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Glass Ceramic Joined SiC/SiC: Properties after Nuclear Irradiation

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Joining SiC/SiC for Nuclear Applications: DEMO ?

Å *several meters long* joints

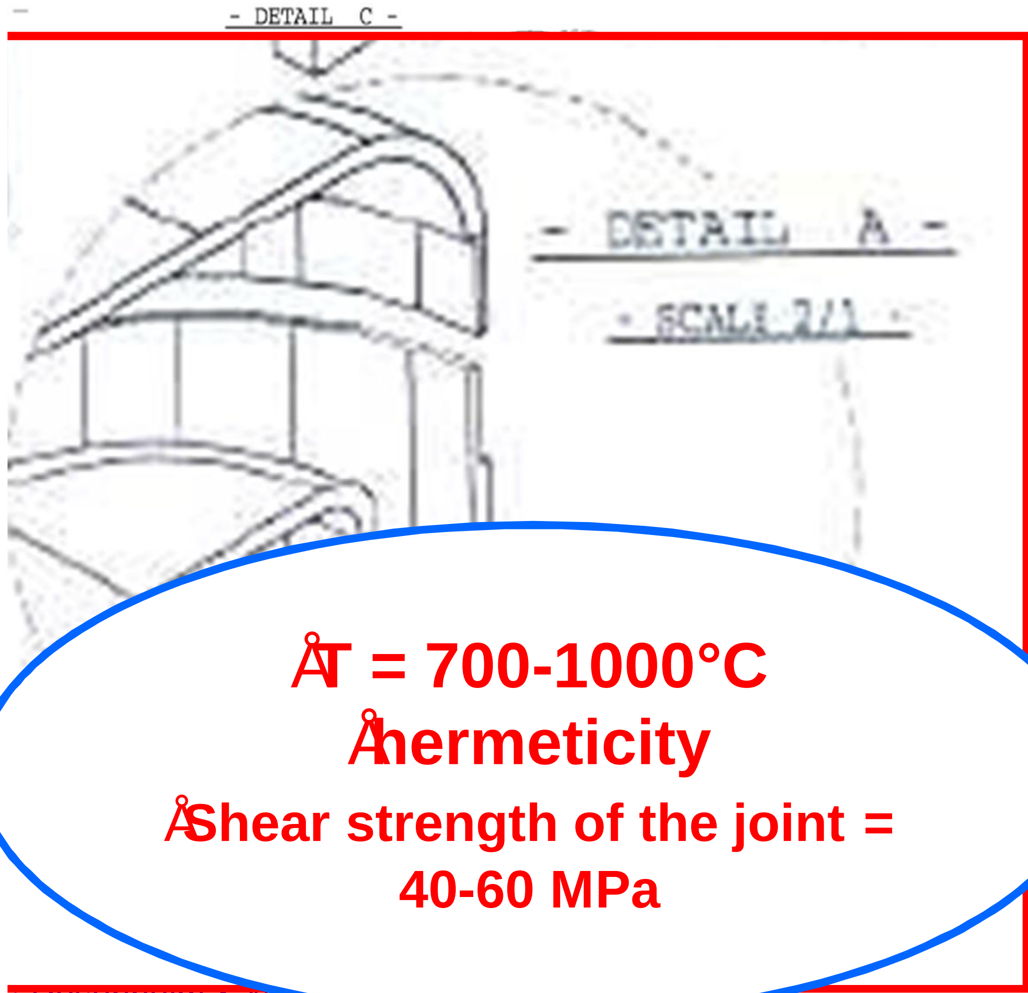
Å Neutron irradiation resistance

Å Low activation and transmutation

Å Thermo-mechanical reliability

Å pressure-less methods

Å Localized heating



ÅT = 700-1000°C

Åhermeticity

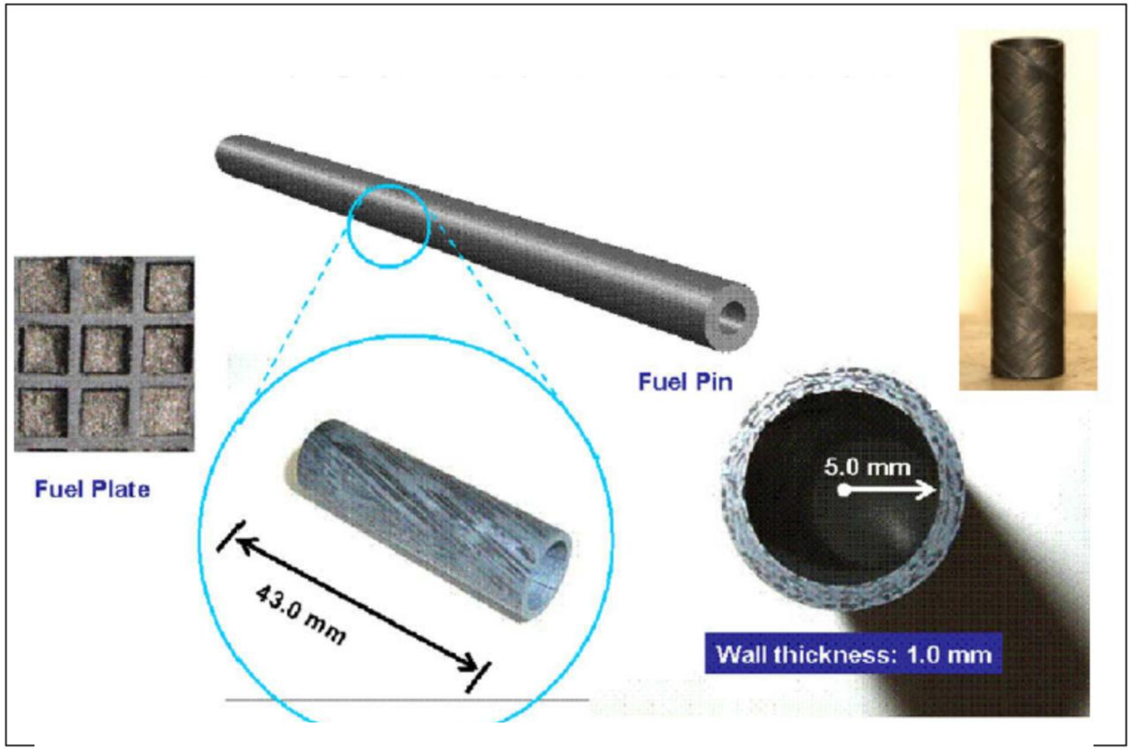
ÅShear strength of the joint = 40-60 MPa

Joining SiC/SiC for new generation fission reactors components?

silicon carbide-based components:

fuel cladding tubes in advanced light water reactor (LWR)

control rod sleeves in a very high temperature reactor/next generation nuclear power reactors



NITE SiC/SiC fuel pin or fuel plate for GFR;
courtesy of Ceramic Tubular Products (CTP, USA) triplex tube

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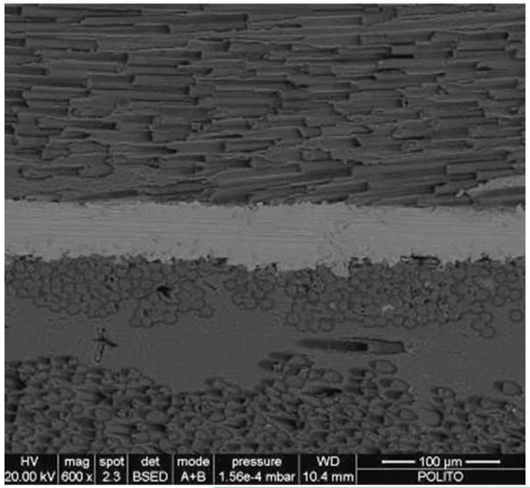
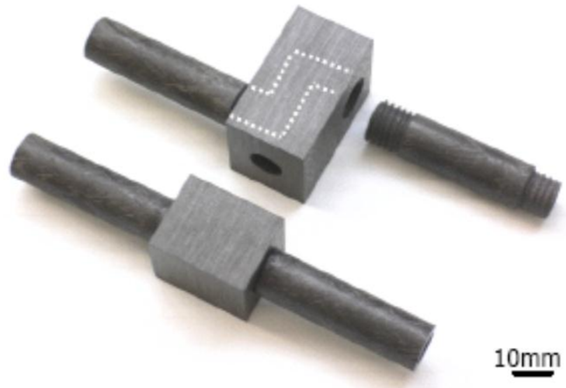
Joining SiC Ceramics and Composites for Nuclear Reactors beyond ITER: DEMO

development of joining materials and technology

é .?





Examples of Potential Materials and Techniques for Joining SiC-based Materials for Nuclear Applications

Method	Typical strength	Radiation performance	On-going / recent R&D
Diffusion bonding w/ metallic inserts	>~150 MPa shear	Expectedly good with adequate insert materials	NASA, Bettis, EU fusion
Transient eutectic-phase joining	~250 MPa tensile	Expectedly good with process optimization	Kyoto U., Dresden, etc.
Glass-ceramics Joining	~50 MPa apparent shear	Positive result from EU program	Politecnico di Torino
Brazing	N/A	Generally poor High induced activation	Snecma, ENEA, etc.
Reaction bonding (SiC+Si)	~200 MPa shear	Unstable due to residual silicon	NASA
Reaction bonding (TiSiC)	50 MPa	Unknown	PNNL, Politecnico di Torino
Polymer joining	~10 MPa shear	Unstable due to residual oxygen and nano-crystalline phase	PNNL, etc.
Transient Liquid Phase Metal Joining	N/A	Unknown	Politecnico di Torino
Selective area CVD	N/A	Should be good	U.Conn, etc.

