ICFRM-18
Aomori 2017

EU program update

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EU program update

- POLITO:
  - Pressure-less joining materials:
    - glass-ceramics,
    - Refractory Metal (RM)-wrap
    - SPS joining in collaboration with QMUL & Nanoforce, UK
  - Modelling and testing of joined SiC by torsion and other tests
  - Ion irradiation of joints
  - W/Fe functionally graded joints by sputtering (poster)
  - *EU project funded (Il Trovatore)(October 2017)*
  - *Autoclave tests on joined-coated materials*
  - *J-TECH@POLITO funded (May 2017)*
- CEIT:
  - porous SiC development for application as FCIs in DCLL blankets

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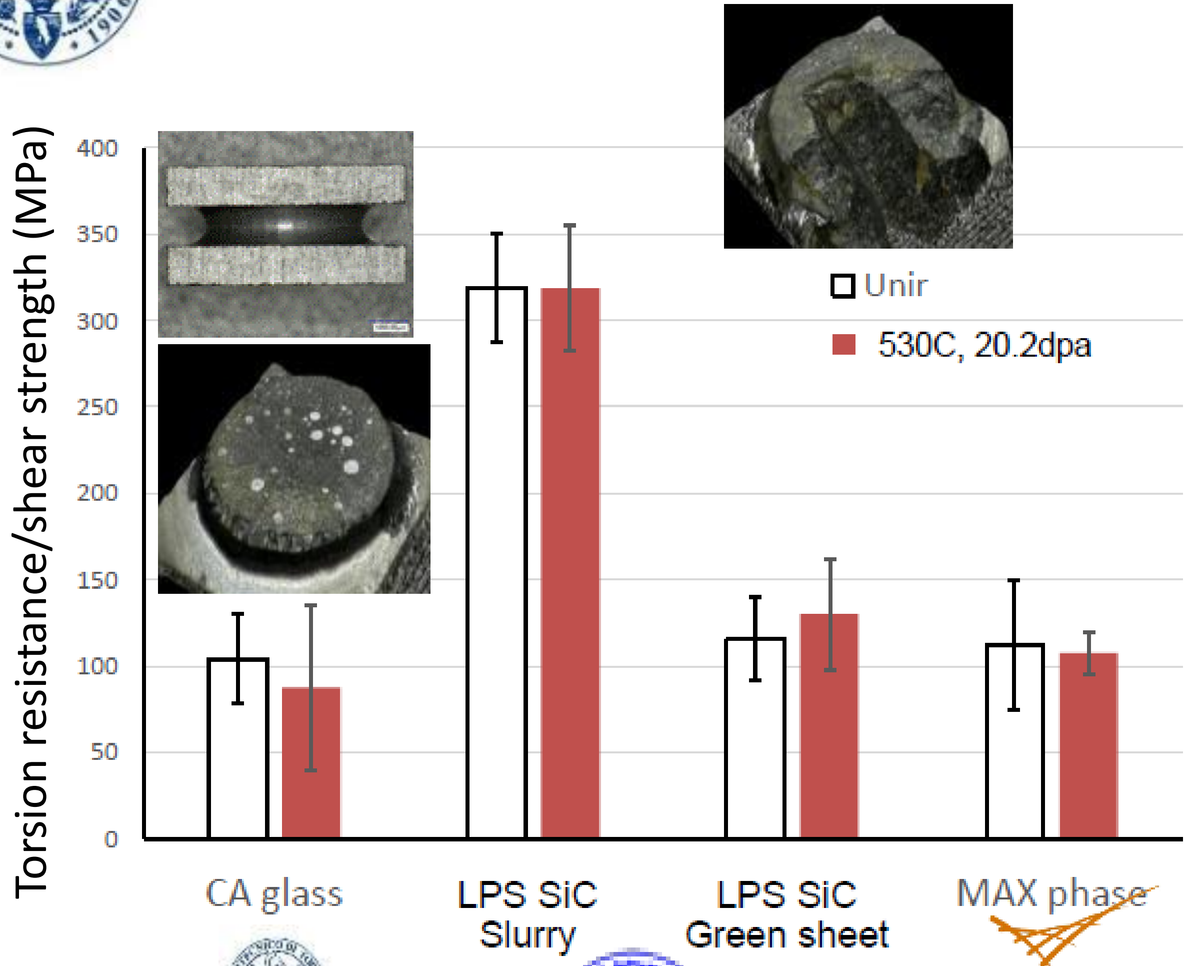
Pressure-less joining materials: glass-ceramics



- These torsion tests are suitable to measure the shear strength of a joined component only if the joining material is brittle *and if the fracture propagates exclusively in the joined area*.

- In this ideal situation, the measured shear strength is geometry independent

- In the case of fracture outside the joined area the measured value is *the torsion resistance of the joined sample*, dependent from the substrate mechanical properties

M. Ferraris - Politecnico di Torino - Italy
Department of Applied Science and Technology

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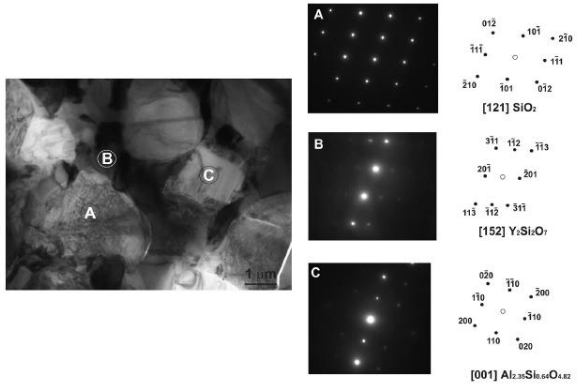
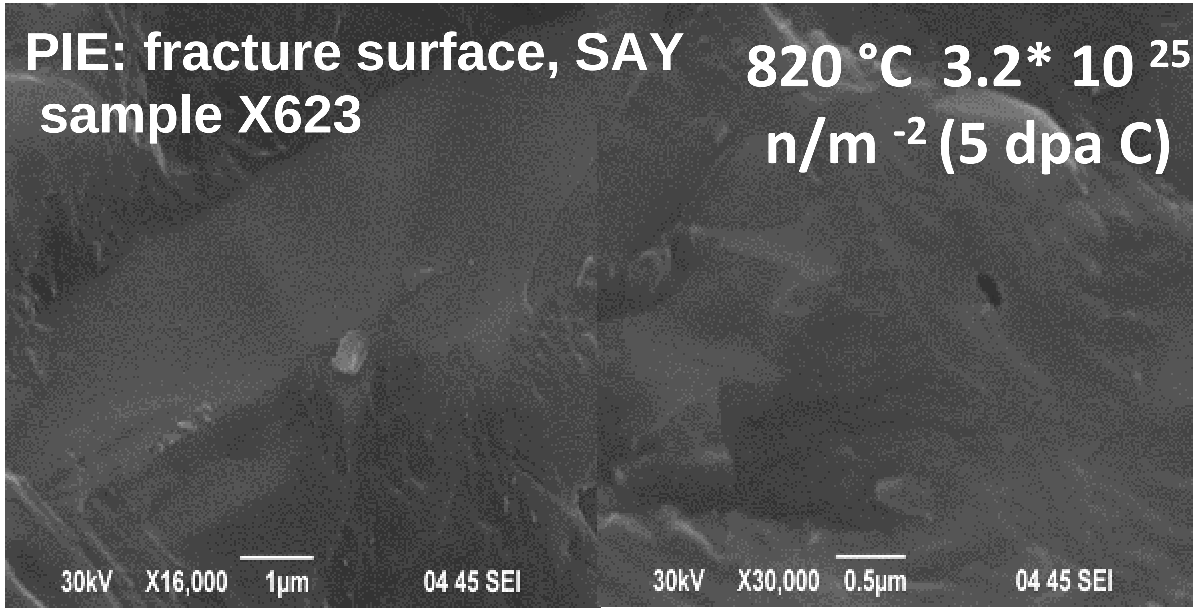
Glass-Ceramic Joining (SAY) of SiC/SiC

Glass ceramic	composition	Tg (°C) parent glass	CTE parent glass (10^{-6})	Joining process (°C) Pressure-less	CTE glass-ceramic (10^{-6})	T soft Glass-ceramic	Crystalline phases (glass-ceramic)
SAY	SiO_2 Al_2O_3 Y_2O_3	910	3.8	1375 1235 (crystallization)	5.5	975	Mullite, cristobalite, keivyite



122 ± 10 MPa bending strength *before*

89±17 MPa bending strength *after irradiation* at 820 °C 3.2* 10 ^{25} n/m ^{2} (E>1MeV , 5 dpa C)



