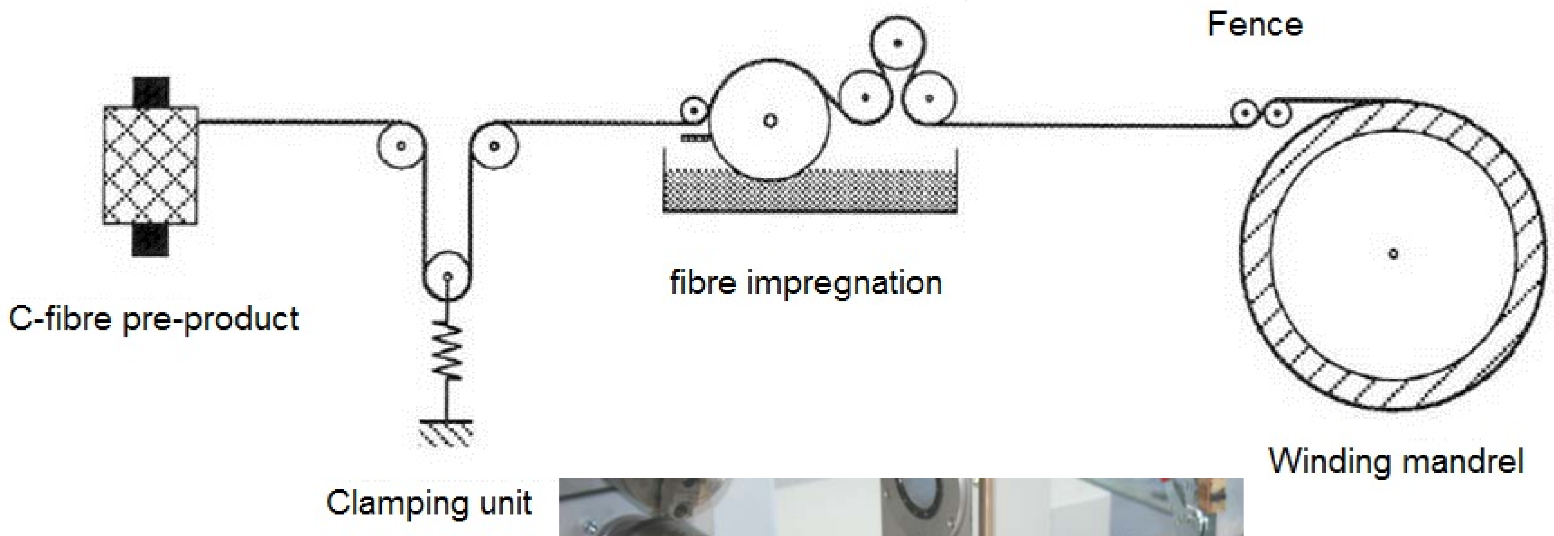


Lay-up of Oxide Fabrics into RTM-mould





Manufacture of Radomes via Filament Winding and PIP Processing I: Filament Winding



Manufacture of Radomes via Filament Winding and PIP Processing II: Reinfiltration in Resin Bath

Investigated PIP Process via Warm Pressing

Fibre coating
Variation of oxide fabrics
Variation of phenolic resin

↓

Polymerisation
Powdery polysiloxane precursor

↓

Pyrolysis
Polysiloxane → SiOC

↓

Reinfiltration
Wet polysiloxane precursor bath

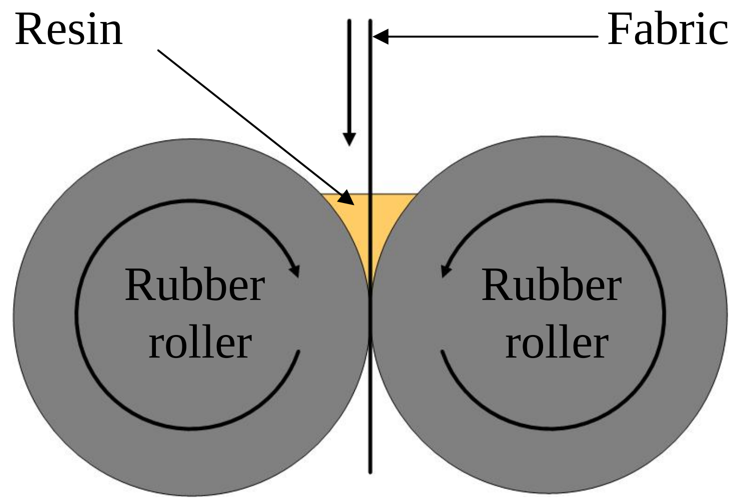
↓

Pyrolysis
Polysiloxane → SiOC

(Reinfiltration and Pyrolysis repeated 3x)