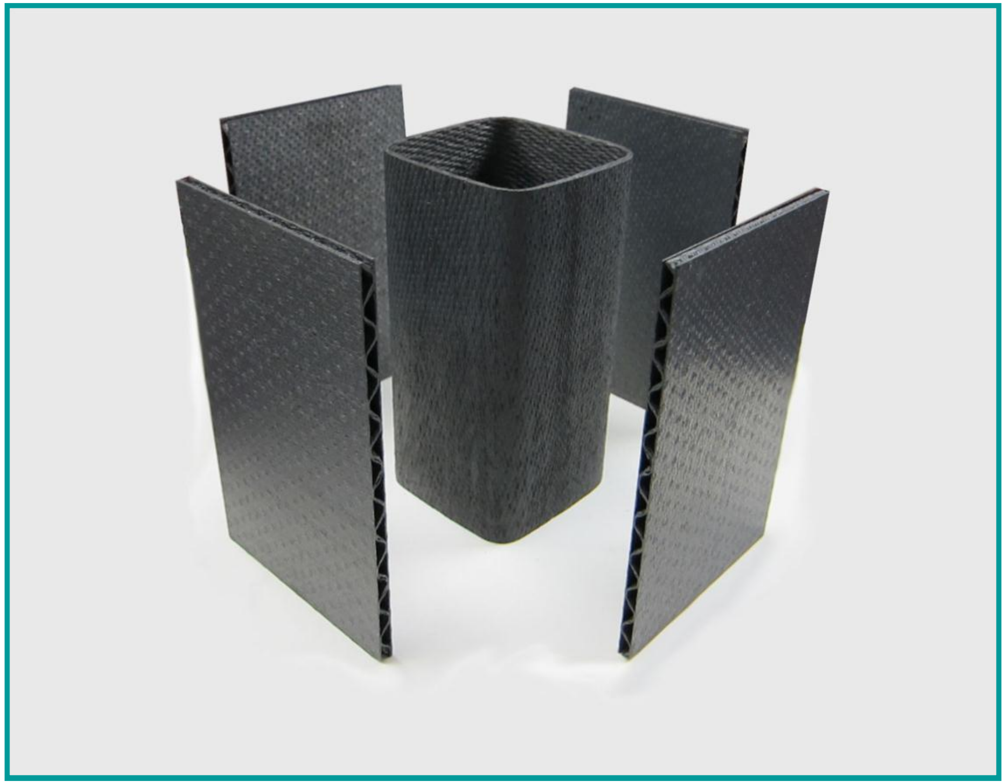
US/Japan TITAN CollaborationTask 2-2 SiC/SiC Joining and Coating

Y. Katoh (Oak Ridge National Laboratory, USA) et al. -- 33rd International Conference on Advanced Ceramics and Composites January 2009 Daytona Beach, Florida (USA)

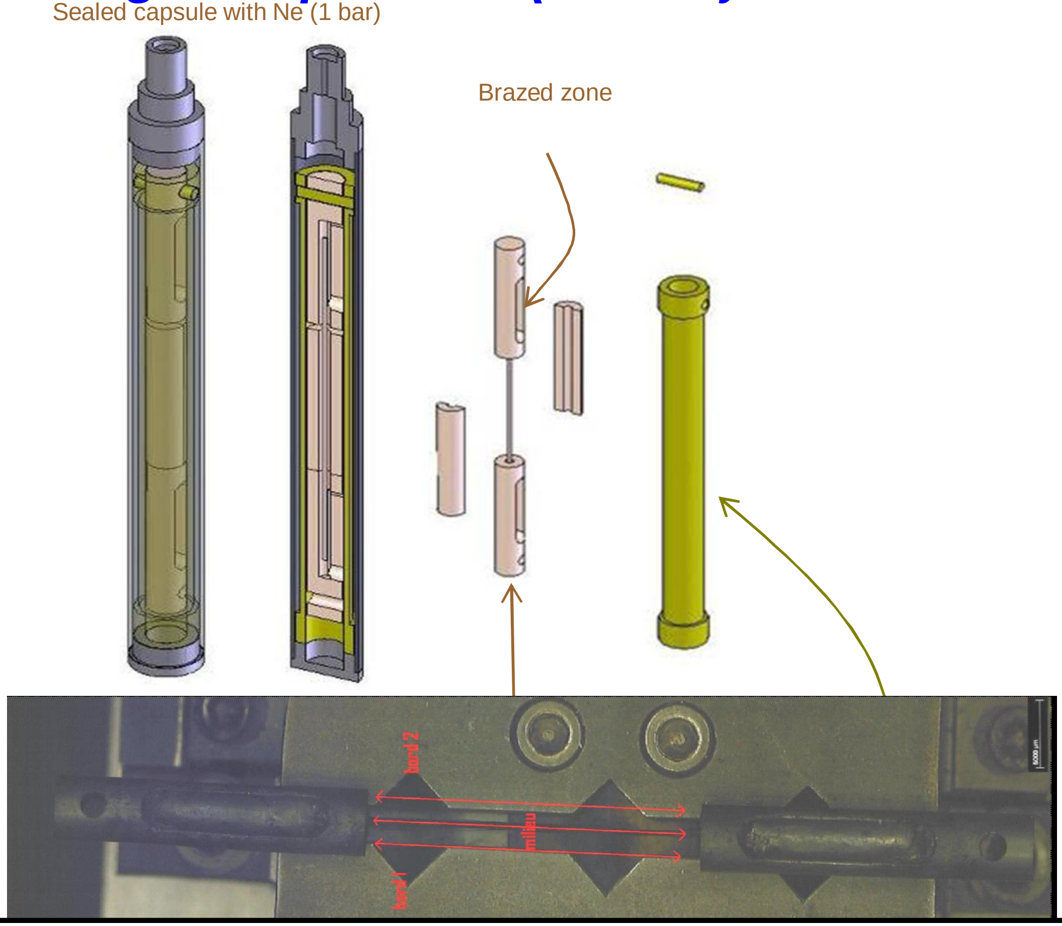
Fabrication of SiC/SiC Flow Channel Insert nested-Design for a DCLL fusion reactor

(*courtesy of R.J. Shinavski Hyper-Therm HTC, USA*)

- Edges of thermal panels must be sealed
- **Plan is to bond Nuclear Grade SiC/SiC inserts in the edges using Ti_3SiC_2 MAX phase**
- MAX phase has unproven irradiation resistance



Test the behavior of brazed joint and SiC/SiC minicomposite mechanical behavior after irradiation at high temperature (courtesy of A. Michoux-CEA, France)



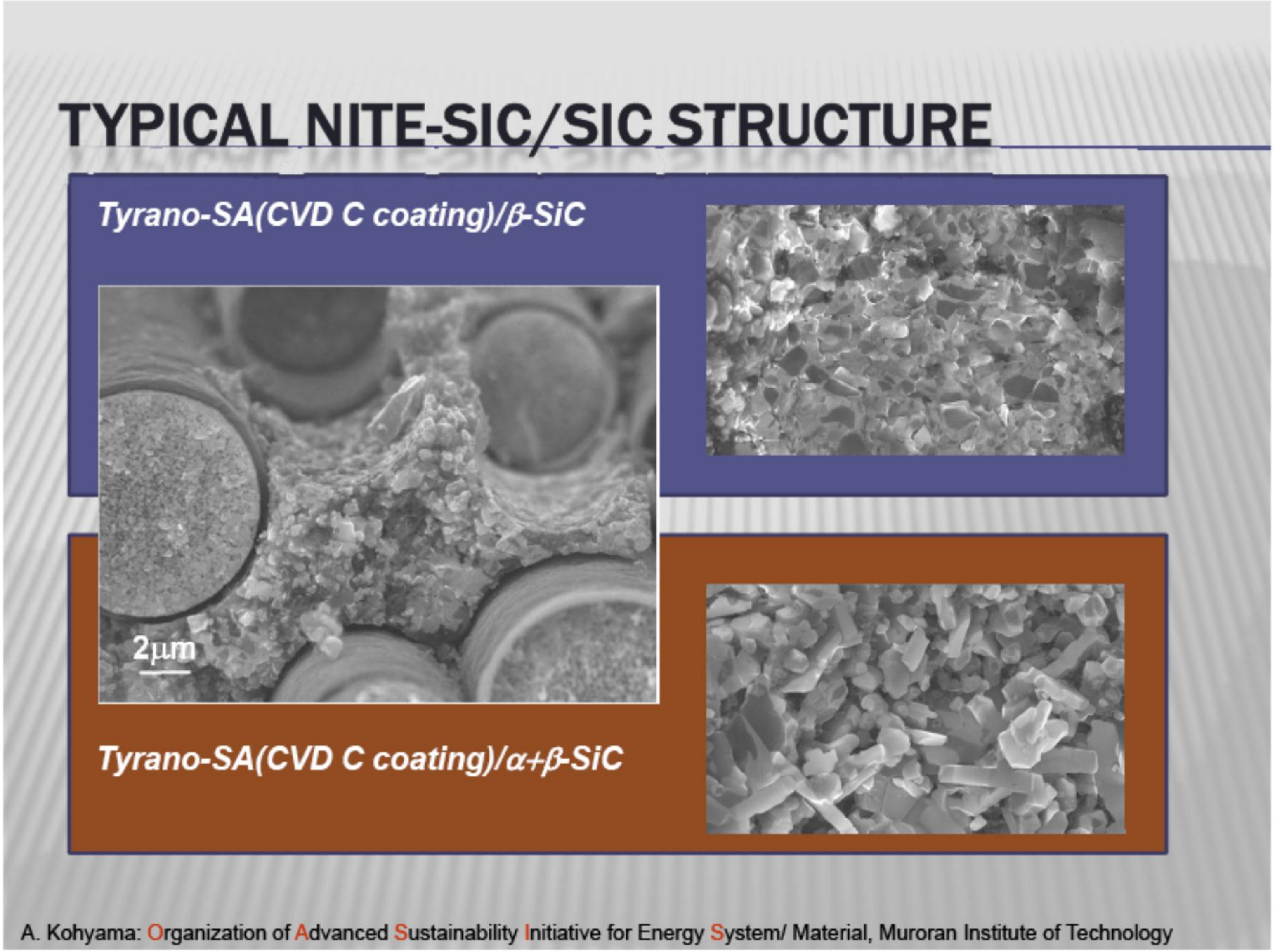
Assisted laser brazing of SiC/SiC minicomposites

Irradiation conditions :

Temperature ~ 900-950°C

1,7 dpa and 3,2 dpa

No dimensional change - Good behavior of the brazed joint


TYPICAL NITE-SIC/SIC STRUCTURE
Tyrano-SA(CVD C coating)/β-SiC
2μm
Tyrano-SA(CVD C coating)/α+β-SiC
A. Kohyama: Organization of Advanced Sustainability Initiative for Energy System/ Material, Muroran Institute of Technology

ñin-situò crystallized NITE SiC/SiC after heavy ion irradiation at DuET facility of Kyoto University *(courtesy of Prof. A. Kohyama Muroran Univ. Japan)*

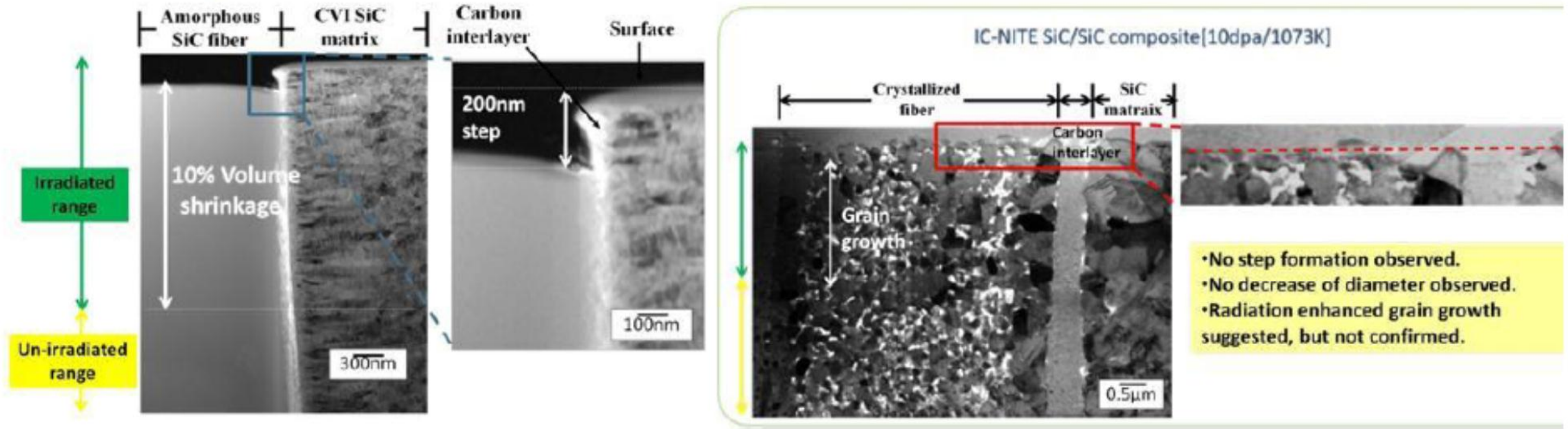


Fig. 5 (a) Radiation induced shrinkage of amorphous SiC fibers in CVI SiC matrix composite (10 dpa/873K)

Fig. 5 (b) Radiation damage in IC-NITE SiC (10dpa/1073K)

Amorphous SiC fiber: 200 nm step by the shrinkage of amorphous SiC fiber by radiation induced crystallization

ñin-situò crystallized NITE SiC/SiC:
no step formation at the surface, no decrease of fiber diameter observed.