Glass-Ceramic Joining (SAY) of SiC/SiC

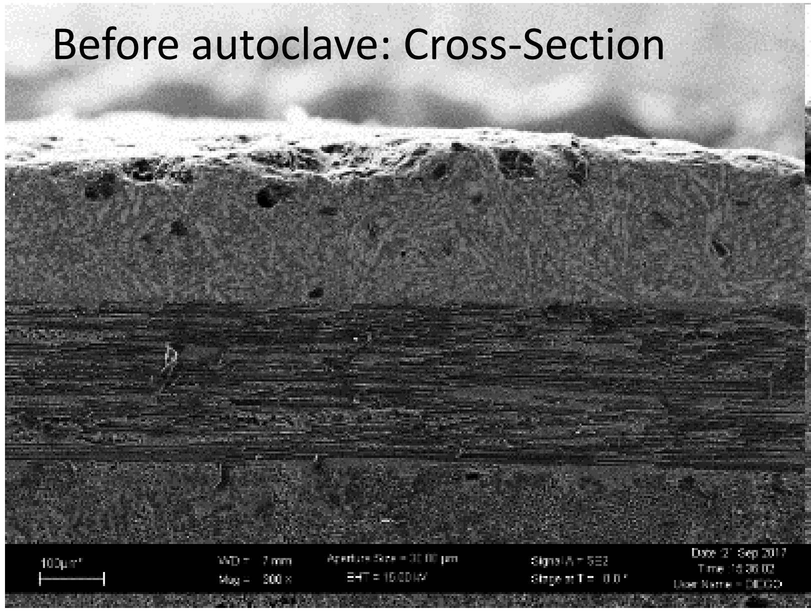
- Preliminary tests in autoclave

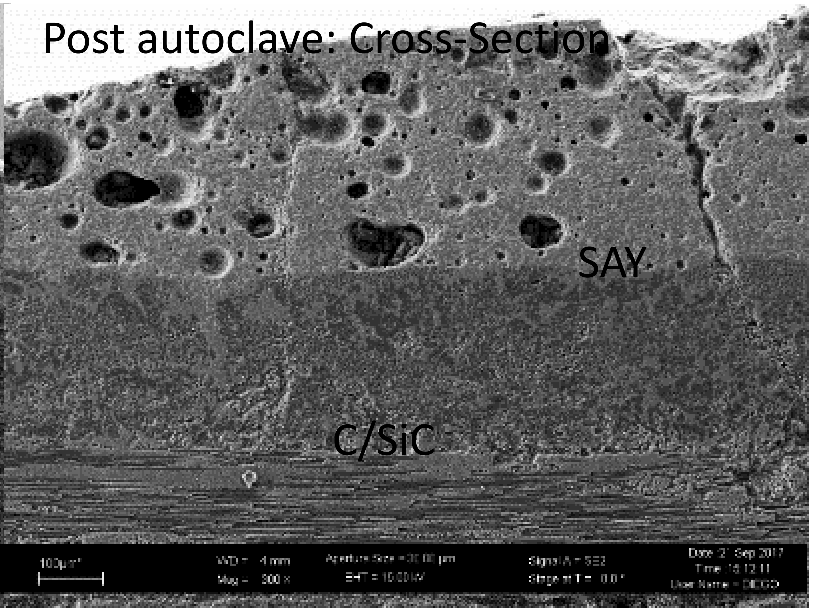
High Pressure Chemical Reactor
100 ml, Supercritical Fluid Technology

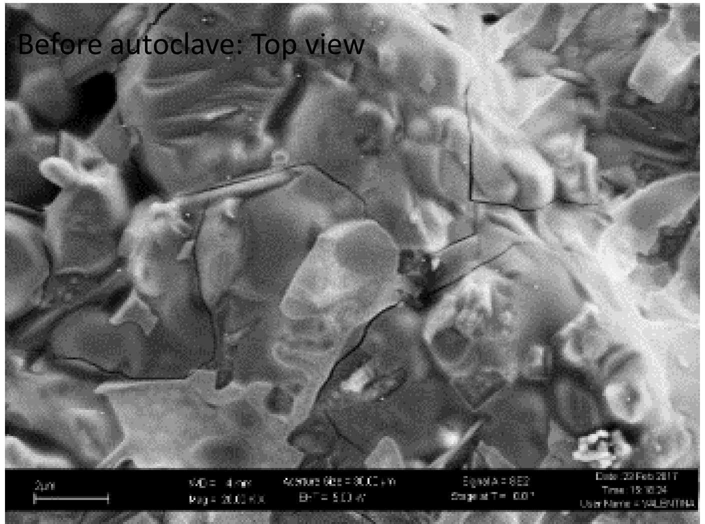
(POLITO, courtesy of Prof. L. Manna)

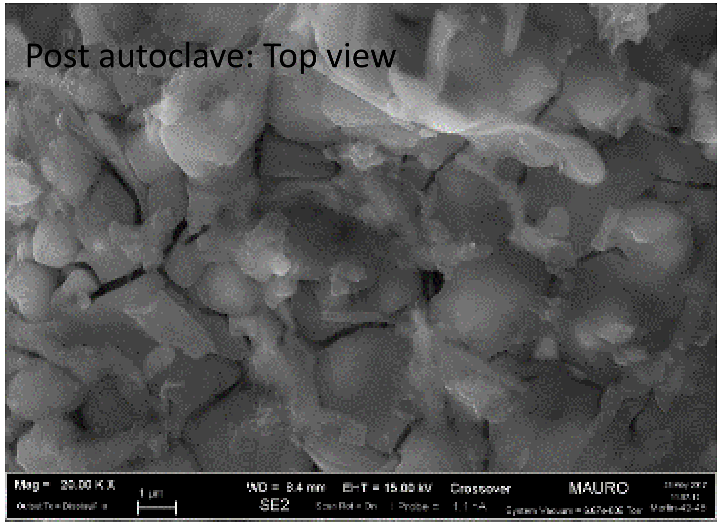
Temperature: 312-325°C
Pressure: 150 bar
Stirring: 60 rpm
Time to dwell temperature: 45 min
Dwell time: 8 Hours
Cooling time: 30 min

8-9 ppm oxygen dissolved in water









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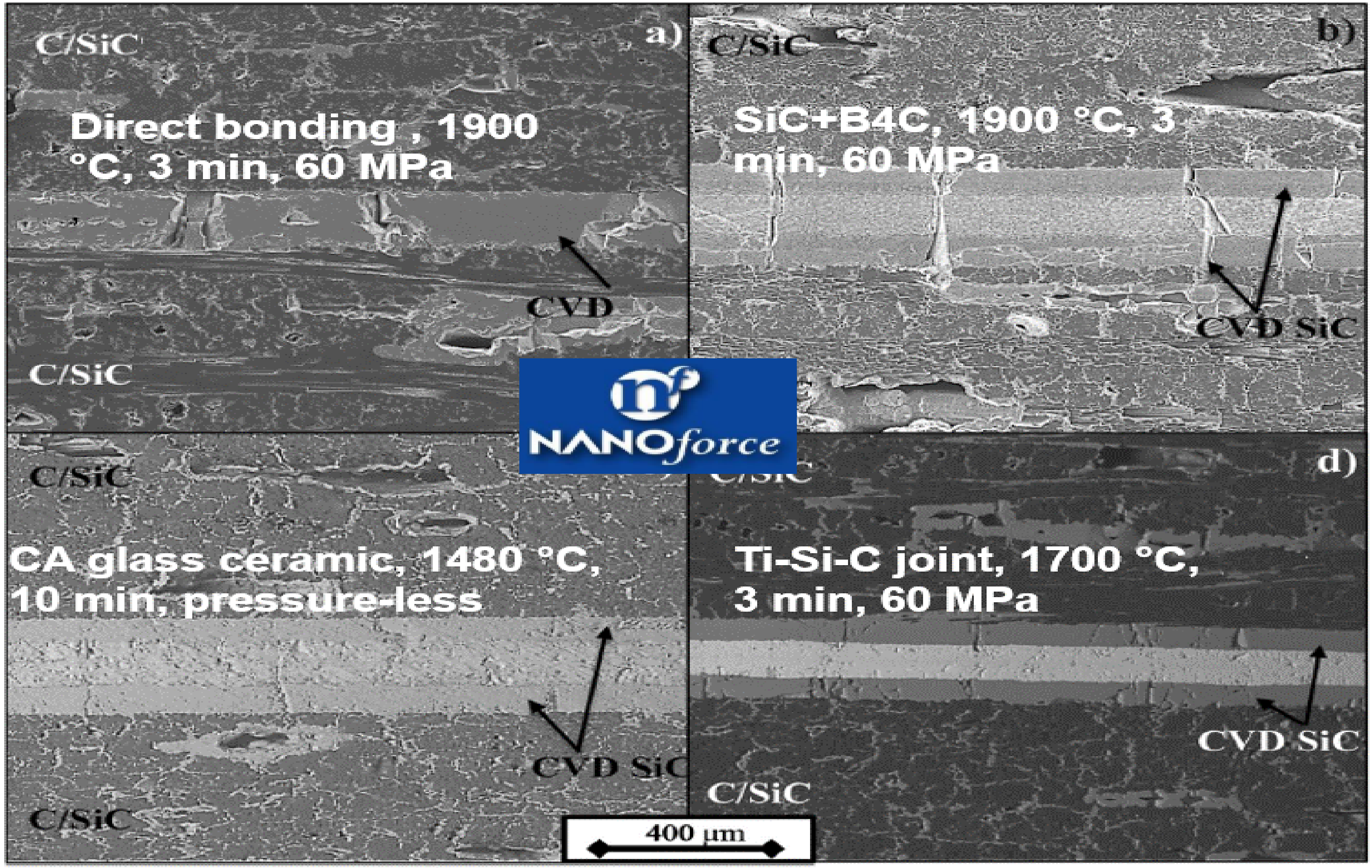
Pressure-less joining materials: SPS joining in collaboration with QMUL & Nanoforce, UK

