Joining and Coating of SiC/SiC Composites

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Methods of Joining SiC/SiC to SiC/SiC

Methods of ‘low activation’ joining of SiC/SiC to SiC/SiC:

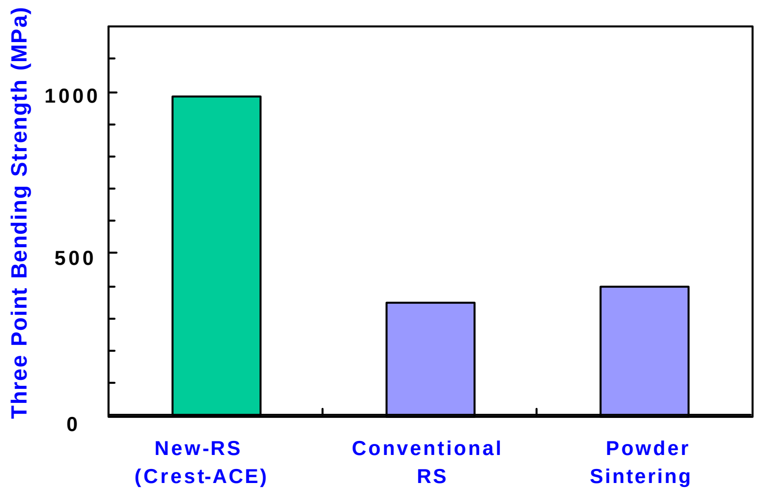
- Reaction bonding
  - **Complex slurry approach**
  - ARCjoinT by M. Singh
  - Etc.
- Preceramic polymer adhesives
  - Conventional PCS solvent
  - **Preceramic polymers for improved strength, e.g., PVS**
  - **Preceramic polymers designed for stoichiometric SiC**
- Glass-ceramics adhesives
  - **CA(calcia-alumina) for joining and coating**
- Mechanical fasteners
  - **SiC/glass-ceramics fasteners**
  - SiC/SiC fasteners

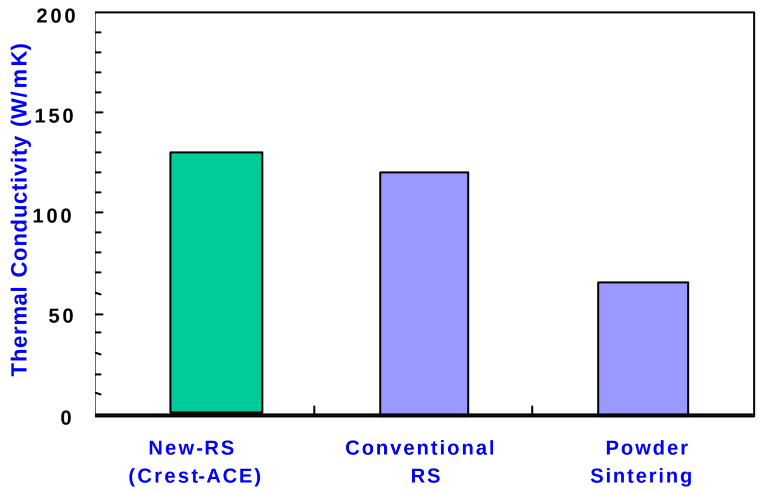
Reaction Bonding by Complex Slurry Approach

- **Reaction bonding – a promising technique for permanent joining.**
  - Neutron irradiation effects need to be studied.
- **C-SiC slurry + molten Si infiltration**
  - Straight-forward application of the monolithic SiC production process via 'reaction-sintering' developed by Toshiba and optimized in CREST-ACE.
  - Good wettability with SiC.
  - Mechanical properties are very promising.
  - Very good thermal conductivity.
- **C-Si-SiC slurry without melt-infiltration**
  - Application of the matrix SiC forming process via reaction-bonding being developed at Kyushu Industrial Research Institute.
  - Good wettability with SiC.
  - Mechanical property is not comparable with melt-infiltration products due of significant porosity.
  - Fairly good thermal conductivity.