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Reaction bonding (SiC+Si)	~200 MPa shear	Unstable due to residual silicon	NASA
Reaction bonding (Ti3SiC2/SiC)	50 MPa	Unknown	PNNL, Politecnico di Torino
Polymer joining	~10 MPa shear	Unstable due to residual oxygen and nano-crystalline phase	PNNL, etc.
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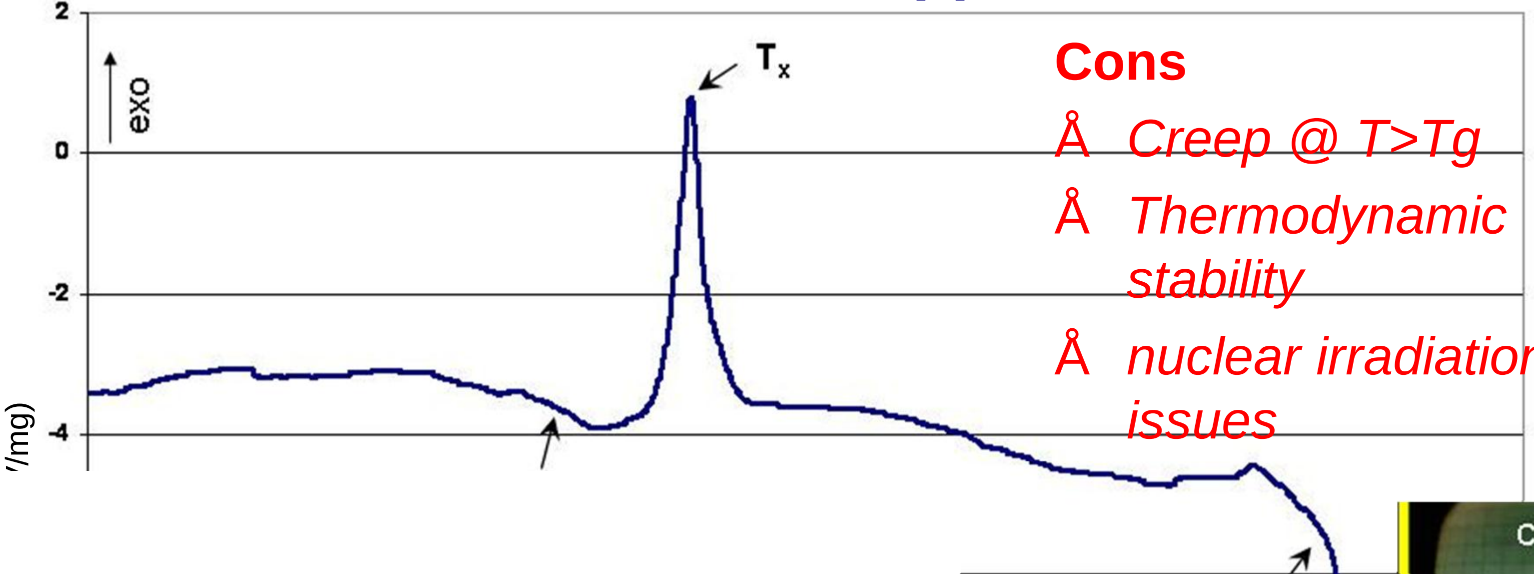
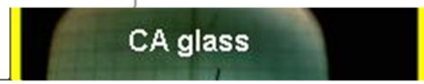
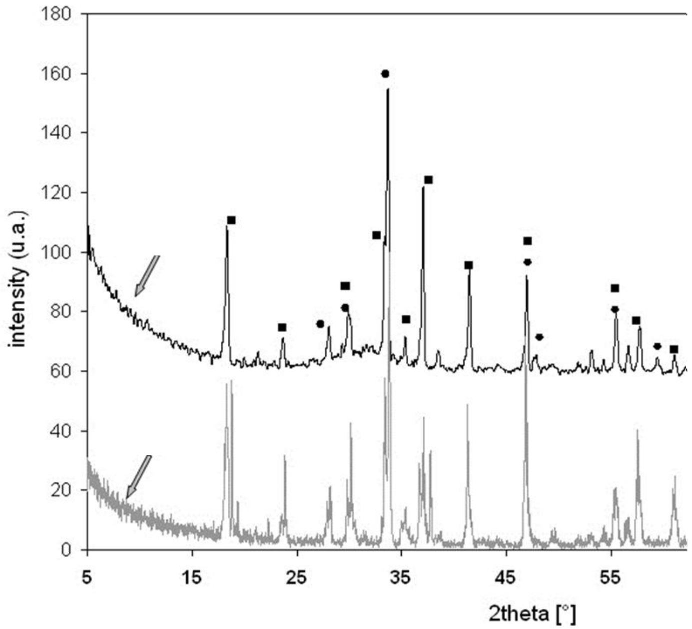
Glasses and glass-ceramics as joining materials for nuclear applications?

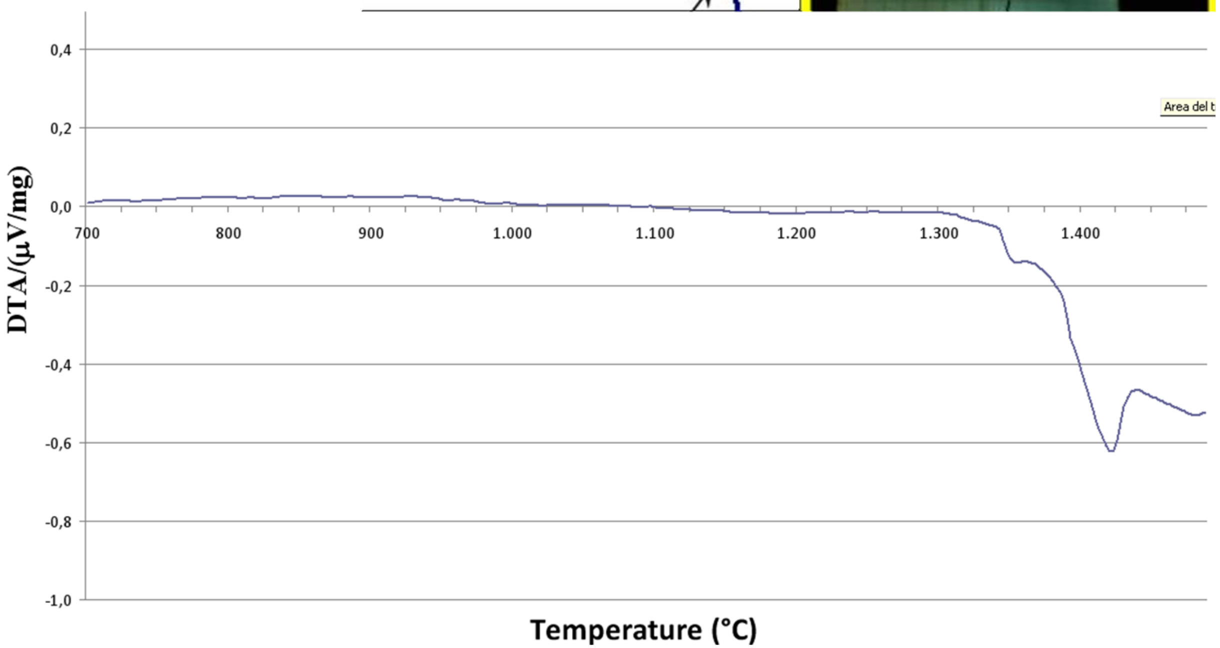


Cons

- *Creep @ T>Tg*
- *Thermodynamic stability*
- *nuclear irradiation issues*







Glasses and glass-ceramics as joining materials for nuclear applications?

2 M. Ferraris et al. / Journal of Nuclear Materials xxx (2010) xxx–xxx

Table 1

Composition, T_g and CTE of the parent glass, joining process temperature, time and applied pressure to obtain the glass–ceramic joining materials, CTE and softening temperature (T_{soft}) and crystalline phases of the glass–ceramic joining materials.

Refs.	Joining material	Composition (parent glass) (wt.%)	$T_g \pm 5$ (°C) (parent glass)	CTE[a] (parent glass) (10^{-6} °C^{-1})	Joining process	CTE[a] (glass–ceramic) (10^{-6} °C^{-1})	$T_{soft} \pm 10$(°C) (glass–ceramic)	Crystalline phases (glass–ceramic)
[12]	SAY	54 SiO_2, 18.07 Al_2O_3, 27.93 Y_2O_3	910	3.8	1375 °C, 20 min; 1235 °C, 1 h, heating rate 1000 °C/h, pressure-less	5.5	975	Mullite, cristobalite, keivyite
[13]	SAMg	60 SiO_2, 30 Al_2O_3, 10 MgO	867	3.1	1280 °C, 1 h, pressure-less	3	985	Cordierite, mullite
[6,11]	CA	49.7 CaO, 50.3 Al_2O_3	850	9.42[b]	1480 °C, 1 h, pressure-less	5.2	1380	3CaO Al_2O_3, 12CaO$_7$$Al_2O_3$
	SiC, SiO_2, Al_2O_3, Y_2O_3	88 SiC, 12 (SiO_2, Al_2O_3, Y_2O_3)	NA	NA	1400–1900 °C, 20 MPa	–	–	β-SiC, $Al_5Y_3O_{12}$

[a] CTE measured by TMA between 400 °C and 700 °C, heating rate of 20 °C/min.
[b] Calculated by Sciglass 6.6 (ScienceServe GmbH, Germany).