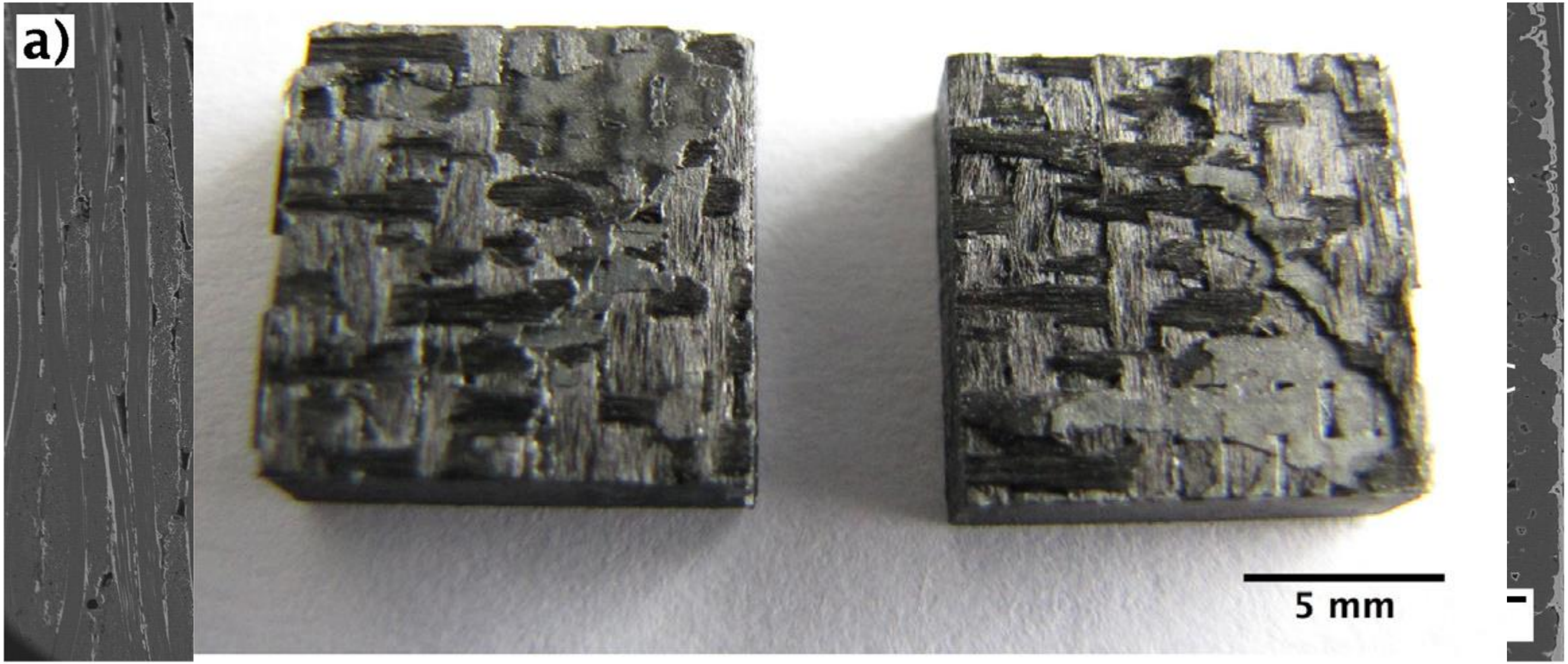
- **SPS direct bonding**
- ***pressureless* SPS successful with glass-ceramics as bonding material**
- **SPS suitable for MAX phases as bonding material**



Flash joining of CVD-SiC coated C f/SiC composites with a Ti interlayer P Tatarko, S Grasso, TG Saunders, V Casalegno… - Journal of the European Ceramic Society, 2017
Pressure-less joining of C/SiC and SiC/SiC by a MoSi2/Si composite PK Gianchandani, V Casalegno, F Smeacetto… - International Journal of Applied Ceramic Technology, 2017
Surface engineering of SiCf/SiC composites by selective thermal removal F Valenza, V Casalegno, S Gambaro, ML Muolo… - International Journal of Applied Ceramic Technology, 2017

Joining of C/SiC and SiC/SiC with pre-sintered Ti_3SiC_2 MAX phase by SPS: 1300 °C, 50 MPa, 5 minutes

Journal of the European Ceramic Society 36 (2016) 3957–3967



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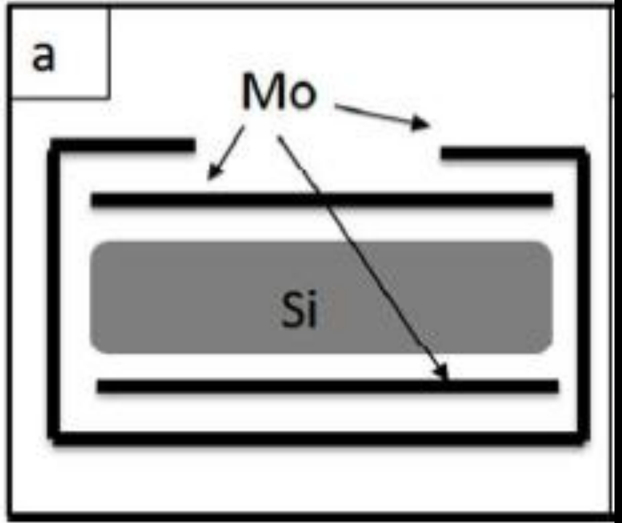
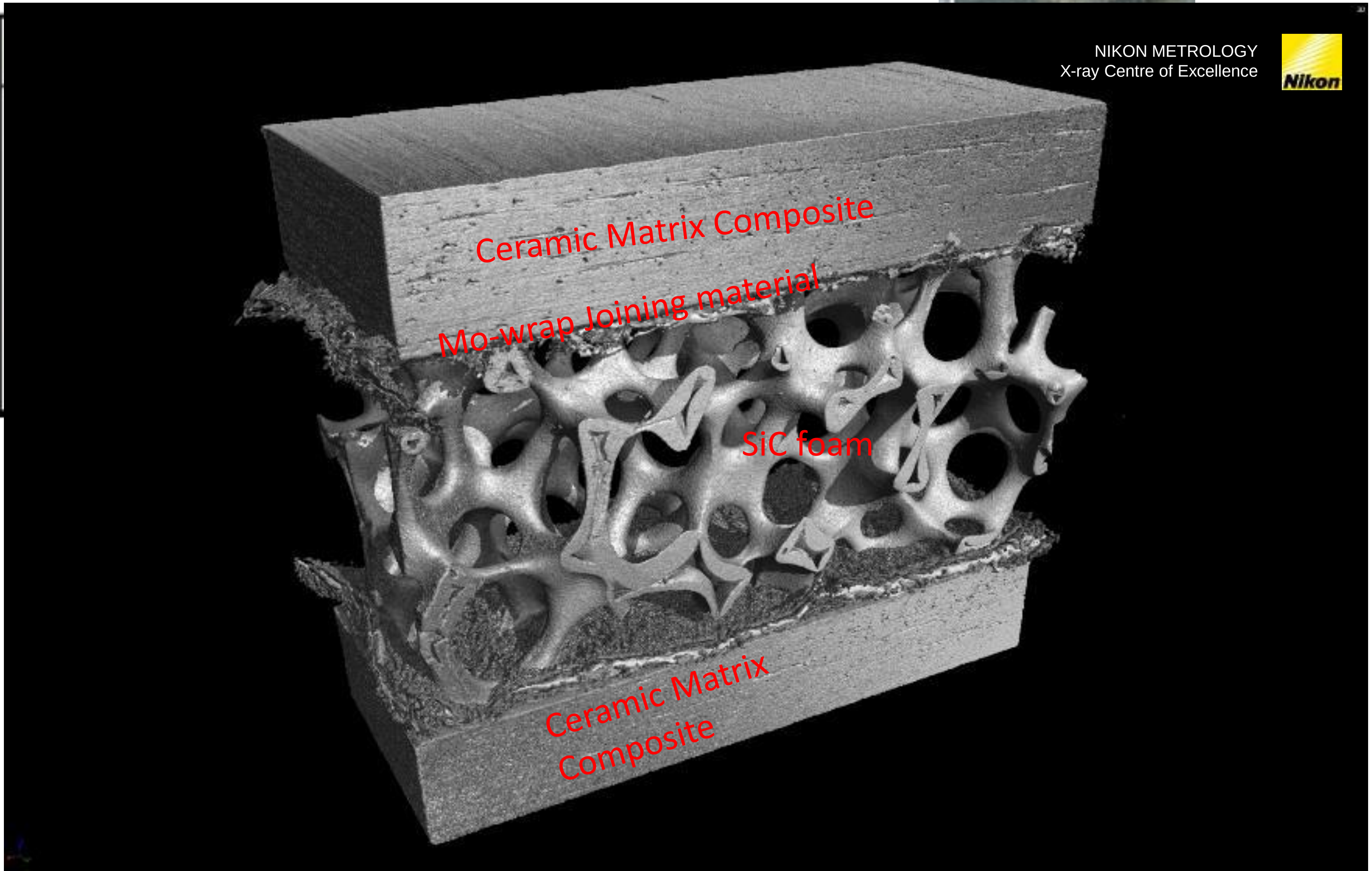
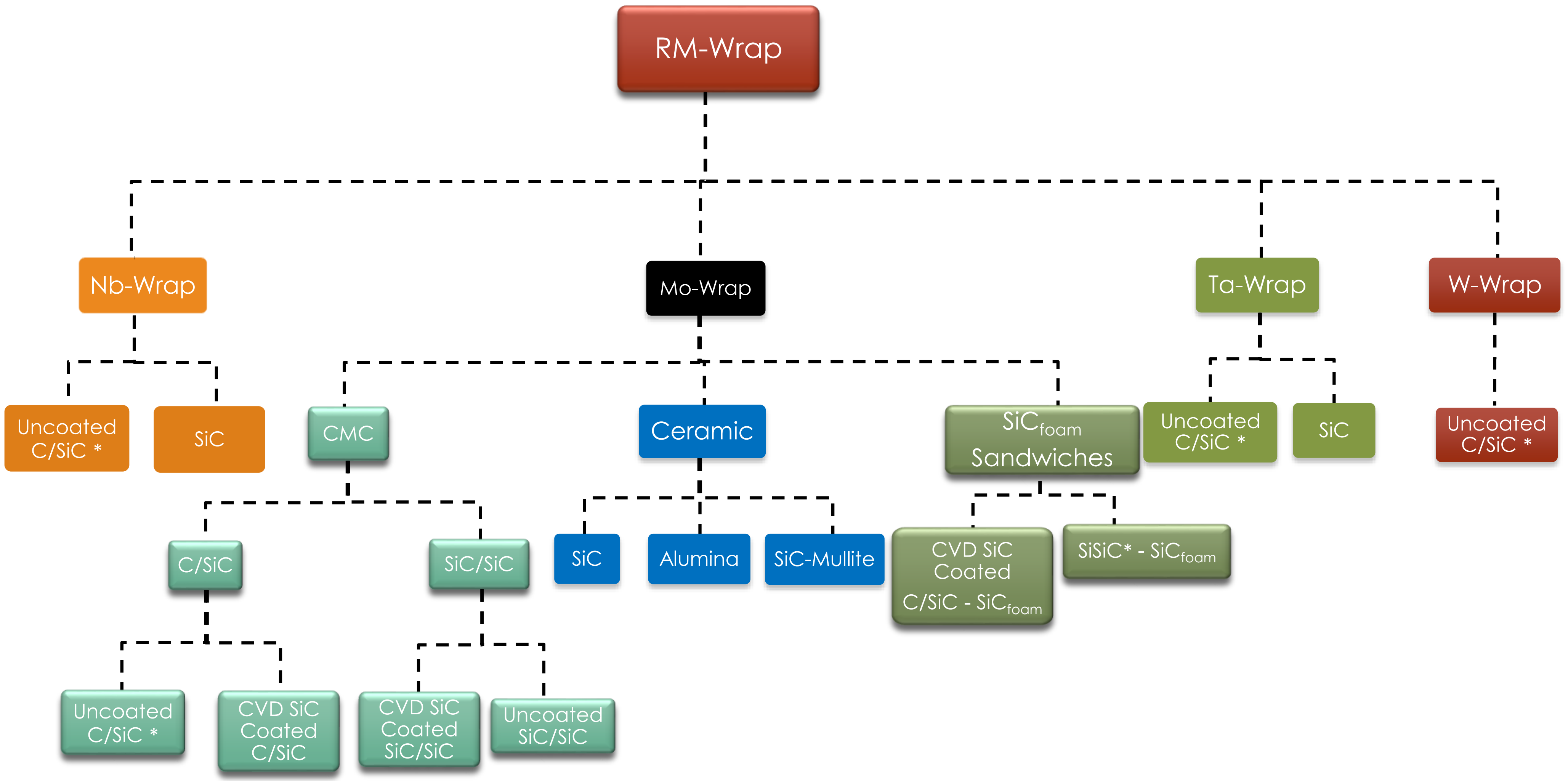
Wrap-joining: another pressure- less joining technique suitable for porous SiC-based components