Reaction Bonding by Complex Slurry Approach

CREST-ACE / Kyoto University / Toshiba





- **‘C-SiC slurry + melt infiltration’ process for monolithic SiC production has greatly been improved through the optimization of process conditions.**

Newly Developed Preceramic Polymers

- **Preceramic polymer: joining with intermediate-to-high strength.**
  - Initial products are in amorphous phases.
  - Neutron exposure crystallizes the polymer-derived SiC rather quickly at elevated temperatures.
  - Neutron irradiation effects need to be studied.
  - Joining strength will be limited by porosity. Residual porosity are controlled by particulate loading.
- **PVS (polyvinylsilane) for improved strength and handling ease.**
  - Inert and low viscosity liquid at an ambient temperature.
  - Improved mechanical property cf. PCS-derived ceramics.
- **New polymer precursor designed for stoichiometric composition.**
  - Under development in CREST-ACE.
  - Product will be nearly-single phase SiC.
  - Irradiation-induced strength degradation in excess carbon will not affect the joining integrity.
- **New polymer precursor designed for improved thermal conductivity.**
  - Under development in CREST-ACE.

Polyvinylsilane as Preceramic Polymer

- One-dimensional chain of the unit structures (a) and (b) at a molecular ratio of 1:1.

(a) $- SiH_2 - CH_2 - CH_2 -$

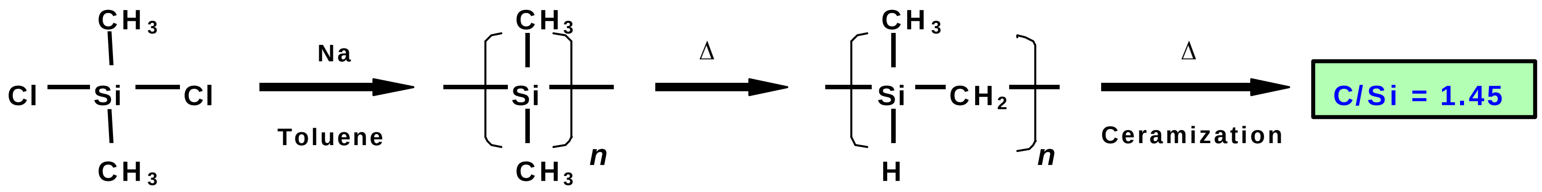
(b) $- CH - CH_2 -$ (with SiH_3 attached to CH)

- Chemically stable and low viscosity liquid in air at an ambient temperature. Ease of handling and particulate loading.
- Enhanced cross-linking by the presence of abundant Si-H bonds.
- Enhanced densification is achievable by pressurizing in a gel-like state.
- The strength of SiC/PVS-SiC composites after single infiltration is comparable with the saturated strength of multiple-infiltrated SiC/PCS-SiC composites.

Polymer Precursor for Stoichiometric Composition

Conventional Polymer for PCS

C/Si = 2.0

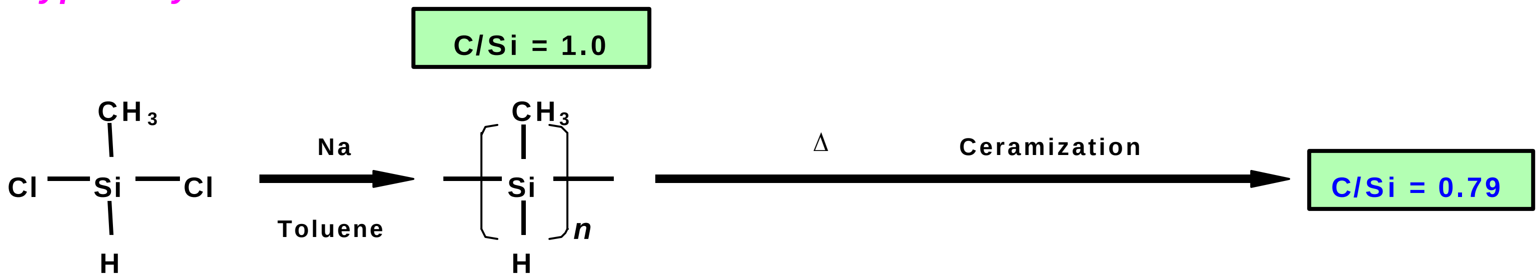


Dimethyldichlorosilane

Polydimethylsilane (PDMS)