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Glasses and glass-ceramics as joining materials for nuclear applications?

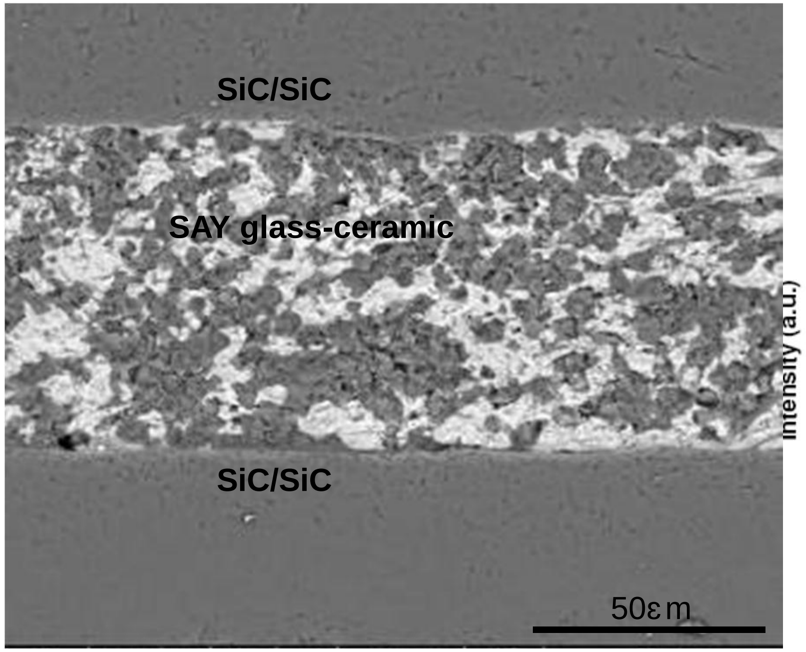
* neutron-induced radioactivity of elements present simulated by European Activation System EASY-2007 code package

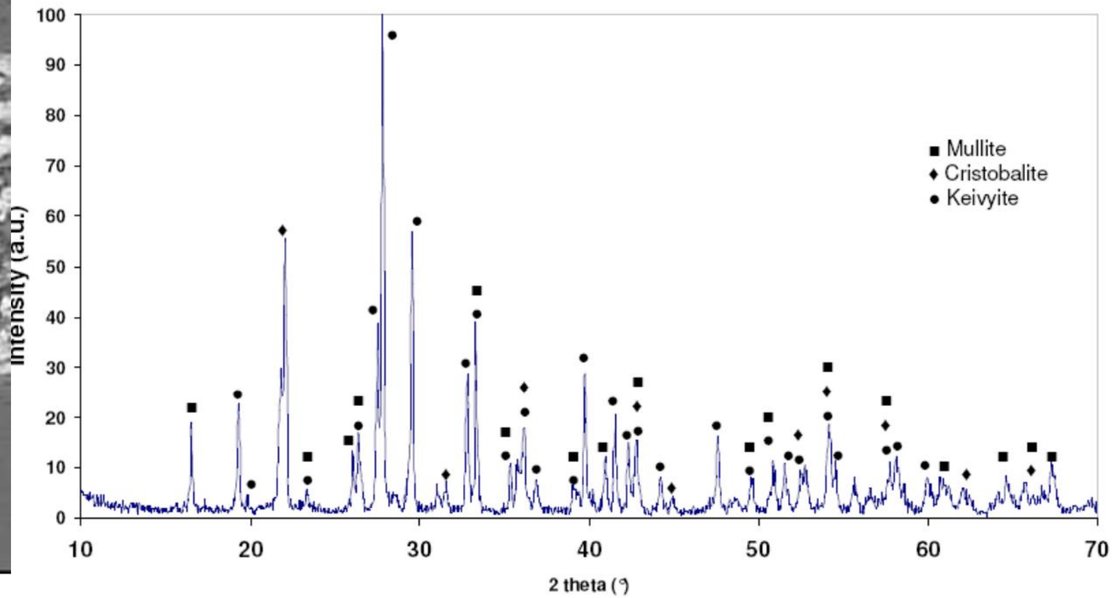
$SiO_2 - Al_2O_3 - Y_2O_3$

PRESSURE-LESS JOINING PROCESS: slurry and heat treatment

1375°C 20 min ; 1235°C 1 hour

Tx





CTE (400-700°C)= $5.49\ 10^{-6}\ °C^{-1}$

Figure 3. XRD measurement conducted on the SAY glass-ceramic after heat treatment at at 1375°C for 20 min and at 1235°C for 1 hour, heating rate of 1000°C/h, in Ar flow.

Glasses and glass-ceramics as joining materials for nuclear applications?

SiO2 - Al2O3 - Y2O3 Glass ceramic

1516 CARBON 47 (2009) 1511–1519

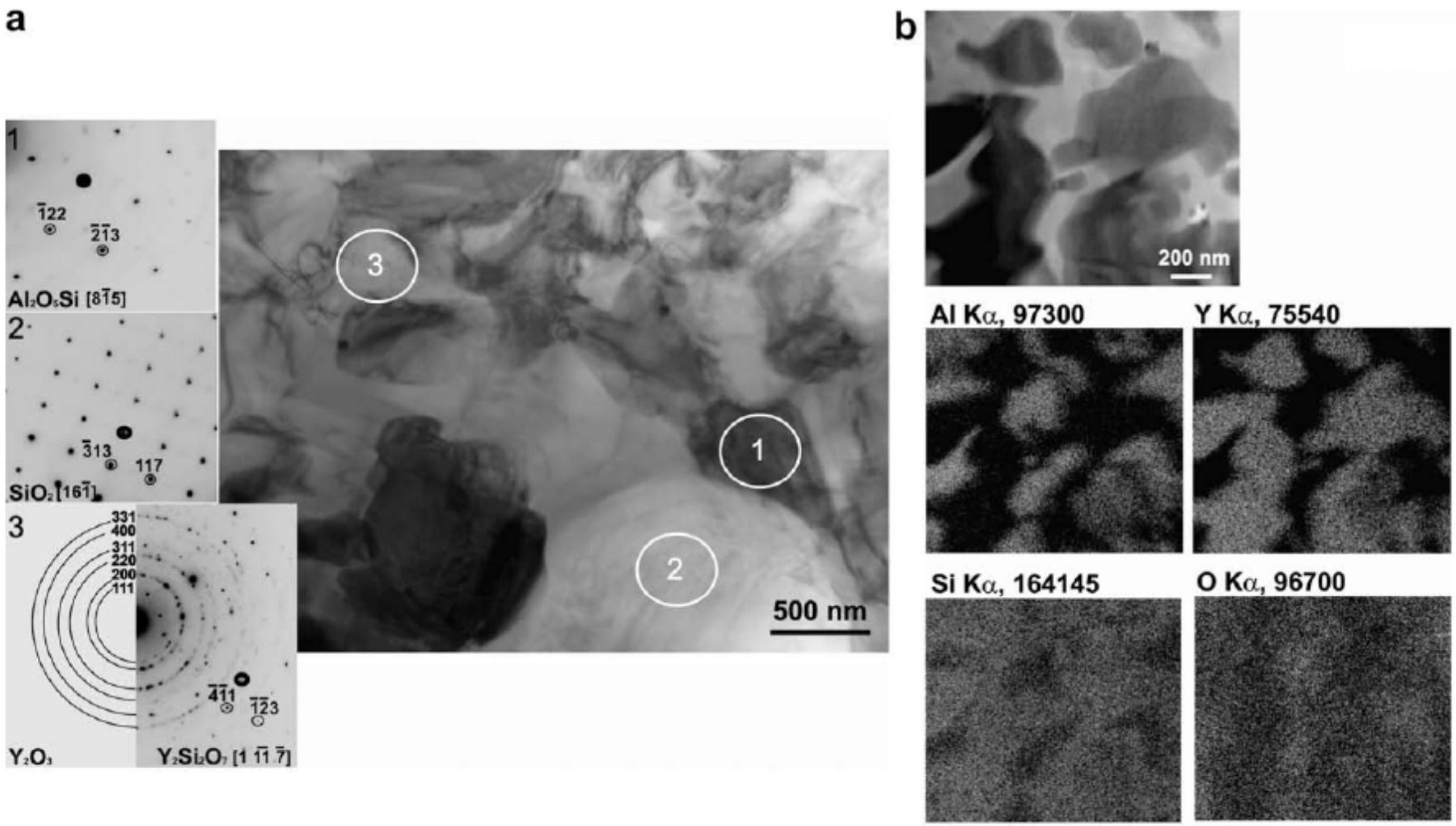


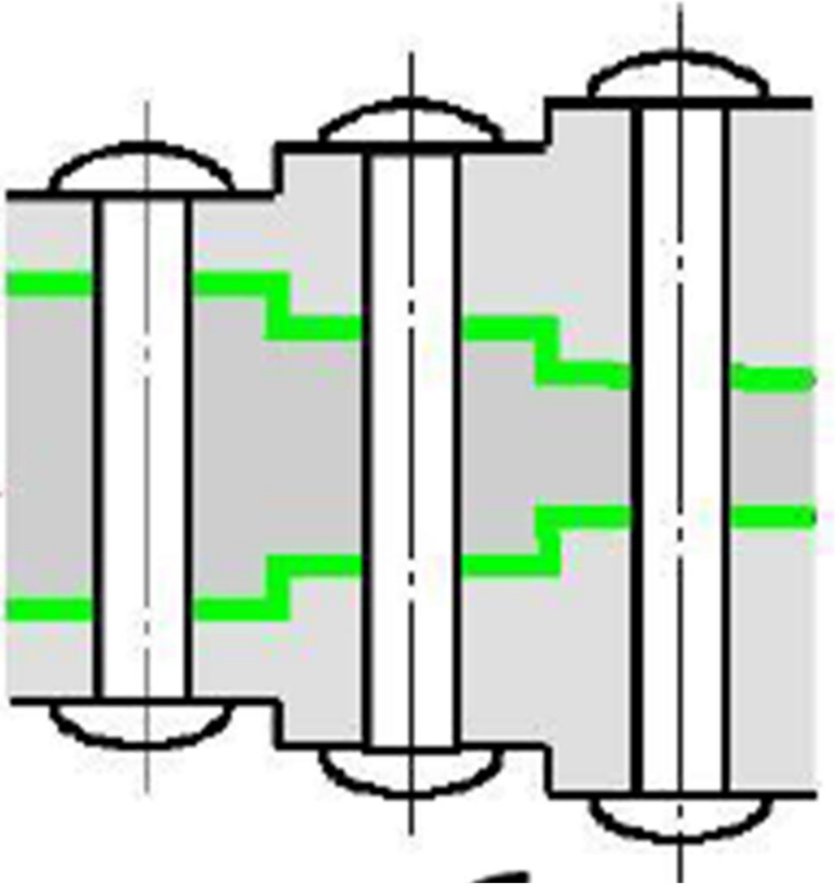
Fig. 8 – (a) Microstructure of the SAY glass-ceramic coating on LD-C/C substrate and SAED patterns taken from the coating and their identification; cross-section TEM image; (b) elemental distribution maps in the SAY glass-ceramic coating, STEM-EDS.