↓

Oxidation



Fabrics coating on a Foulard

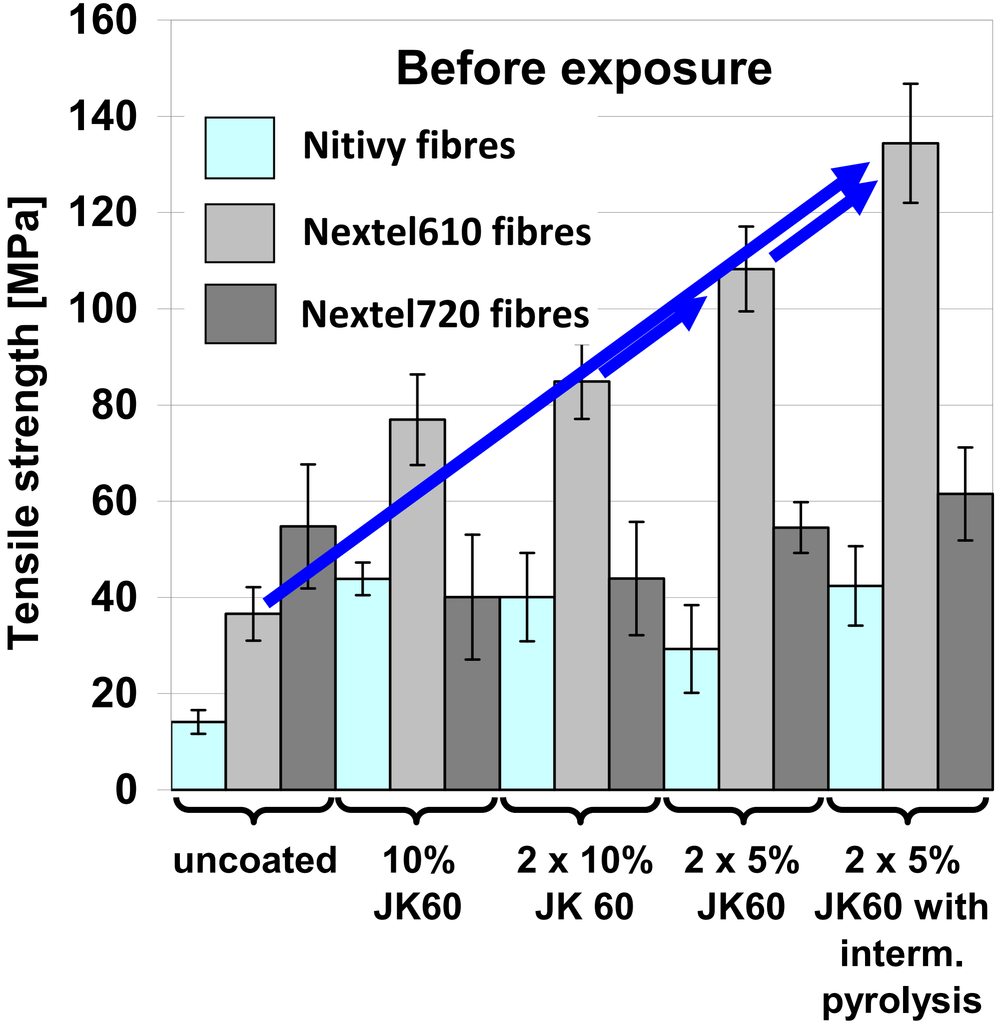
Variation of the Fugitive Coating

Type	A *	B	C	D	E
Phenolic resin content JK60 [mass-%]	0	10	10	5	5
Coating cycles	0	1	2	2	2
Pyrolysis cycles	0	0	0	0	1