Polycarbosilane (PCS)

New Type Polymer for PMS



Methyldichlorosilane

Polymethylsilane (PMS)

Polymer Design

Copolymer (PCS + PMS) —— Δ Ceramization ——→ **C/Si = 1.0**

CREST-ACE / Kyoto University / Ube Industries

Glass-Ceramic Joining

- **Glass-ceramic adhesives : Suit for quick and non-permanent uses. Applicability to permanent uses is up to future development.**
  - Fair strength has been achieved via a pressure-less process.
  - Neutron irradiation effects need to be studied.
- **CA (calcia-alumina) glass-ceramics joining.**
  - Fair strength has been achieved via a pressure-less process.
  - Joining strength can be tailored by a composition control.
  - Excellent wettability with CVI-SiC matrix.
  - Ease of decomposing at above softening temperature.

Properties of CA-Joint

M.Ferraris, et al.

Thermal characteristic

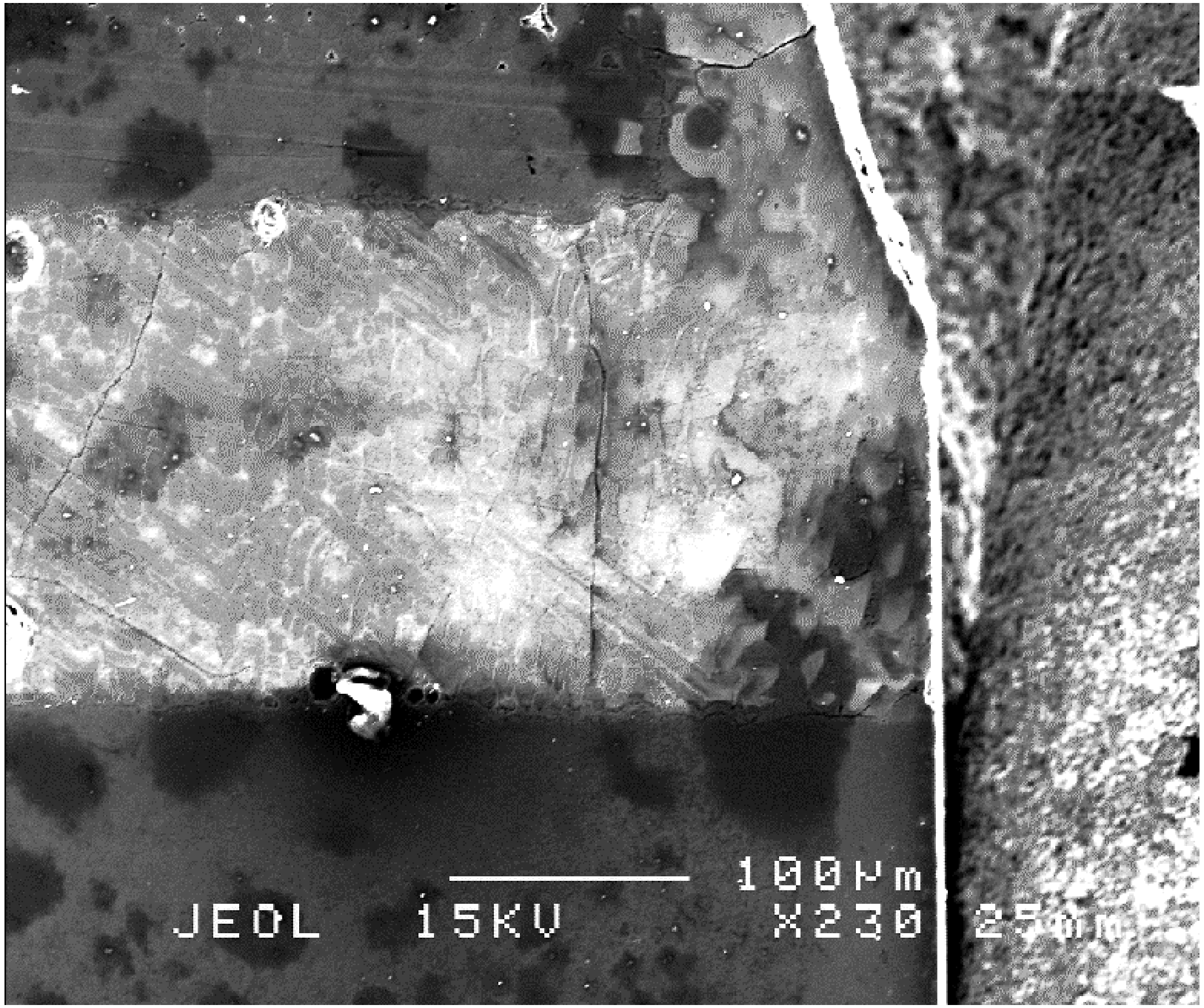
Grass transition Temperature	Glass softening point	Thermal expansion coefficient 25°C	Thermal expansion coefficient 350°C
850°C	1380°C	7.43 $\times 10^{-6}$ K^{-1}	9.42 $\times 10^{-6}$ K^{-1}