Applied Ceramic Technology





Other «wraps» under testing



- If not differently specified, joints are between similar materials (e.g. SiC means joining between two SiC parts); SiC-mullite and Sandwiches are exceptions.
- RM-Refractory Metals
- CMC- Ceramic matrix composites
- SiC-foam$_f$: Silicon carbide foam
- SiSiC-Silicon carbide obtained by Liquid Silicon Infiltration (LSI)
- * silicon coating before RM-wrap

- **Preliminary results of Tungsten-Wrap are promising**
- **Nb/Ta Wrap can be applied on several samples as with Mo-Wrap**
- **Wide range of combination Mo, Nb, Ta and W- Wraps individually and together**

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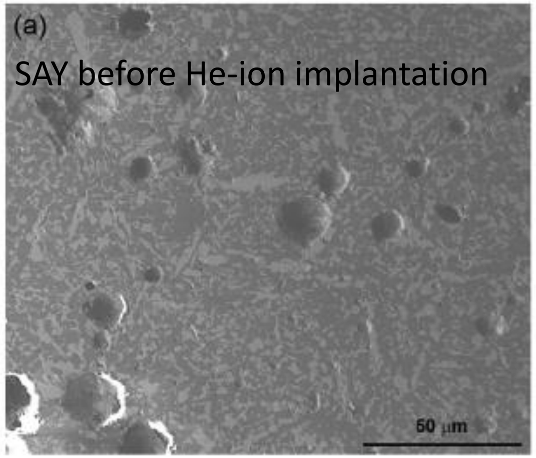
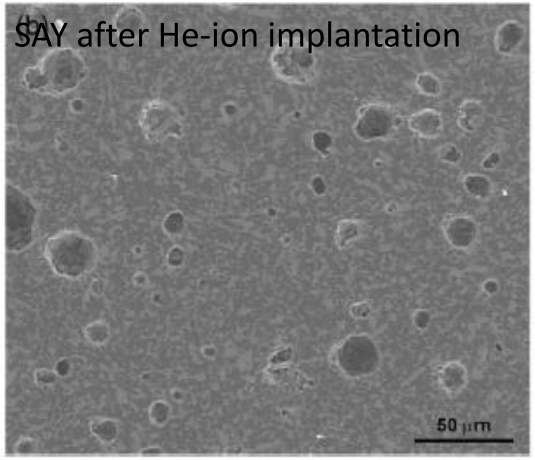
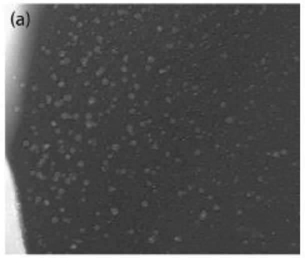
He-ion irradiation effects on glass-ceramics for joining of SiC-based materials

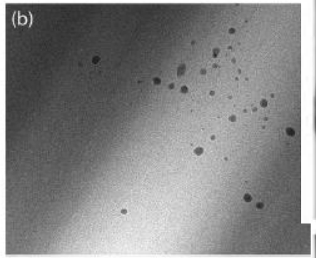
- Glass ceramics (SAY, CA) irradiated with 5.5 MeV He ions at an average temperature of 75 °C to a fluence of almost 2.3 10^{18} cm^2 (about 40 dpa in the ion implantation region) to simulate damage expected in nuclear plants.
- No amorphization was found even in correspondence to the He implantation zone.
- agglomerates of He-bubbles in the implantation region for the CA and a more homogeneous He-bubble distribution in the SAY.
- The radiation damage induced only occasional micro-cracks, mainly located at grain boundaries (CA) or within the grains (SAY)
- Si-ion irradiation on joined samples, ongoing

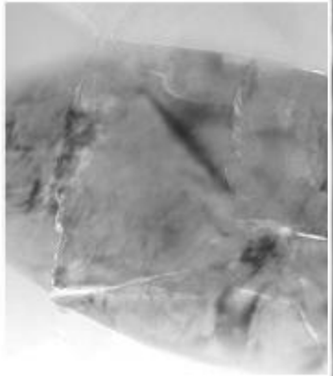
Journal of Nuclear Materials 472 (2016) 28e34