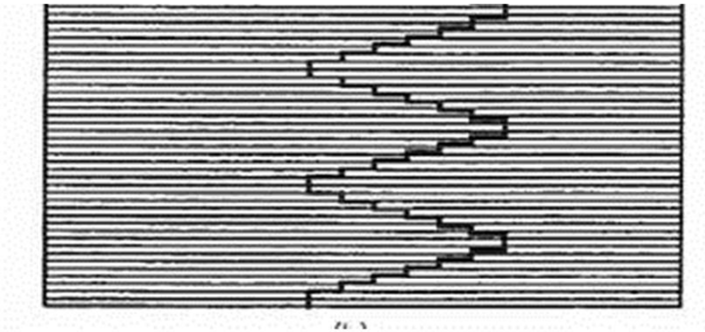
Joining not-flat surfaces

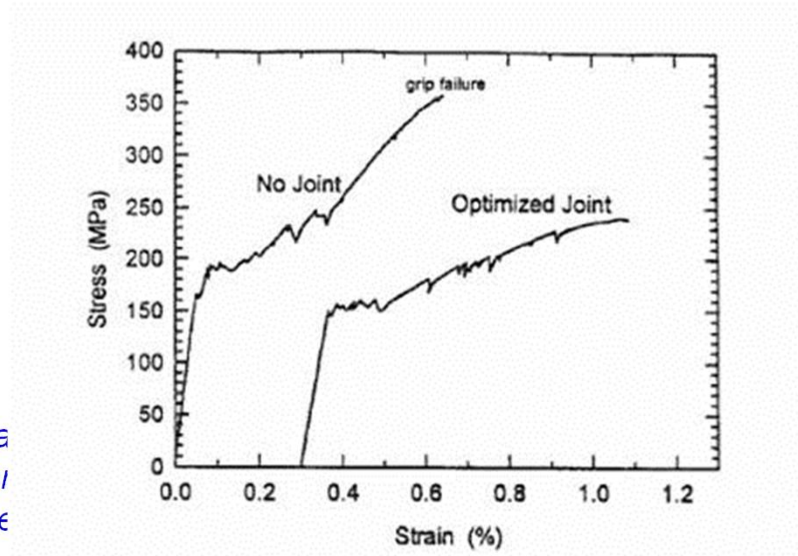
J. Am. Ceram. Soc., 81 [12] 3307-12 (1998)

3 —D FEM Stress Analysis and Strength Estimation of Stepped-lap Adhesive Joints of Dissimilar Adherends under Static Bending Moment

Toshiyuki SAWA (Hiroshima University, Japan) Kohei ICHIKAWA (University of Yamanashi, Japan)







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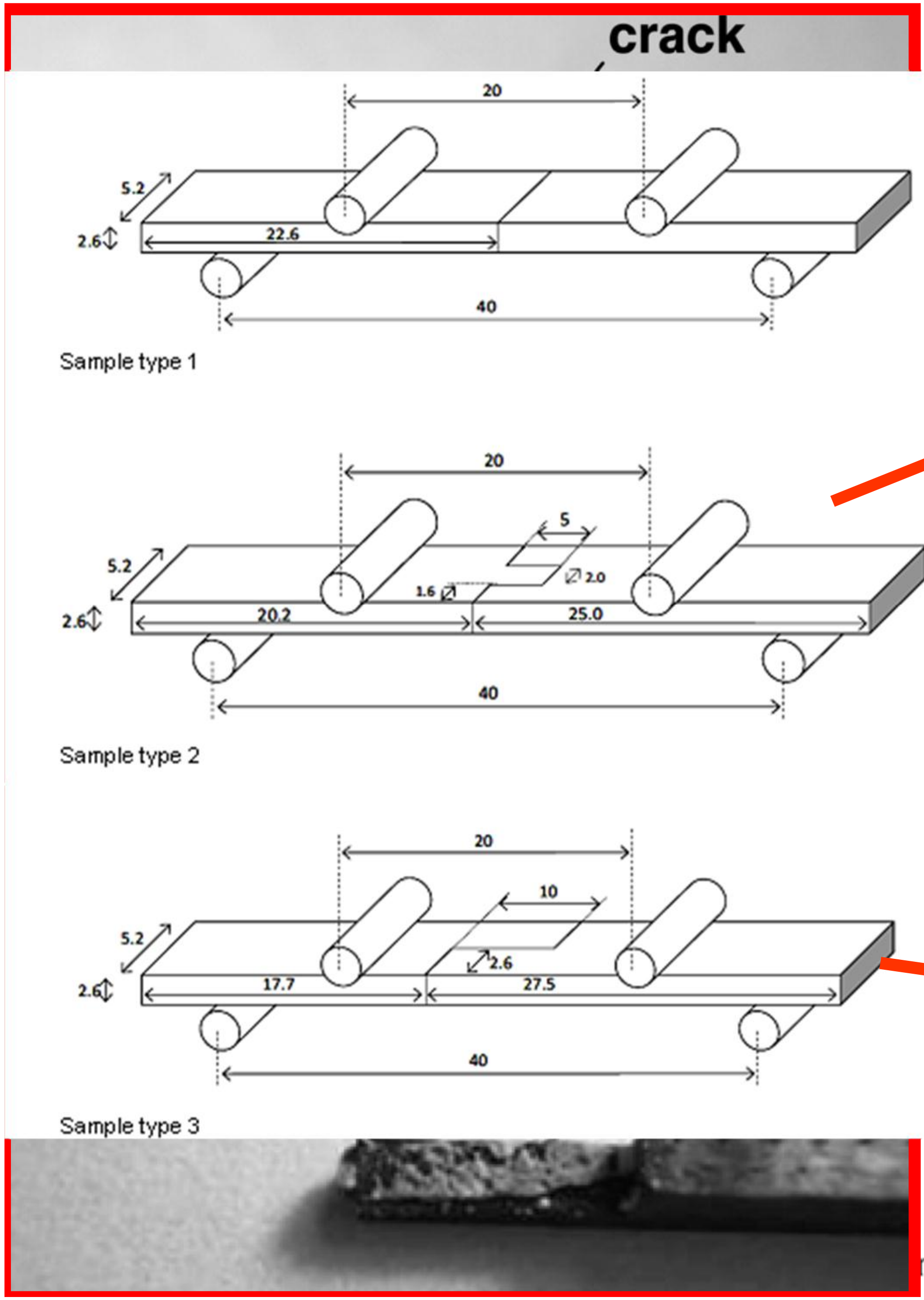
Joining not-flat surfaces



Average flexural strength [MPa]
24±2
122±10
149*
418±45
283±8

's for Fusion Applications

Doul Science

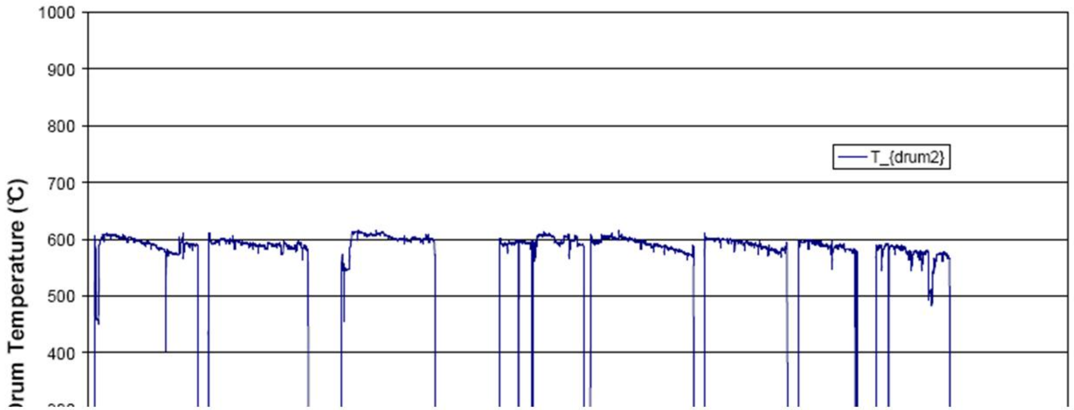
EU projects: Extremat and FEMaS

FEMaS-CA

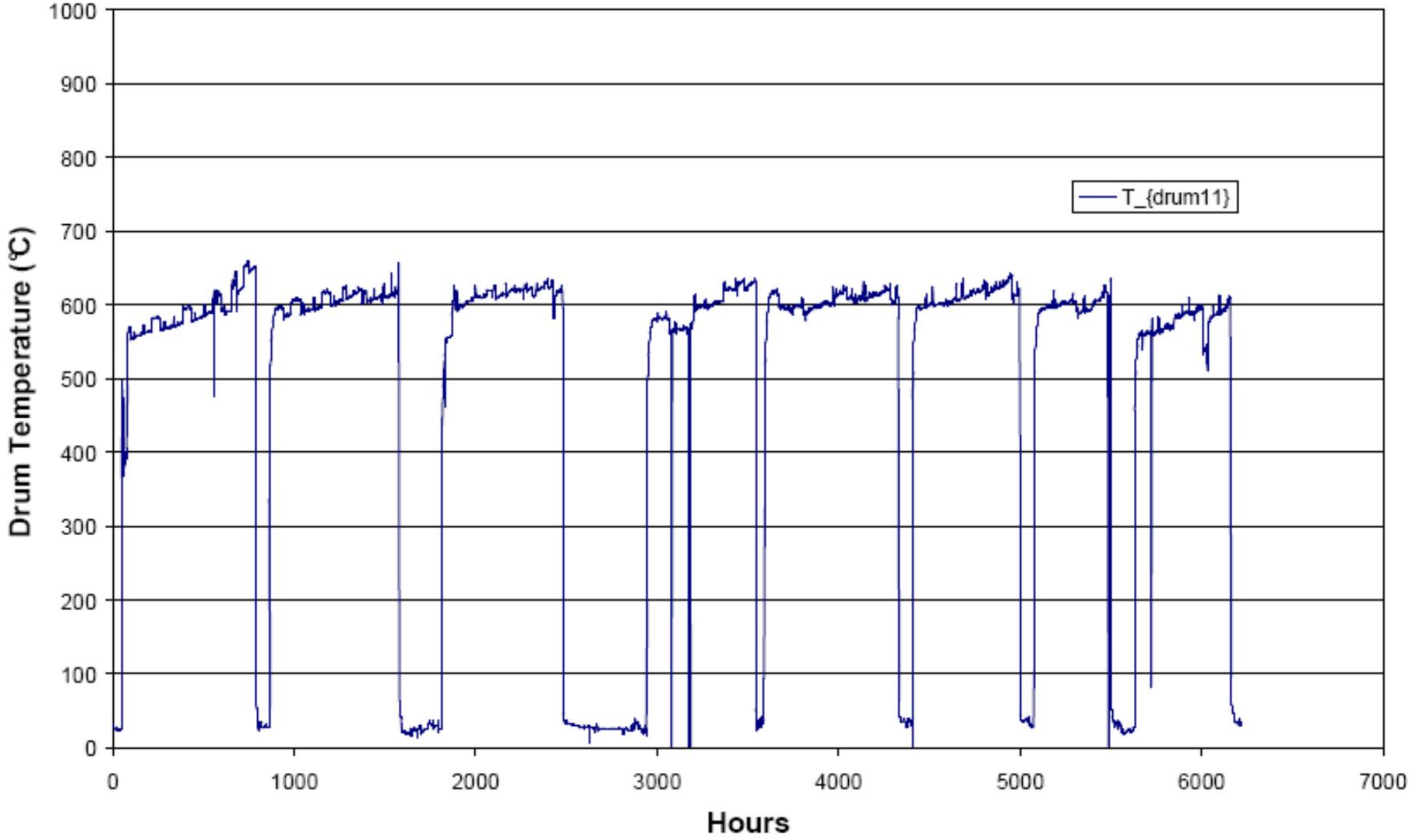
- Extremat I : 300 - 550°C
- Extremat II : 600 - 900°C