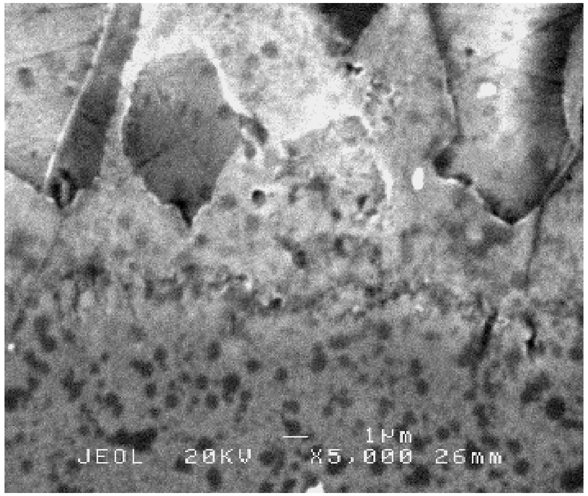
Mechanical property

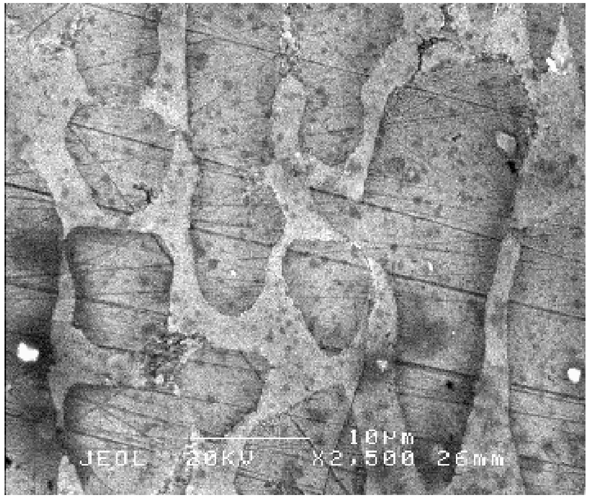
Shear strength (Room temperature)
28 MPa

- Results from the first batch of CA are shown.
- The thermal properties are tailorable via compositional adjustment.
- Shear strength can be improved by a pressurized processing.

SEM Micrographs of CA-joined SiC/SiC





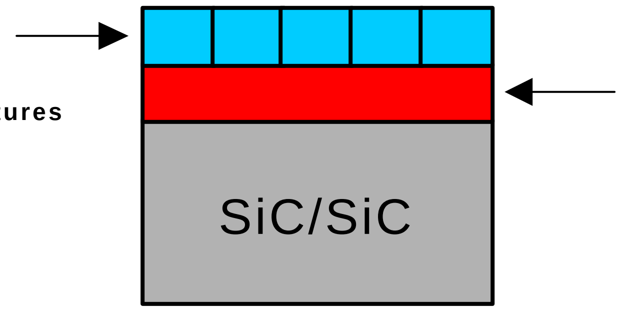


- Excellent wetting of CA with SiC is demonstrated.
- Elongated crystallite islands are distributed in the glass matrix.