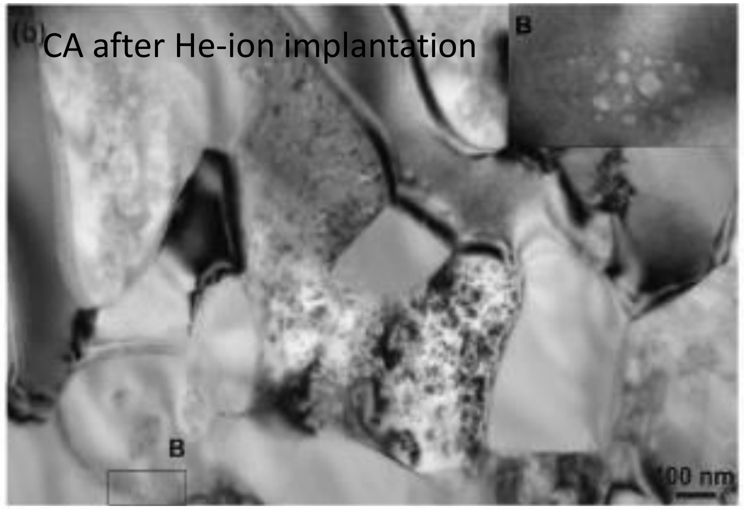






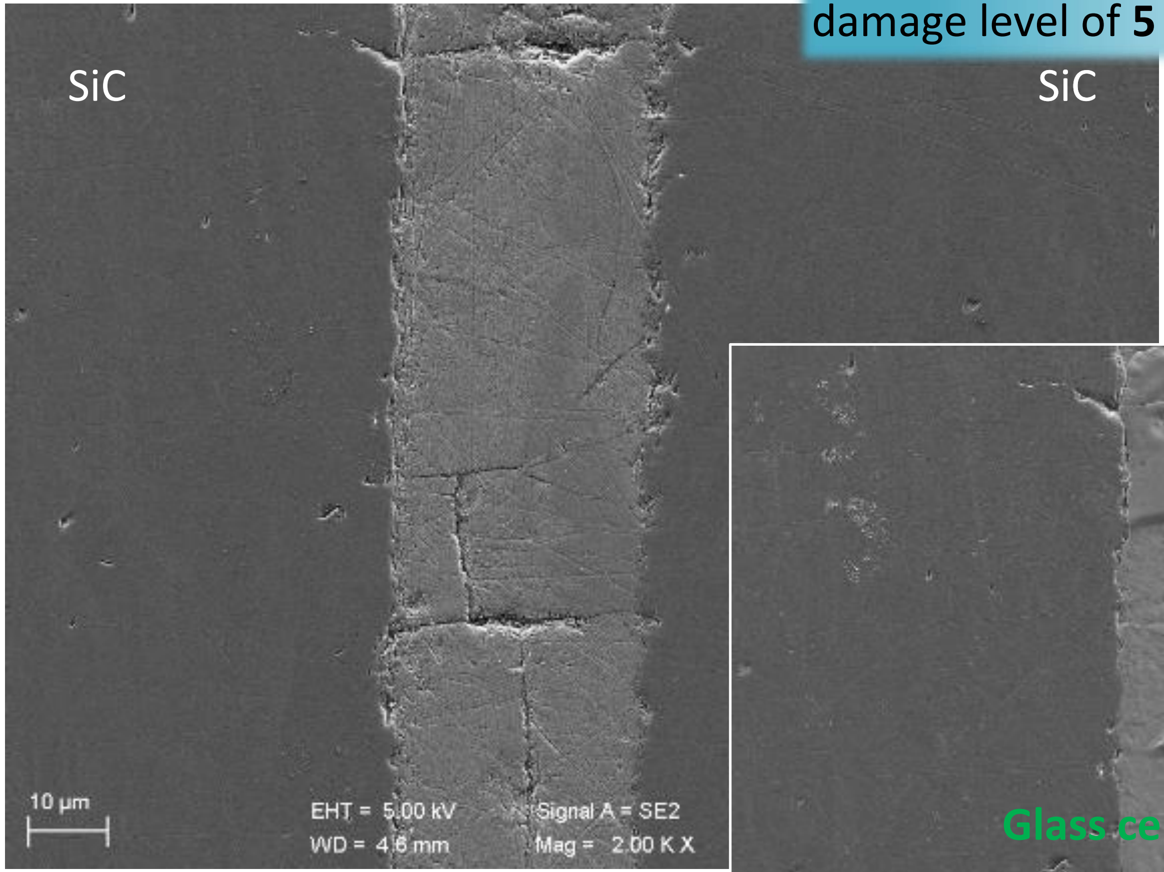






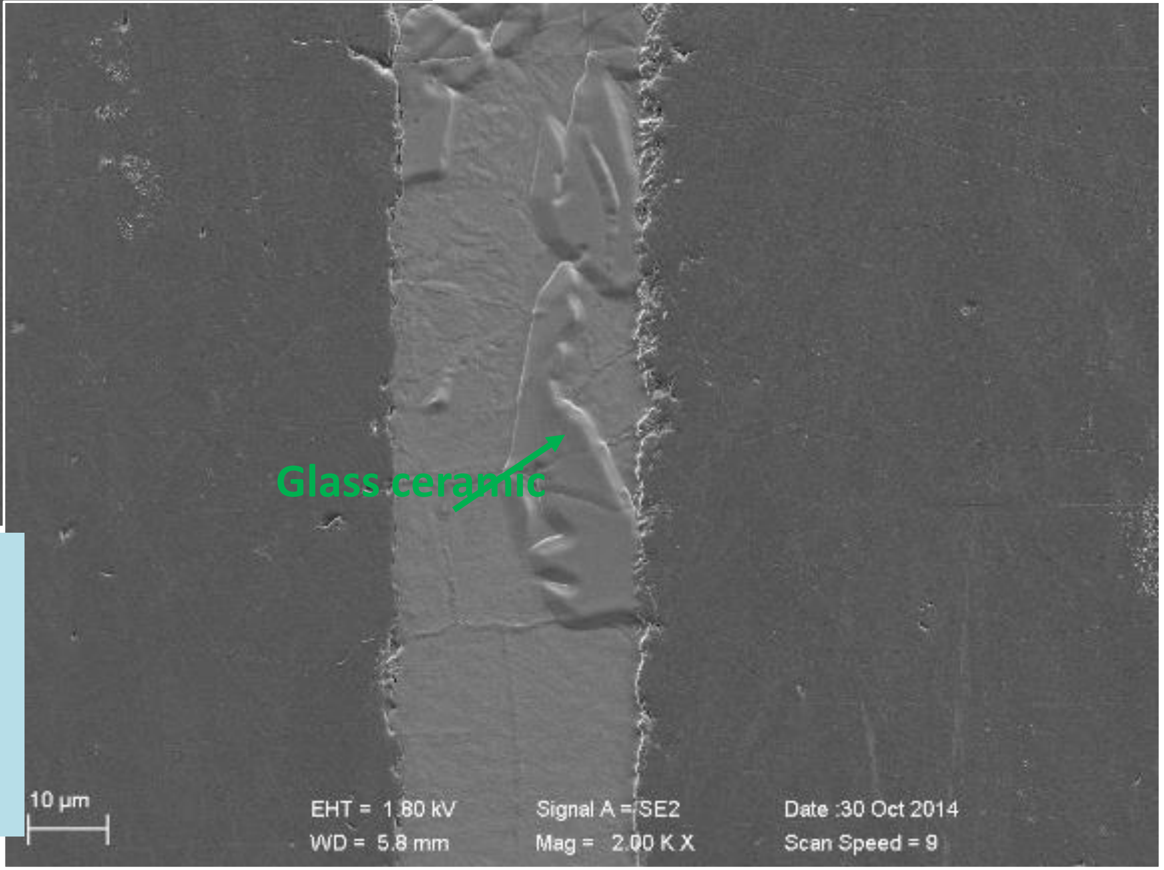

Journal of Nuclear Materials 472 (2016) 28–34
Contents lists available at ScienceDirect
Journal of Nuclear Materials
journal homepage: www.elsevier.com/locate/jnucmat
ELSEVIER
He-irradiation effects on glass-ceramics for joining of SiC-based materials
L. Gozzelino a, b, *, V. Casalegno a, G. Ghigo a, b, T. Moskalewicz c, A. Czyrska-Filemonowicz c, M. Ferraris a

The CA joined SiC were irradiated with **5.1 MeV Si2+** ions at temperature of **400 and 800°C**, corresponding to a damage level of **5 dpa** averaged over the damage range