- Irradiation: High Flux Reactor (HFR) Petten, NRG

- Post irradiation tests (FEMaS): Hot Cell Laboratory (HCL), Petten, NRG



$16\text{-}22^{*}\ 10^{24}\ m^{-2}$



$.0^{24}\ m^{-2}$

m^{-2}

cations

ceramic
diation

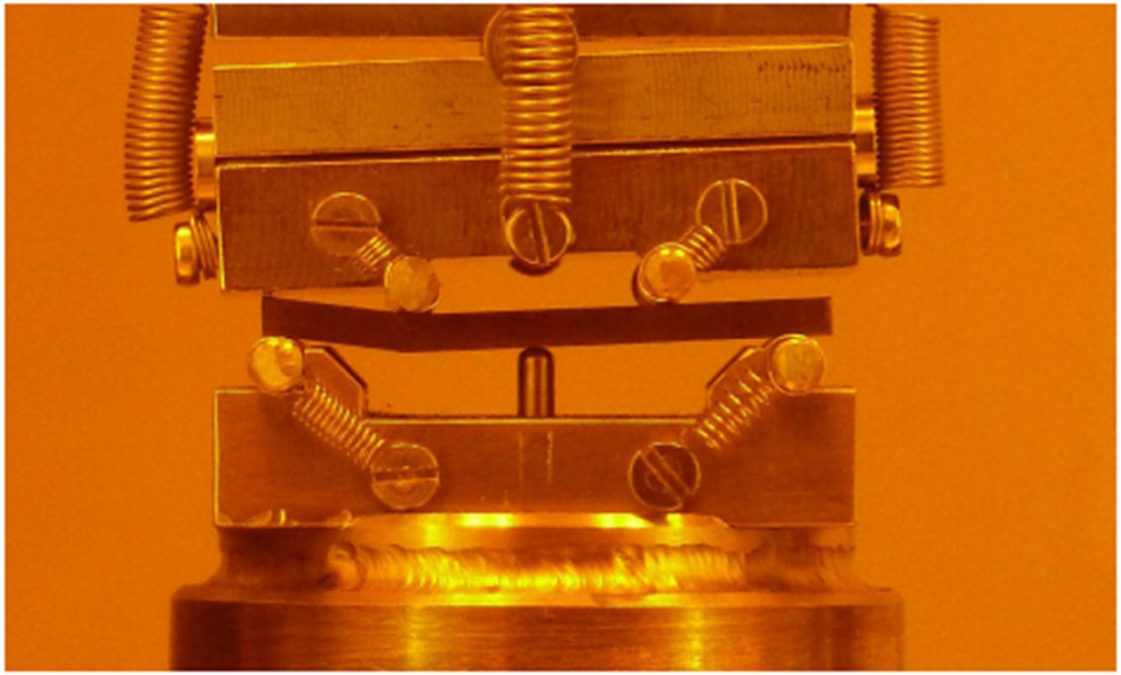


Figure 4.8 Mode S (shearing, Invalid result for bending strength)

Figu

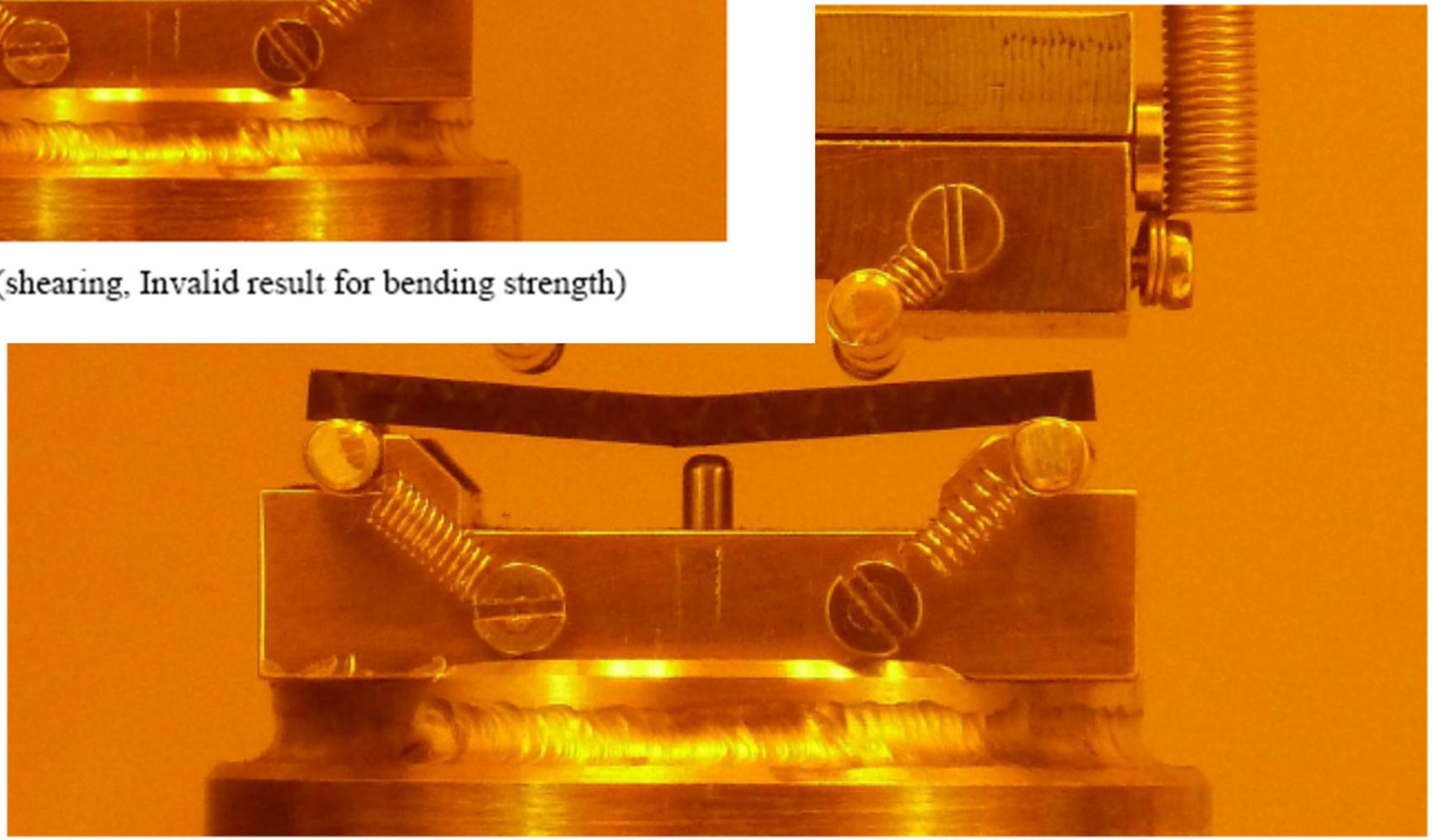


Figure 4.7 Mode T (tensile, Valid result for bending strength)

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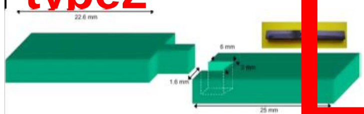
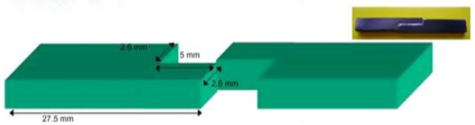
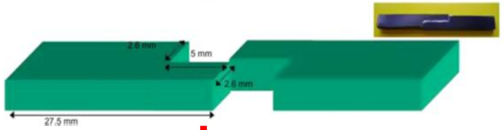
Fusion grade 2D SiC/SiC composite (MTA, Germany). Tyranno Type S, crystalline CVI SiC matrix, carbon interface. SiO_2-Al_2O_3-Y_2O_3 glass ceramic as joining material	Before irradiation bending strength, s (MPa)	After irradiation (NRG, Petten, NL) 550 °C 9-11 * 10 ^{24} m $^{-2}$	 600°C 16-22* 10 ^{24} m $^{-2}$	 820 °C 31-32* 10 ^{24} m $^{-2}$
As received SiC/SiC	418 ± 45	240-304	289	-
SiC/SiC (1420 °C, 30 min, Ar, 1240 1H)	283 ± 8	-	-	-
SiC/SiC type2 	122 ± 10	-	118	80-87
SiC/SiC type3 	149 ± ?	-	65	