Glass-Ceramics as Coating Materials

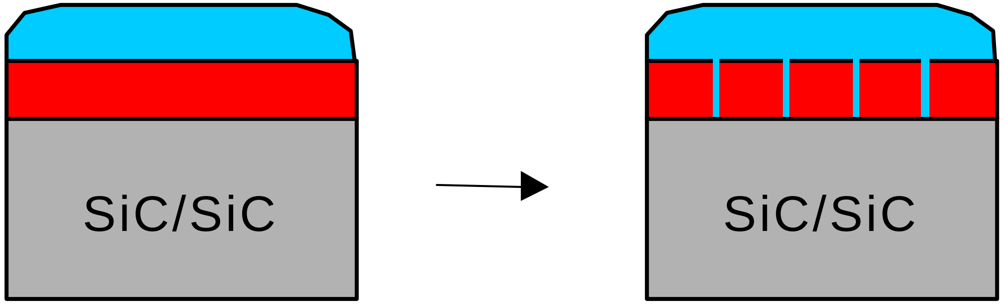
Self healing hermetic sealing by glass / glass-ceramics double-layered coating (Ferraris, et al.)

Second layer:
Glass
for self-sealing at high temperatures



First layer:
Grass-ceramic
for low temperature sealing
(CTE conformed to SiC/SiC)

T < T_{soften}



First layer cracking to be allowed

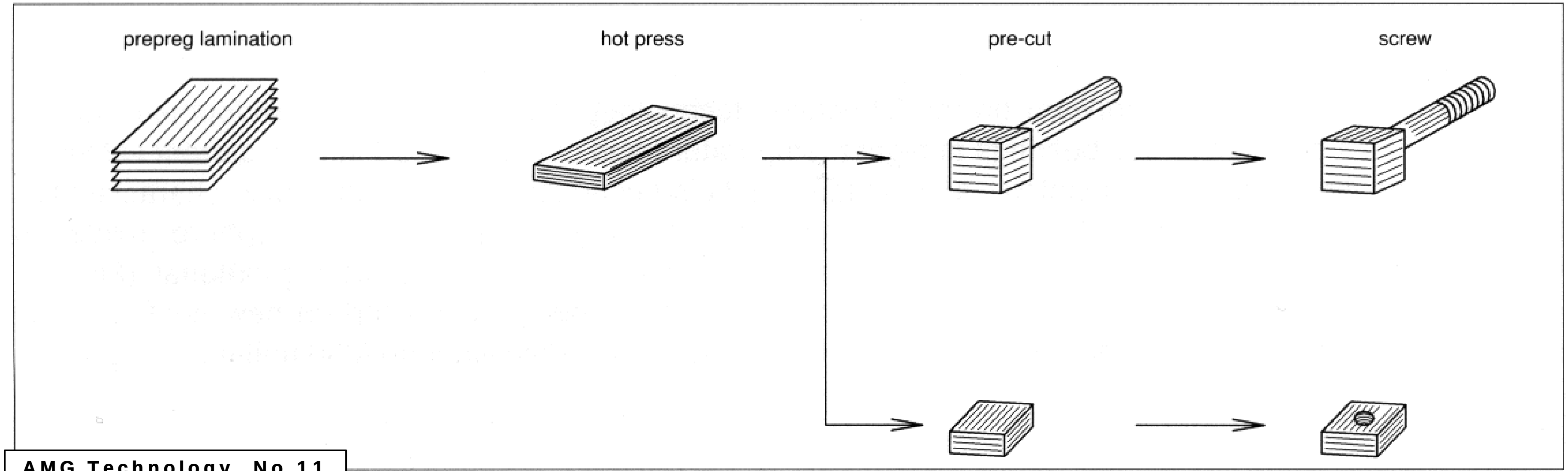
T > T_{soften}

- Experiment has been initiated at Politecnico di Torino.