Irradiation at 400°C

DuET facility, **Kyoto University**

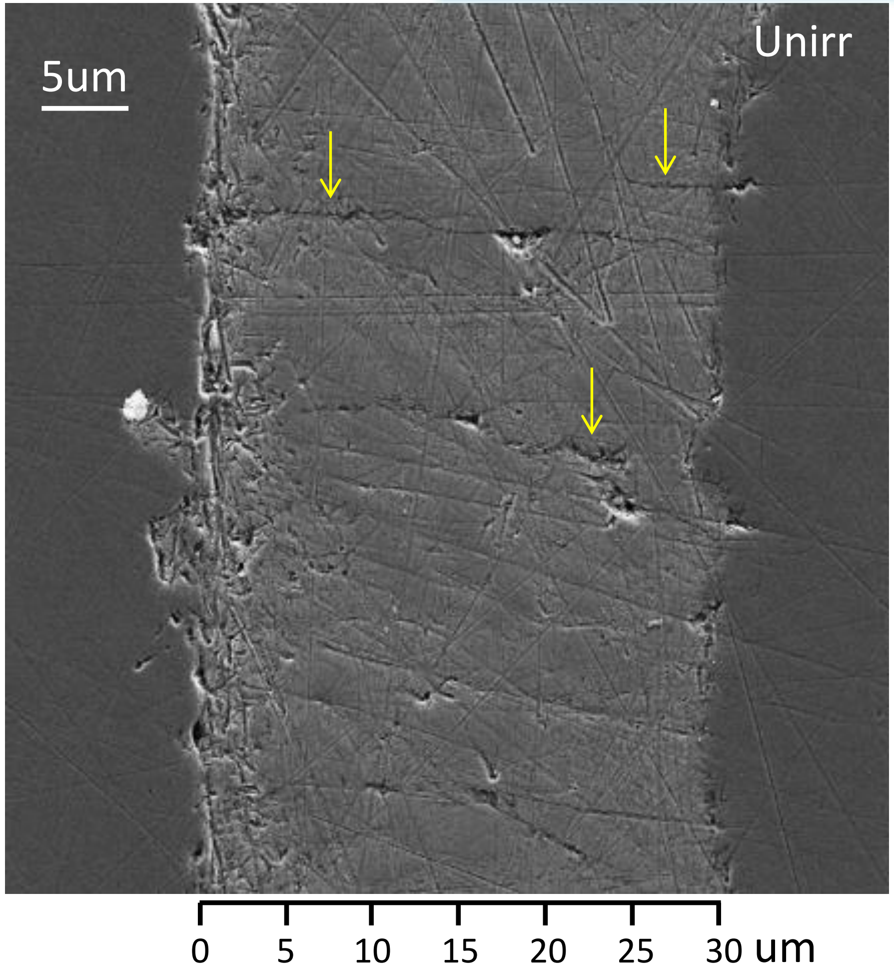


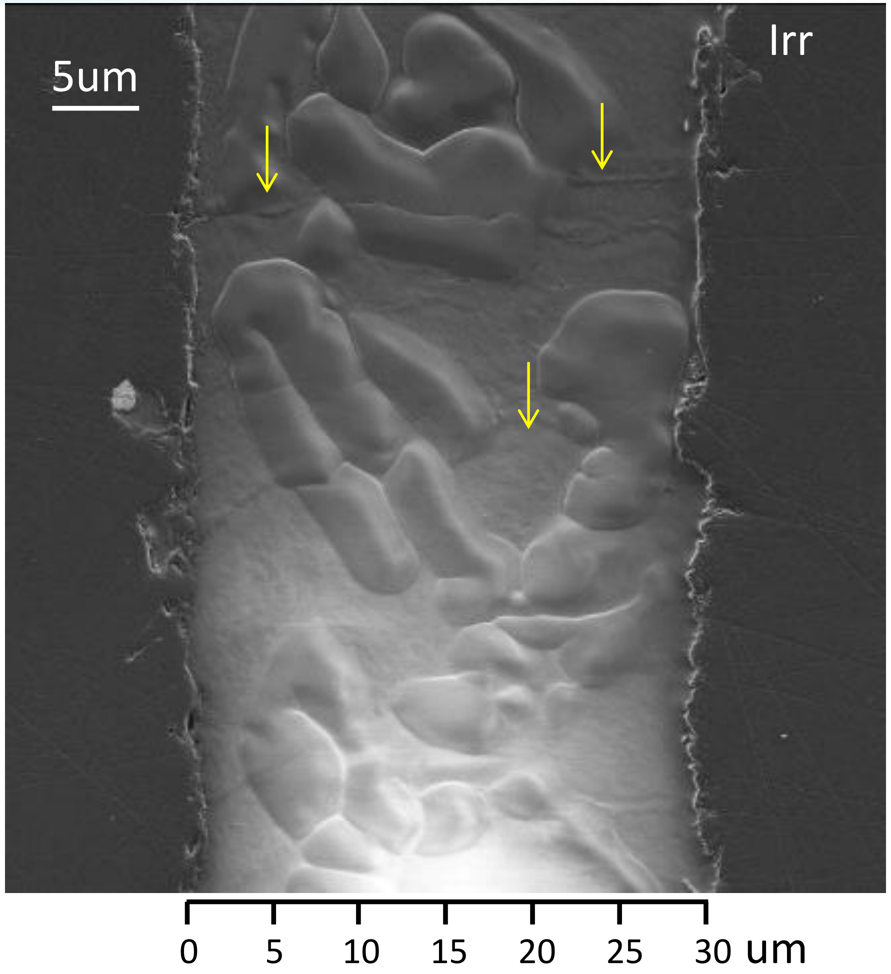
No debonding at the CA/SiC interface has been detected

Crack healing→ due to swelling?

The CA joined SiC were irradiated with **5.1 MeV Si2⁺** ions at temperature of **400 and 800°C**, corresponding to a damage level of **5 dpa** averaged over the damage range

DuET facility, **Kyoto University**





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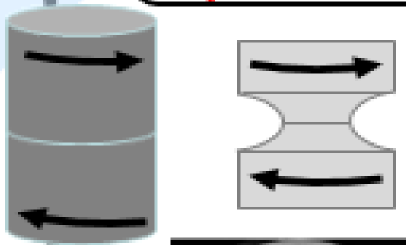
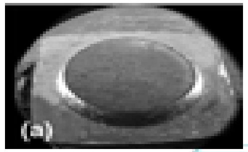
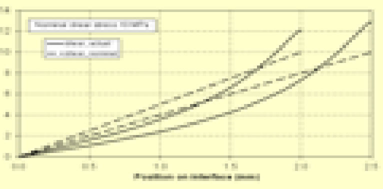
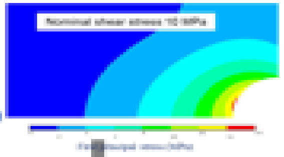
Joint strength measurement under torsion: flowchart (cohesive fracture*)

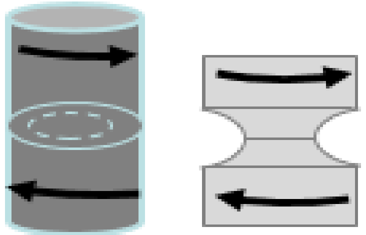
on shear strength of ceramics joined by brittle or ductile materials

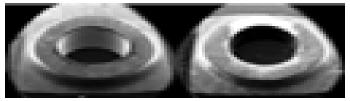












JOINING MATERIAL

BRITTLE

Circular (full) bond: rod or hourglass

If hourglass *: modelling needed
- to check if τ only in the bond section
- to calculate Kt (stress conc. factor)

* Modelling done for THG5, THG4

Torsion tests

$K_t * M_{max}/J * R$

Failure in the joint section: joint shear strength (failure due to max principal stress in the 45° skew plane, σ_{max})

Failure in the adherends: joint strength higher than adherend strength

DUCTILE

Ring shaped (annular) bond: tube or hourglass

Preliminary torsion tests: tentative ring width

$M_{max}/J * R$ with $J = \pi(R_o^4 - R_i^4)/2$

(Elastic-plastic modelling possible if joining material stress-strain curve available)

Further torsion tests: decrease ring width until constant joint strength is obtained

Failure in the joint section: joint shear strength (failure due to max shear stress in the cross section, τ_{max})

Failure in the adherends: joint shear strength higher than adherend strength

*In case of non-cohesive fracture, measured values give nonetheless indication on joint strength

International Journal of Adhesion & Adhesives 70 (2016) 46–52
Contents lists available at ScienceDirect
International Journal of Adhesion & Adhesives
journal homepage: www.elsevier.com/locate/ijadhadh

Bonding of ceramics: An analysis of the torsion hourglass specimen

L. Goglio[a,*], M. Ferraris[b]

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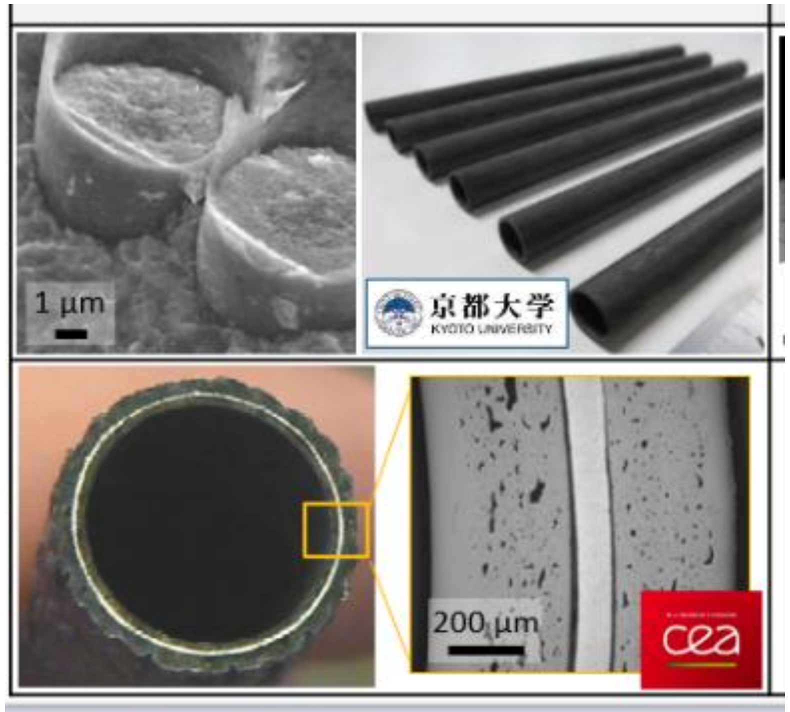
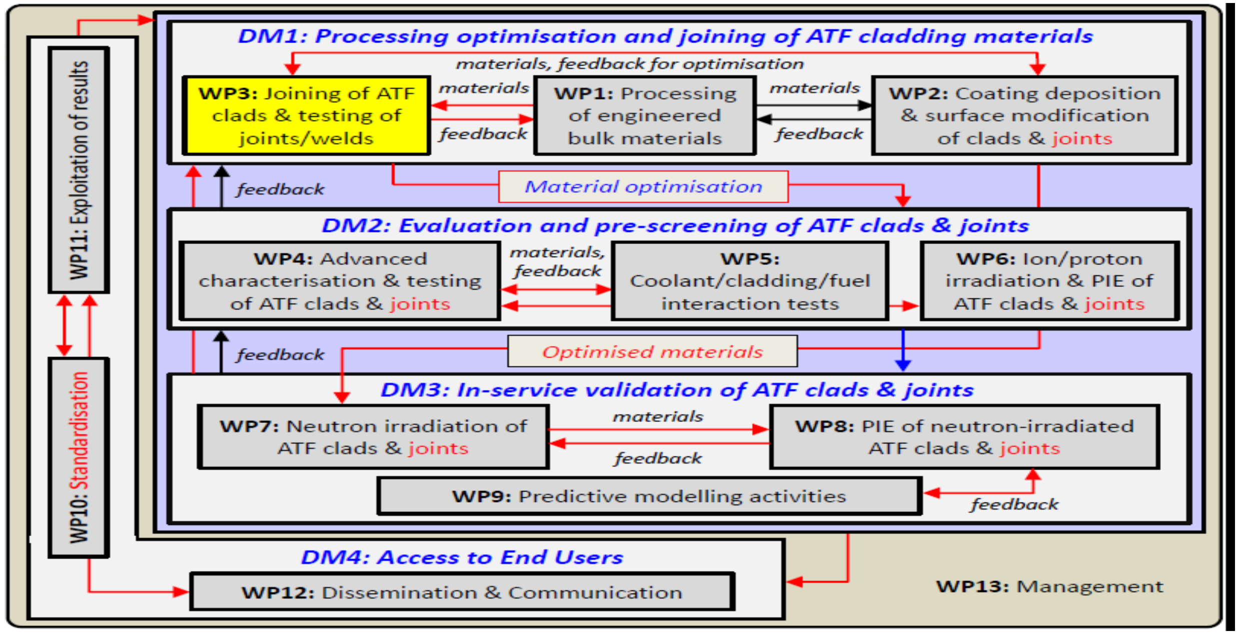
INNOVATIVE CLADDING MATERIALS FOR ADVANCED ACCIDENT-TOLERANT ENERGY SYSTEMS

Overall budget: 5 M€ EU contribution + 1.5 M€ additional contributions

Kick-Off Date: 1st October 2017 Project Duration: 4.5 years (54 months)