Glass-ceramic as pressure-less joining material for nuclear applications

before irradiation

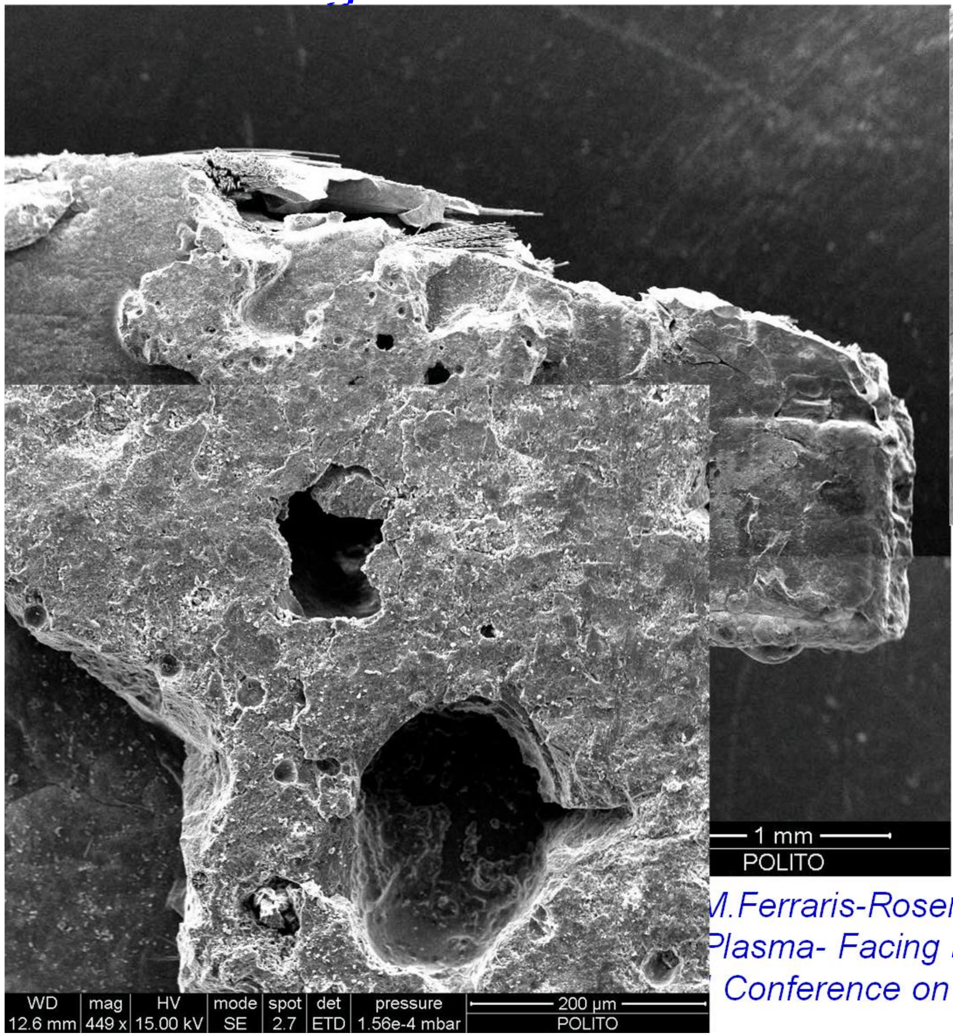
$\sigma_{bending}$ 149 MPa

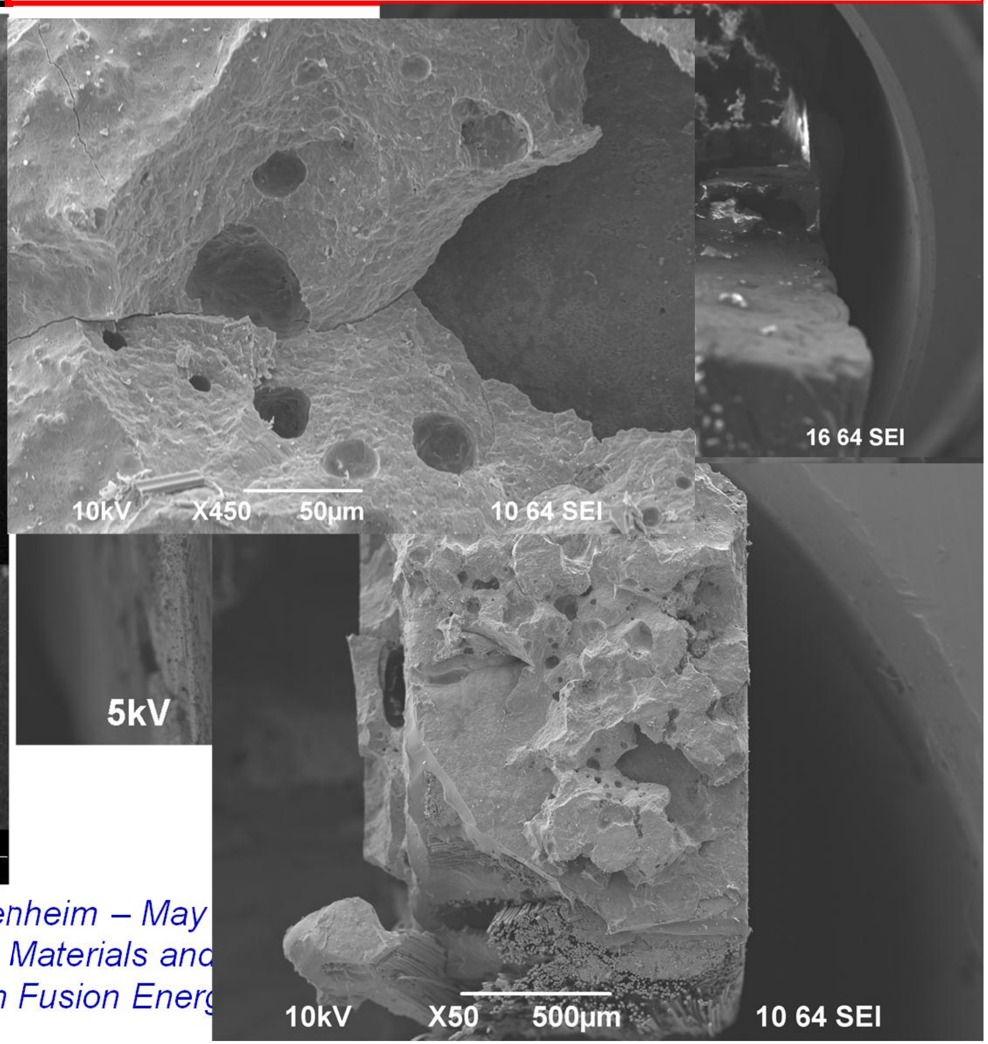


after irradiation 600 °C

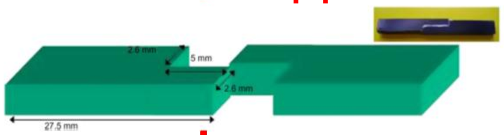
$\sigma_{bending}$ 65 MPa

Fluence 16.3 * 10 ^{24} m $^{-2}$





Glass-ceramic as pressure-less joining material for nuclear applications



before irradiation

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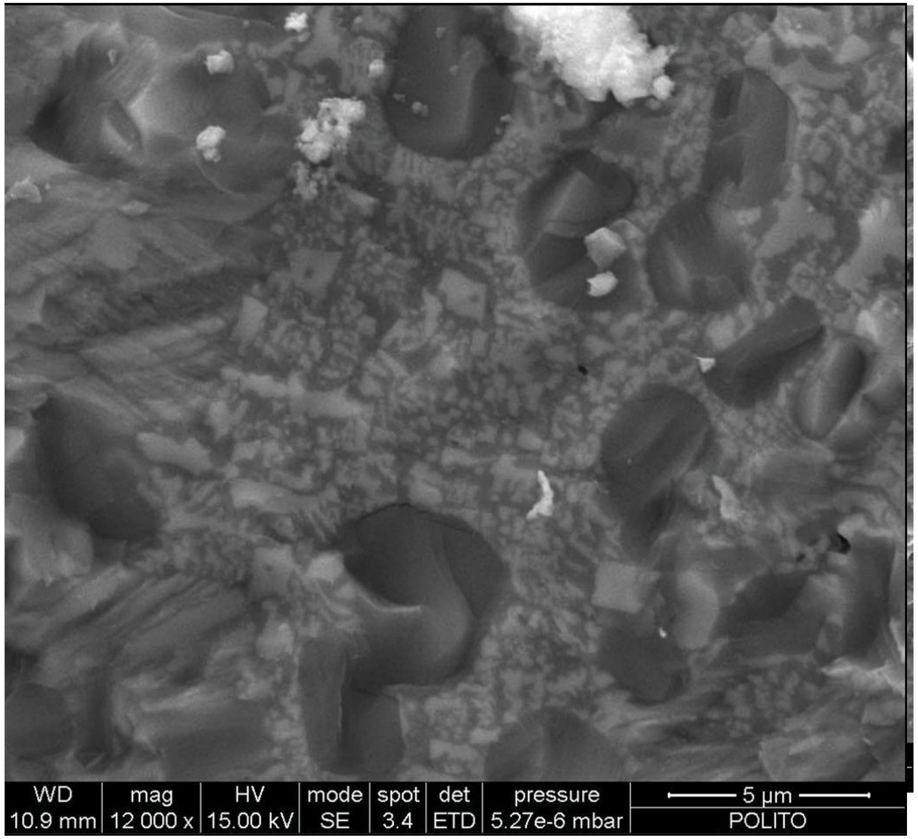
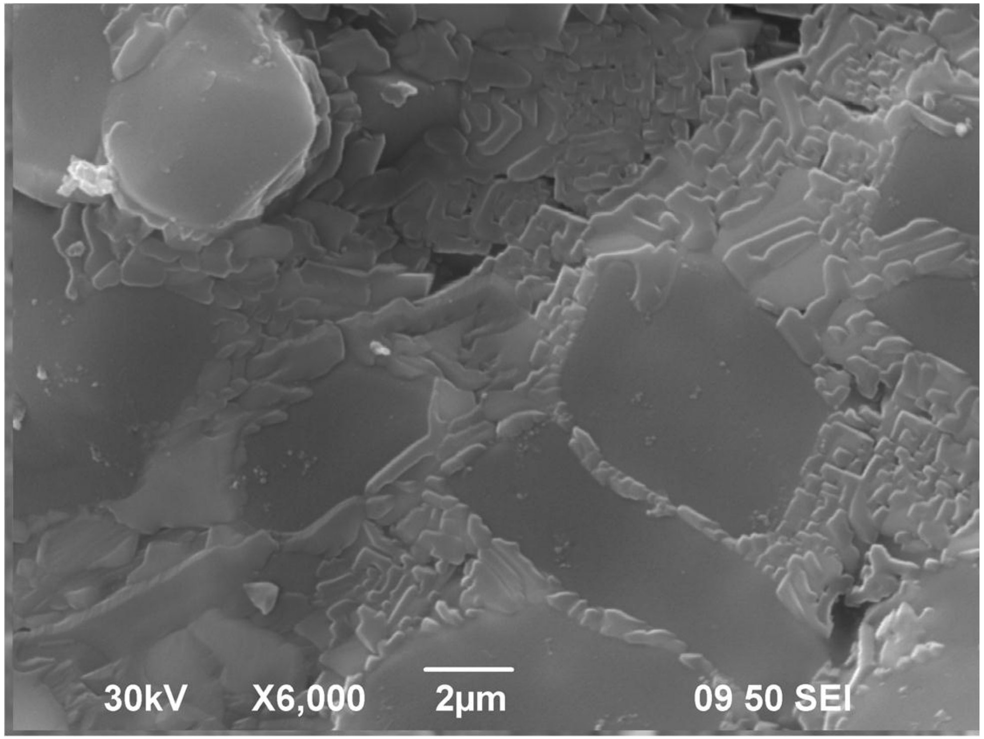
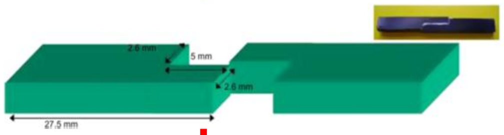
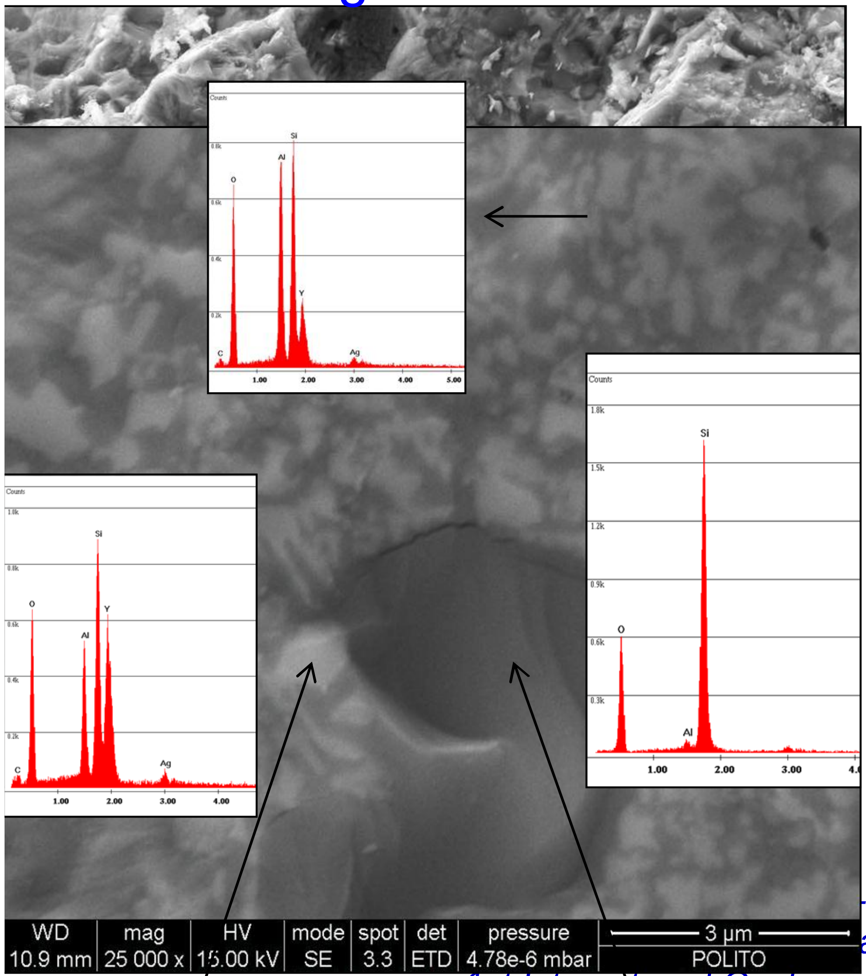