Mechanical Joining of SiC/SiC

- **Tyranno™ / glass-ceramics fasteners**
  - Trial-manufacturing was carried out in the Advanced Material Gas Generator (AMG) program for high efficiency and reduced-NOx gas turbines.
  - The initial material was Tyranno / BMAS composite.
  - The manufactured CMC fasteners can be used at temperatures below 1523K.
  - The fastener size tested was 10mm screw diameter (M10 x P1.5 and E10 x P1.8).
  - The fasteners exhibited sufficient tensile fastening strength in air at elevated temperatures.

Fabrication of CMC Fasteners



AMG Technology, No.11

Fig.4 Manufacturing Flow of CMC Fastener

Glass-ceramics for CMC Matrix

Matrix type	Major constituents	Minor constituents	Major crystalline phase	Maximum use temp.(℃) (in composite form)
LAS- I	LiO_2 Al_2O_3, MgO, SiO_2	ZnO,ZrO_2,BaO	β-Spodumene	1000
LAS- II	LiO_2, Al_2O_3, MgO, SiO_2, Nb_2O_5	ZnO,ZrO_2,BaO	β-Spodumene	1100
LAS-III	LiO_2, Al_2O_3, MgO, SiO_2, Nb_2O_5	ZrO_2	β-Spodumene	1200
MAS	MgO, Al_2O_3, SiO_2	BaO	Cordieraite	1200
BMAS	BaO, MgO, Al_2O_3, SiO_2	—	Barium osumilite	1250
Ternary mullite	BaO, Al_2O_3, SiO_2	—	Mullite	~1500
Hexacelsian	BaO, Al_2O_3, SiO_2	—	Hexacelsian	~1700
Monoclinic celsian	SrO, Al_2O_3, SiO_2	—	Monoclinic celsian	~1700

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Manufacturing Process of CMC Fasteners

U/D-CMC Core (*f* 4)

↓

U/D-Prepreg Sheet Lay-up
$0 < q < 60°$: 24-layers/side
$q = 60°$: 18-layers/side

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Preforming

↓

Hot-pressing

↓

Heat treatment

↓

Machining

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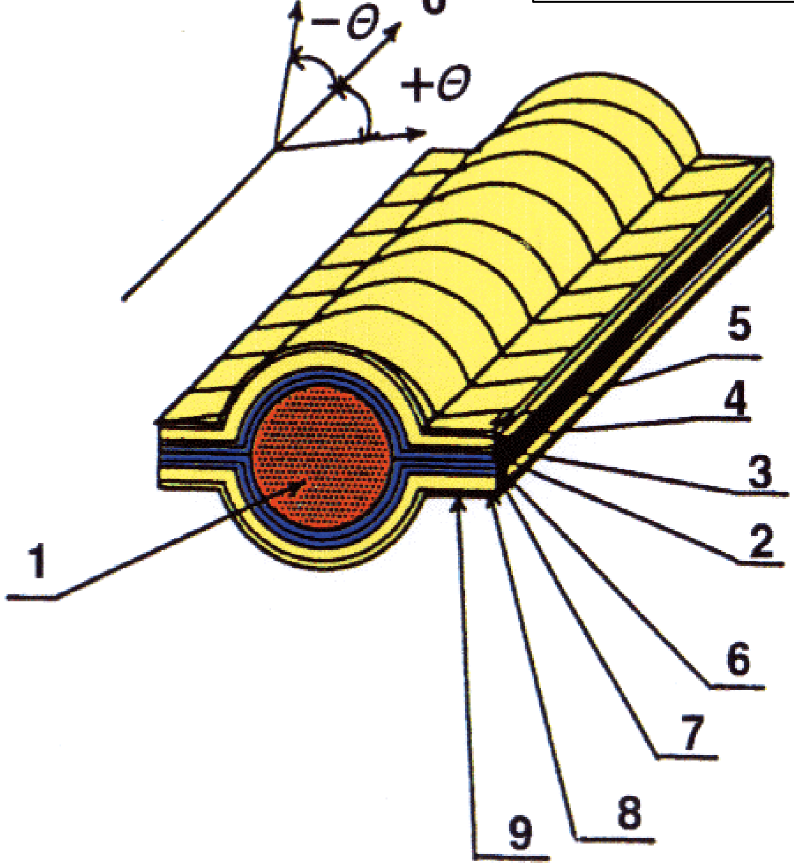