WP3. Joining of ATF clads & testing of joints/welds



J-TECH@POLITO

Advanced Joining Technology at POLITO

Prof. Luca Goglio (DIMEAS)
Prof. Monica Ferraris (DISAT)
Prof. Franco Lombardi (DIGEP)

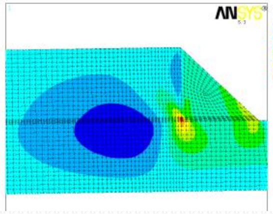
Kick Off Meeting

20 ottobre 2017

THE IDEA

DIMEAS

- Design and testing of assemblies subassemblies and components
- Modeling and structural optimization
- Non-destructive testing



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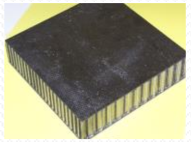
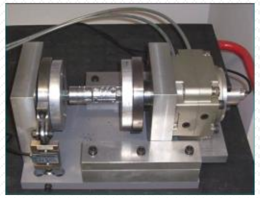
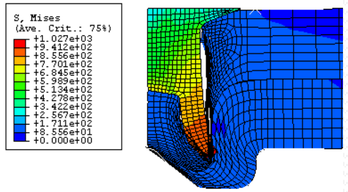
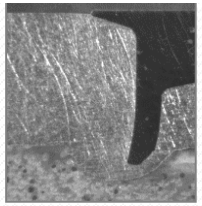


- Materials design, characterization and optimization
- Joining materials design, characterization and optimization



Establishing a top-class centre on advanced joining technology





DIGEP

- Process design, modelling and optimization
- Monitoring and control procedures and algorithms
- Process industrialization
- Cost and sustainability

COMPANIES

element
Consiglio Nazionale delle Ricerche
Henkel
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CRF Centro Ricerche Fiat
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Fraunhofer
Listemann AG
Airbus
KMM.vin
Queen Mary University of London
MT Aerospace
Endurance

J-TECH@POLITO new facilities

- **Customized facility** for **Non Destructive Testing (NDT)** of **joined components** and **materials,** based on X-ray tomography

- **Multipurpose joining & testing facility** including **heating stage/mechanical testing** equipment operating in **vacuum, inert** and **reactive atmosphere, temperature** range **-80;+1450 °C**

- **Friction Stir Welding station**

- **Laser Nd-YAG head** to be applied on an existing 6 axis anthropomorphic robot, thermographic system for inspection

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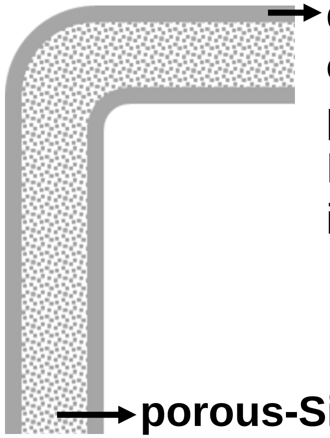
Development of a SiC-sandwich material for FCIs

Candidate material for **FCIs** in the **high-temperature DCLL**: **SiC**

- suitable for high temperature applications
- high chemical stability
- adequate for radiation environments
- ↑ *thermal & electrical conductivities* → insulating SiC material: R&D needed

Possible option: SiC-based sandwich with porous & dense layers



dense CVD-SiC coating
protection against PbLi corrosion & infiltration

porous-SiC core
thermal & electrical insulation

- **k < ~10 W/(m·K)**
- **σ < ~1 S/m**

Enabling Research EUROfusion

- Production of porous SiC
- Characterization of porous & coated SiC samples: mechanical & thermal properties, electrical conductivity, corrosion tests in PbLi
- Analyze the feasibility of using a SiC-based sandwich as material for FCIs
- Exploration of possibilities for industrial fabrication

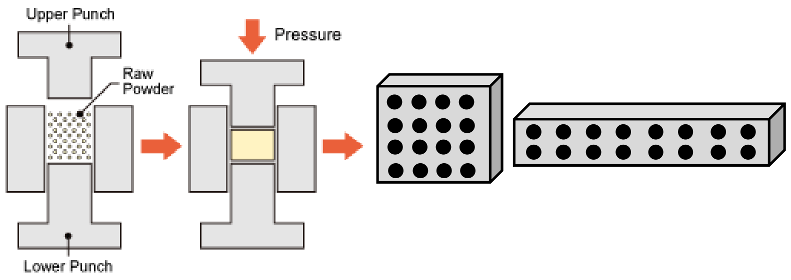
Fabrication procedure

Based in the *sacrificial template* concept: a sacrificial phase is added to the initial powder mixture → phase removed: **pores**

1. Powder mixture

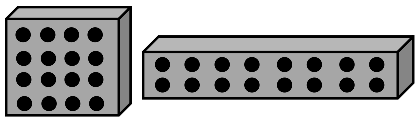
- SiC (0.3 µm)
- Sintering additives (Al_2O_3, Y_2O_3) – 2.5 wt.%
- Sacrificial phase → spherical graphite powder (MCMB, ~15 µm)

2. Uniaxial pressing

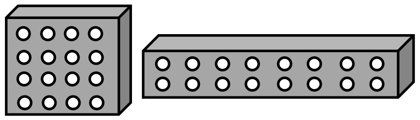


3. Sintering

1900 ^{0}C
30 min



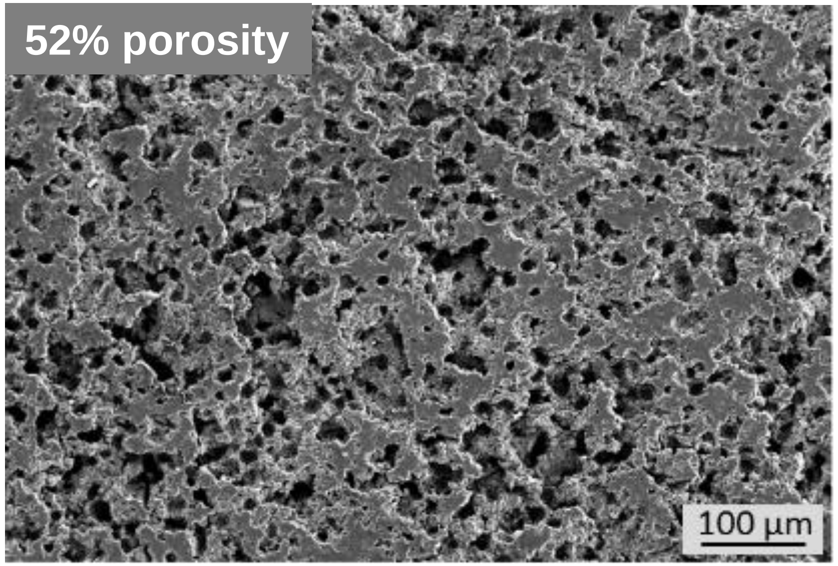
4. Oxidation

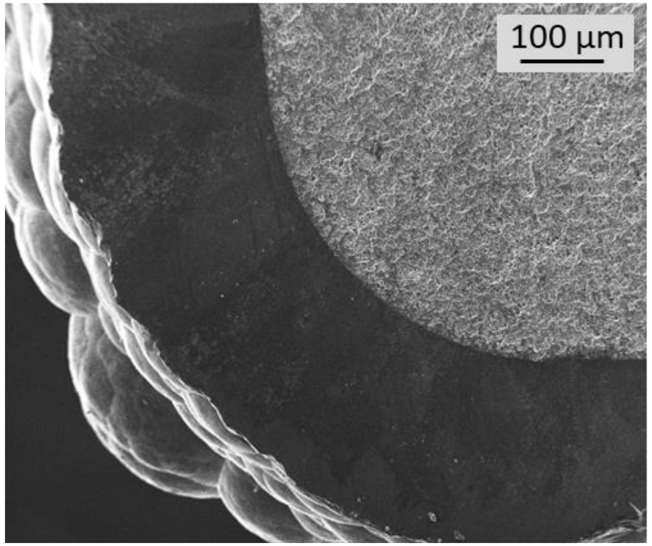


700 ^{0}C
10 h

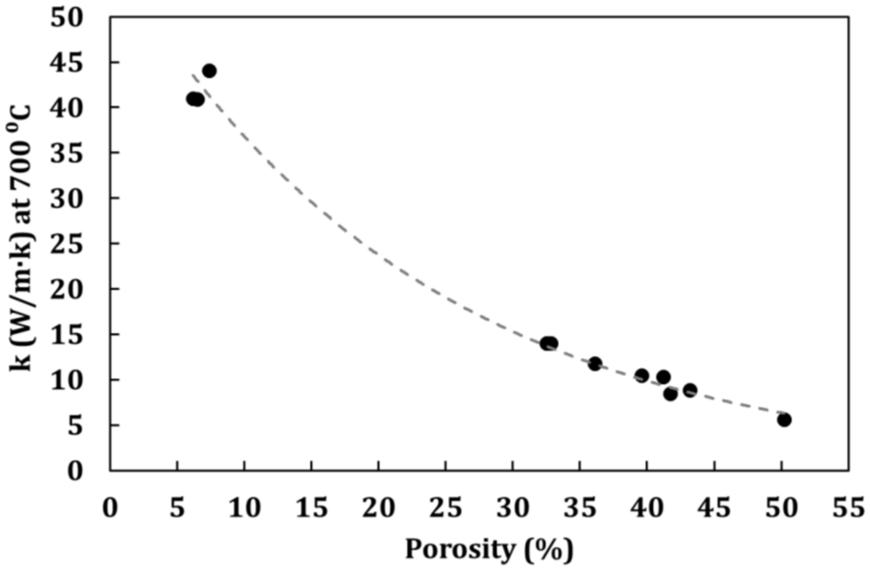
→ reaction between air & graphite
→ **porous samples**