* Configuration N720-A is desized

⇒ OXIPOL variation on 15 sample plates

Tensile Tests: Strengths before Exposure

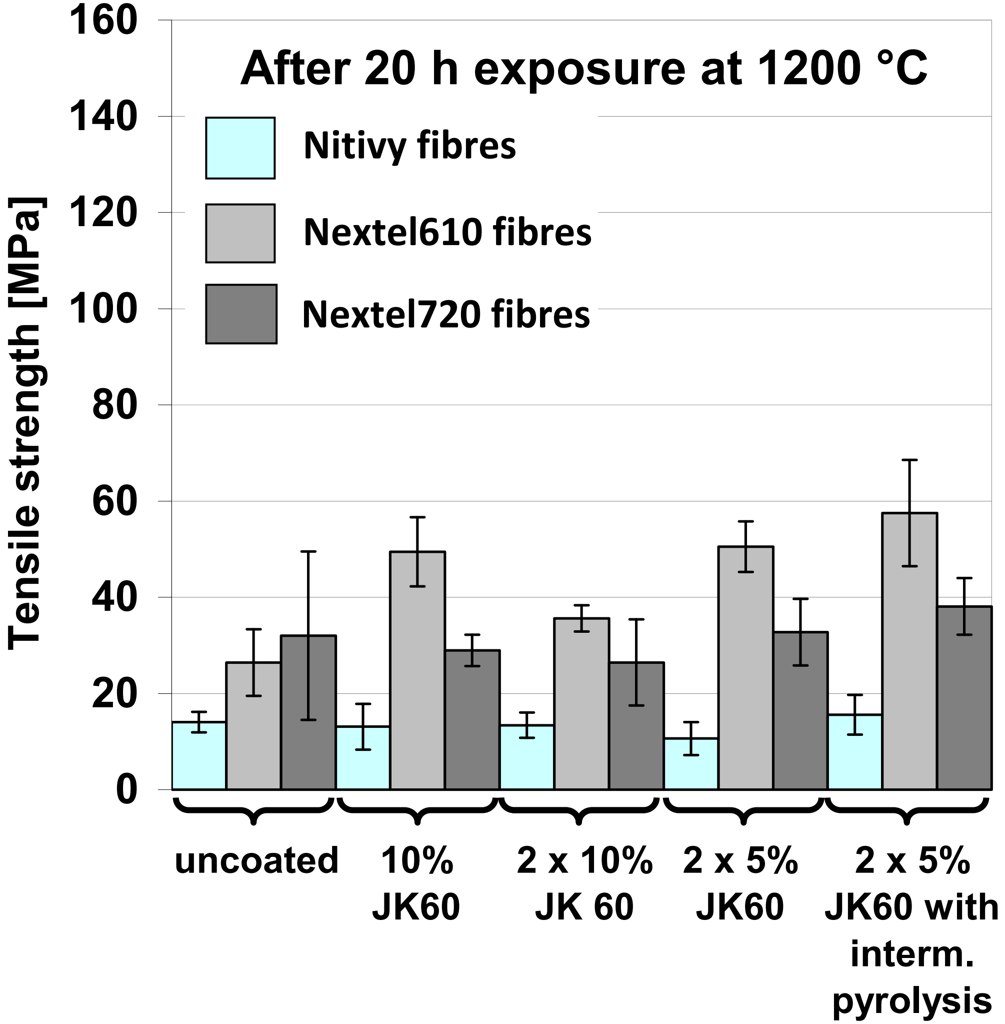


- Coating of N610 increases tensile strength:

$\sigma_{max\ 2\ x\ 5\ \%JK60\ int.\ pyr.} = 3.6 * \sigma_{max\ uncoated}$

- Tensile strength improvement with:
  - Several coating with lower resin concentration
  - Intermediate pyrolysis of the coated fabrics

Tensile Tests: Strengths after Exposure



- Independently of coating:

  Tensile strength decreases after exposure

- These coatings were not adapted for 1200°C

Oxidation Phenomena during Thermal Exposure

- Matrix growing after exposure due to two reactions:
  - Net weight loss

    $C + O_2 \rightarrow CO_x$

  - Net weight gain

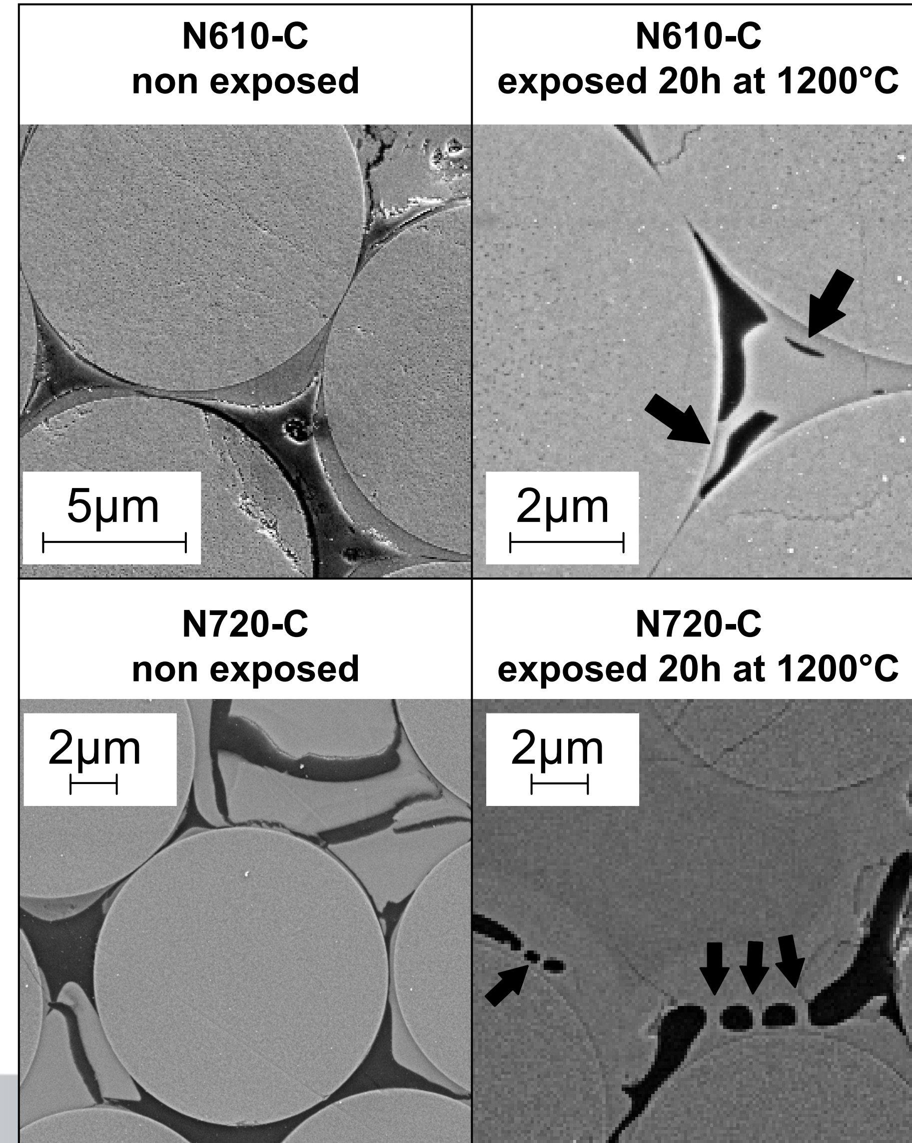
    $Si\text{-}C + O_2 \rightarrow Si\text{-}O + CO_x$

⇒ Close the gap fibres/matrix

⇒ Decrease of energy absorbing effects and tensile strength

⇒ Need for oxidation resistant fibre coating

⇒ For example $LaPO_4$



Summary

- **The manufacture of OXIPOL can be performed by different methods and opens up new application areas**
- **Resin transfer moulding (RTM) is well suited for resins cured via polycondensation and is very efficient for densification of CMC**
- **Filament winding of oxide fibres was successfully applied to manufacture complex structures**
- **OXIPOL manufacture based on warm pressing provides high potential for cost reduction**
- **Fugitive coating is not applicable at high temperature in air due to embrittlement of CMC (matrix degradation and gap closure)**

→ new oxidation resistant coatings providing easy cleavage are needed, e.g. $LaPO_4$