Samples of Tyranno / BMAS Fasteners

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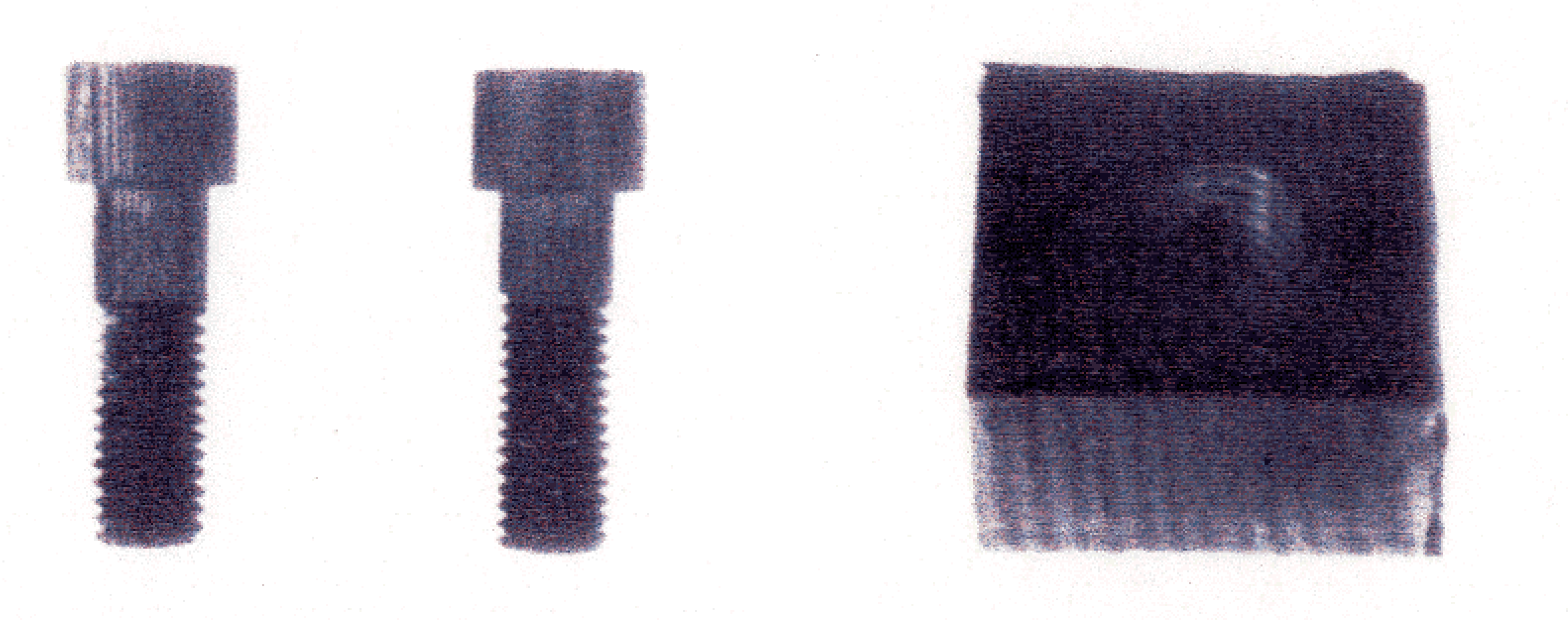
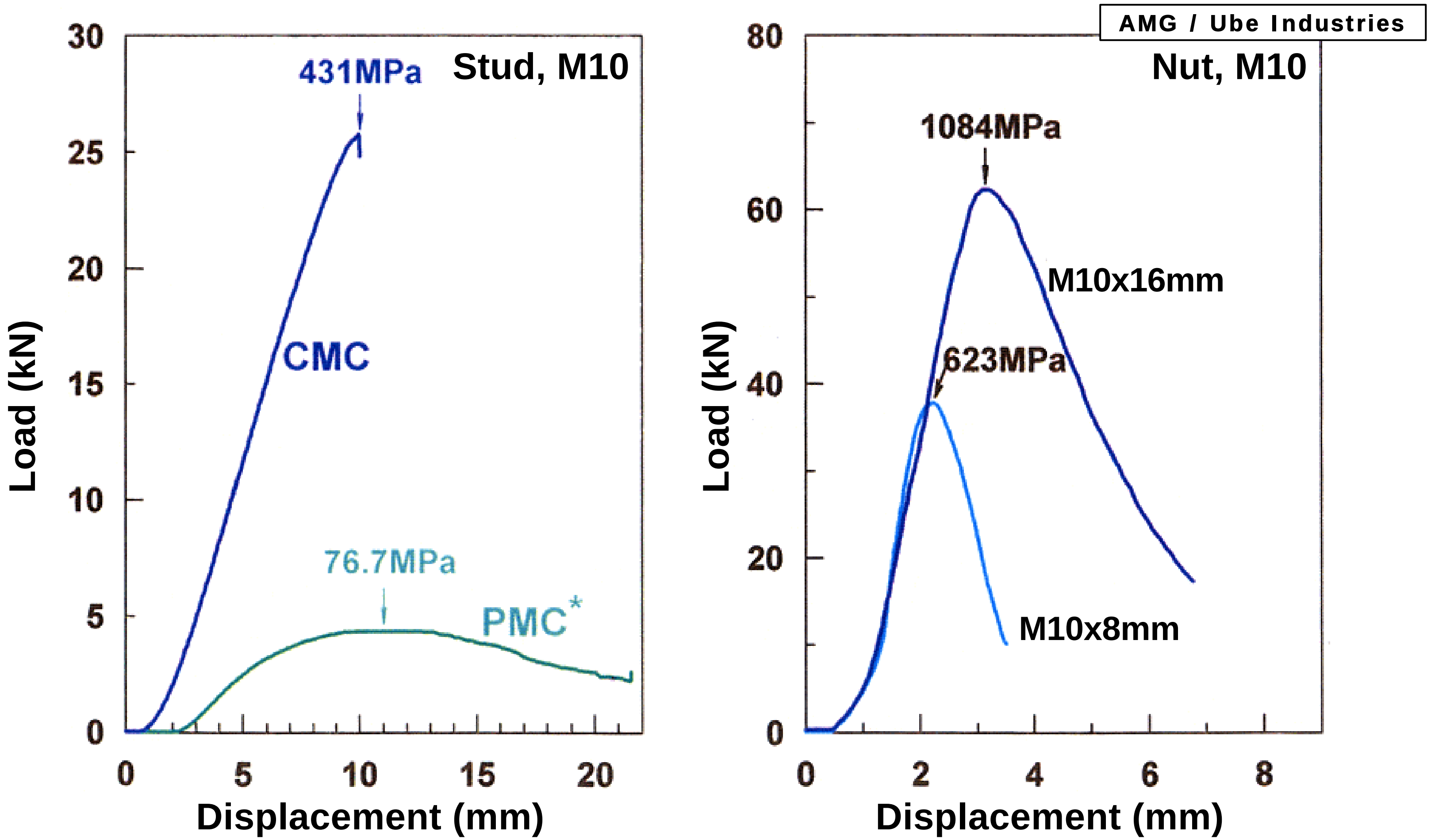


Tensile Strength of Tyranno/BMAS Fasteners



*C/Polyamide composite

Summary

- **Joining SiC/SiC to SiC/SiC is may not a problem but an important technical issue for fusion structural applications.**
- **Several methods of joining have been proposed and studied.**
  - Reaction-bonding is an most established and promising permanent joining method for certain conditions of use.
  - Polymer adhesives is another permanent joining method. Technologies being developed for advanced PIP-SiC/SiC fabrication will be applied to improved polymer-derived joints.
  - Glass-ceramics are attractive as non-permanent joining and hermetic seal coating. Long-term stability must be assessed to be used as a permanent joining.
  - CMC mechanical fastener is another attractive approach. CMC fasteners with very good high temperature tensile strength have been developed in an advanced gas-turbine program.
- **Neutron effects on integrity of these joinings need to be studied.**