▪ SiC sandwich material: microstructure, CVD coating, properties

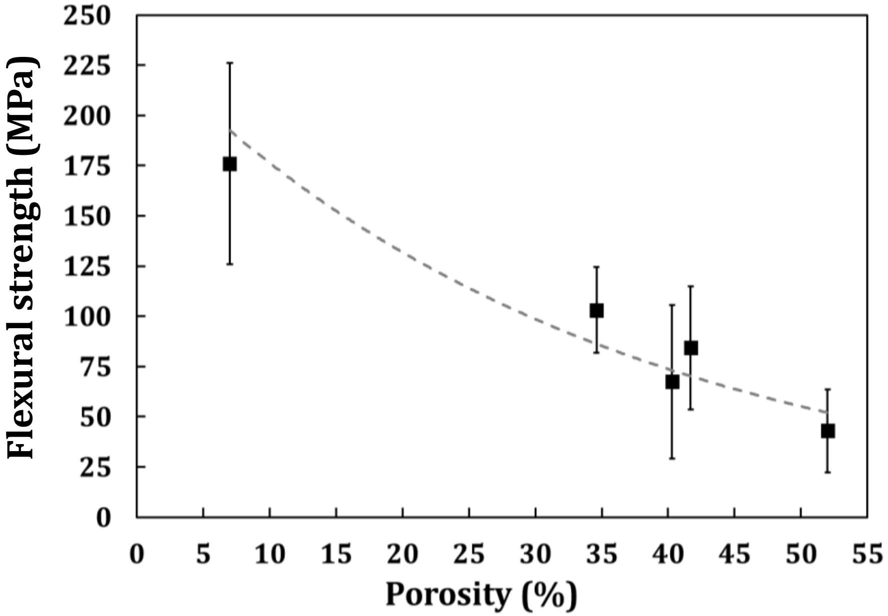




Porous SiC covered by 200-400 µm CVD-SiC



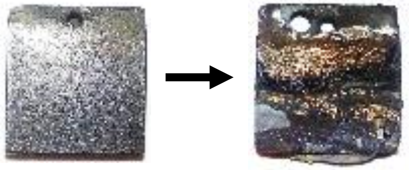
at 700 °C: **k < 10 W/m·K** if **porosity > 40%**



▪ 1st corrosion experiment: flat samples under static PbLi

before **after**

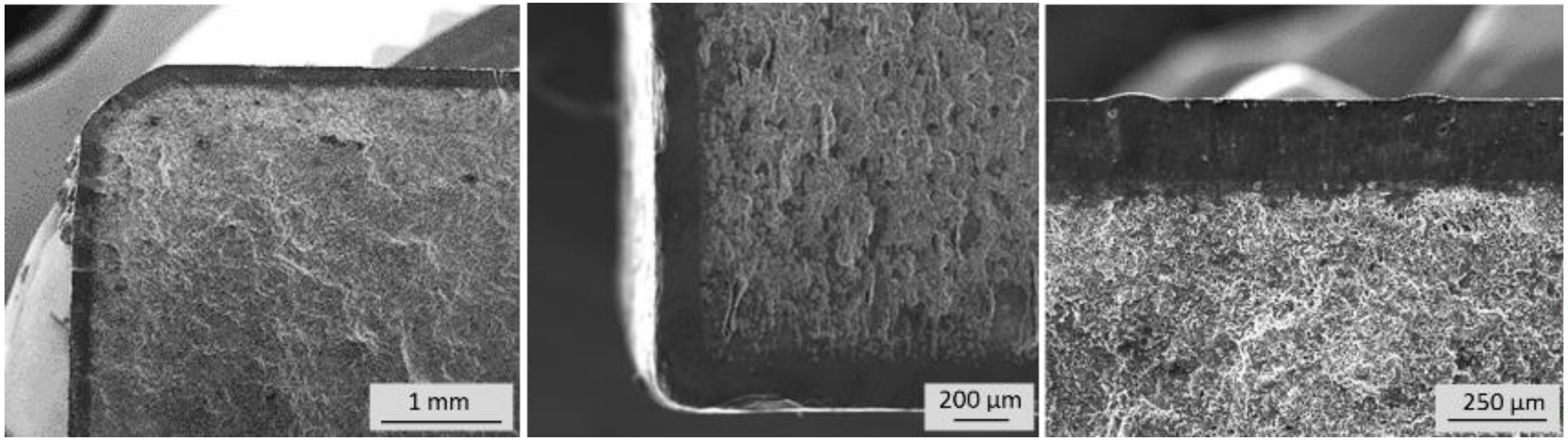
6 lab-size samples



immersed in static PbLi at **700 ^{0}C for 1000 h**

50% porous core with a ~200 µm CVD-SiC coating

▪ **Samples after the experiment**



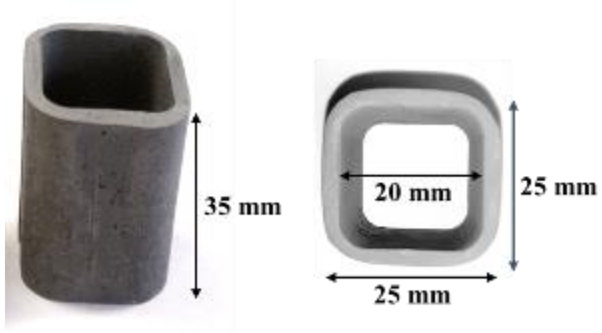
no PbLi infiltration & no signs of corrosion in the dense coating

▪ 2nd corrosion experiment: flat samples under flowing PbLi

- New batch of samples → 40% porous core & CVD-SiC coating of **200-400 µm**
- 11 samples tested
  - 8 subjected to a **1.8-2 T magnetic field**
  - 3 outside the magnetic field as control samples
- PbLi flowing at **~10 cm/s** and ~**550 ^{0}C for 850 h**

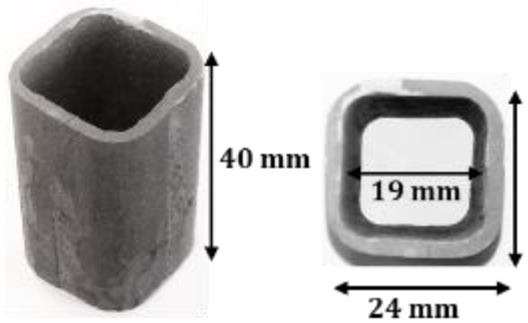
→ no damage, no PbLi infiltration, no signs of corrosion of dense coating

▪ Fabrication of porous SiC hollow samples by gelcasting method: first results

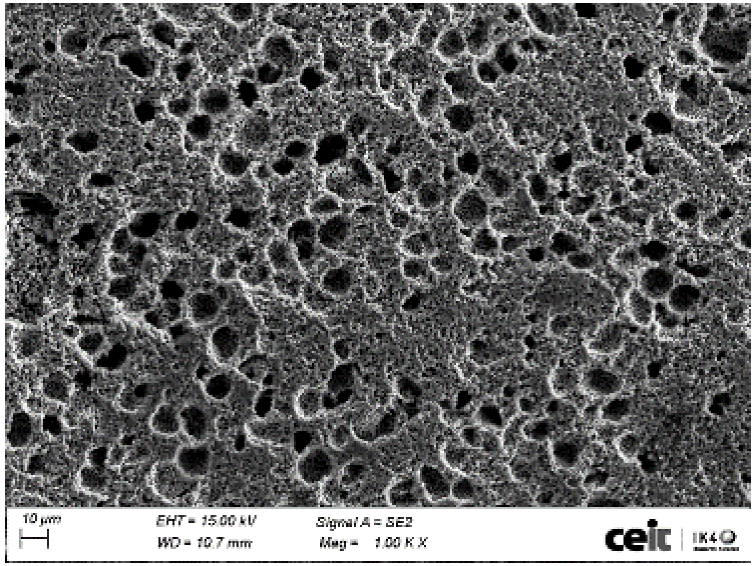


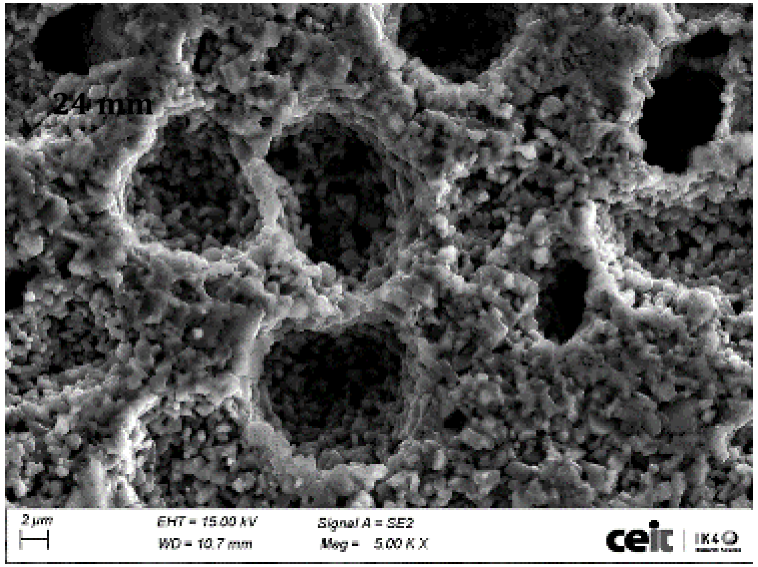
Samples will be coated with CVD-SiC

→ **lab-scale FCI prototypes**



	Initial MCMB (wt.%)	Final porosity (%)	Flexural strength (MPa)
Gelcasting	20	44	79 ± 11
Uniaxial pressing	20	42	84 ± 31





EU program update

Thank you for your attention

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