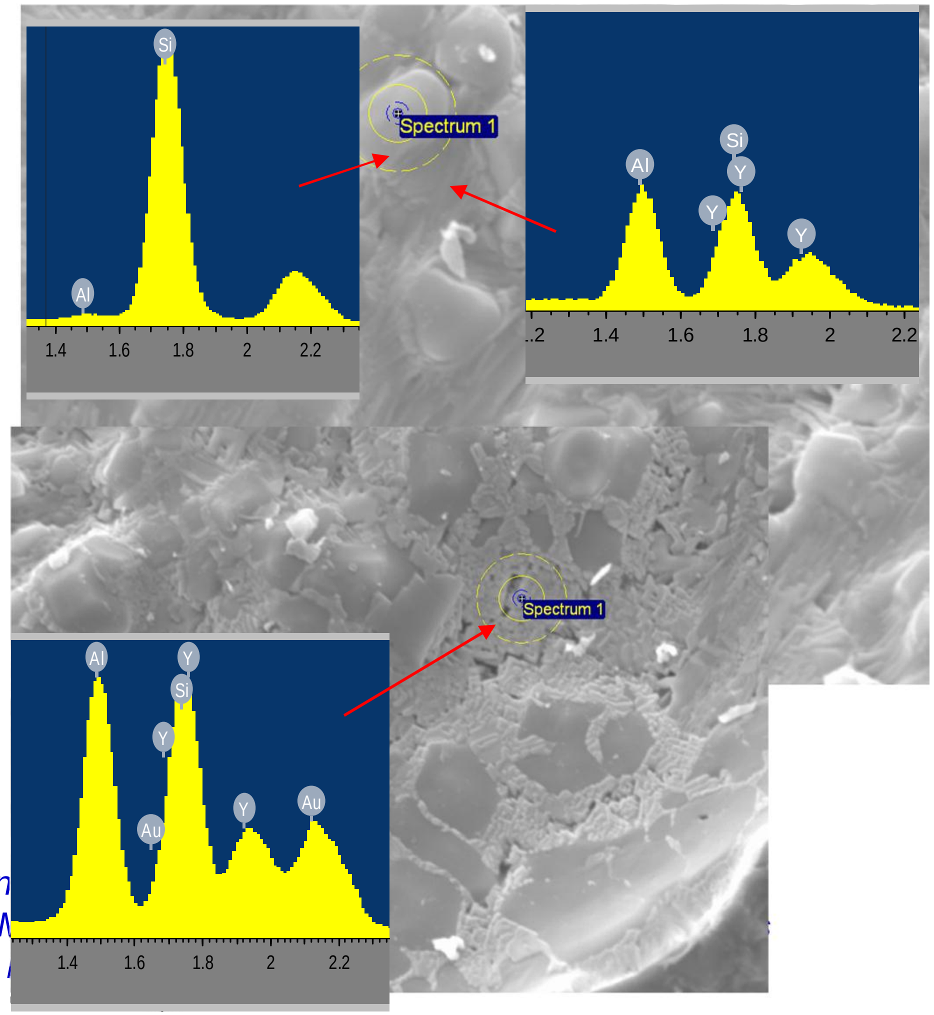


Glass-ceramic as pressure-less joining material for nuclear applications

before irradiation	after irradiation 600 °C
σ bending 149 MPa	σ bending 65 MPa
	Fluence 16.3 * 10 ^{24} m $^{-2}$







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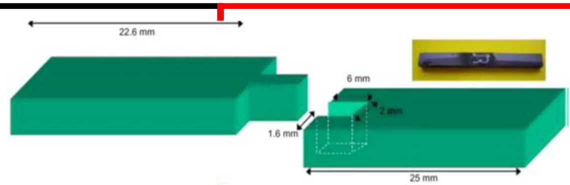
1st International Conference on

Glass-ceramic as pressure-less joining material for nuclear applications

before irradiation

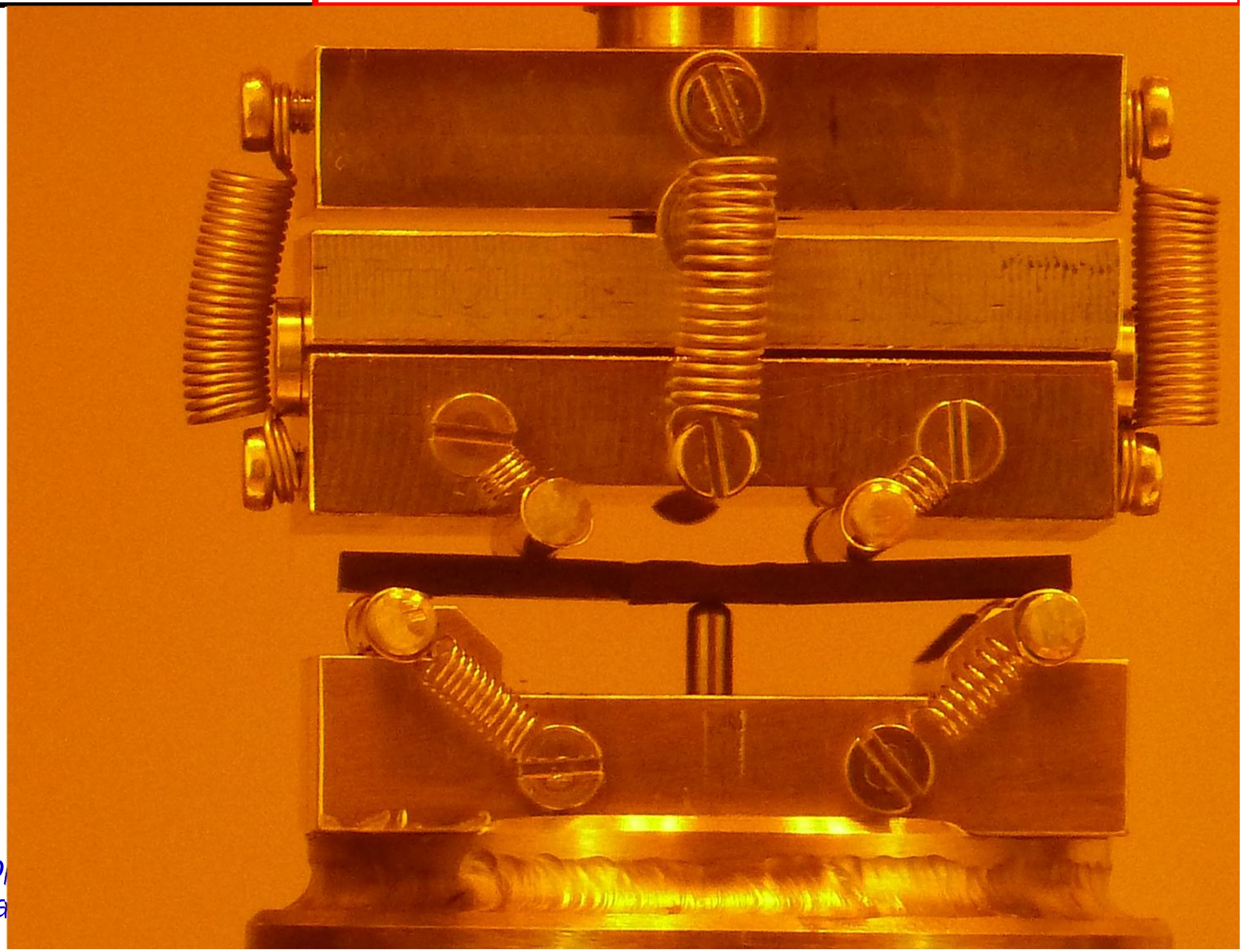
$\sigma_{bending}$ 122 MPa



after irradiation 600 °C

σ bending 118 MPa

Fluence 21.6 * 10 ^{24} m $^{-2}$



before irradiation

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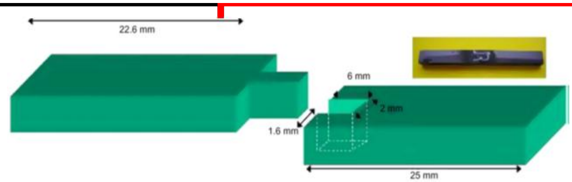

after irradiation 600 °C

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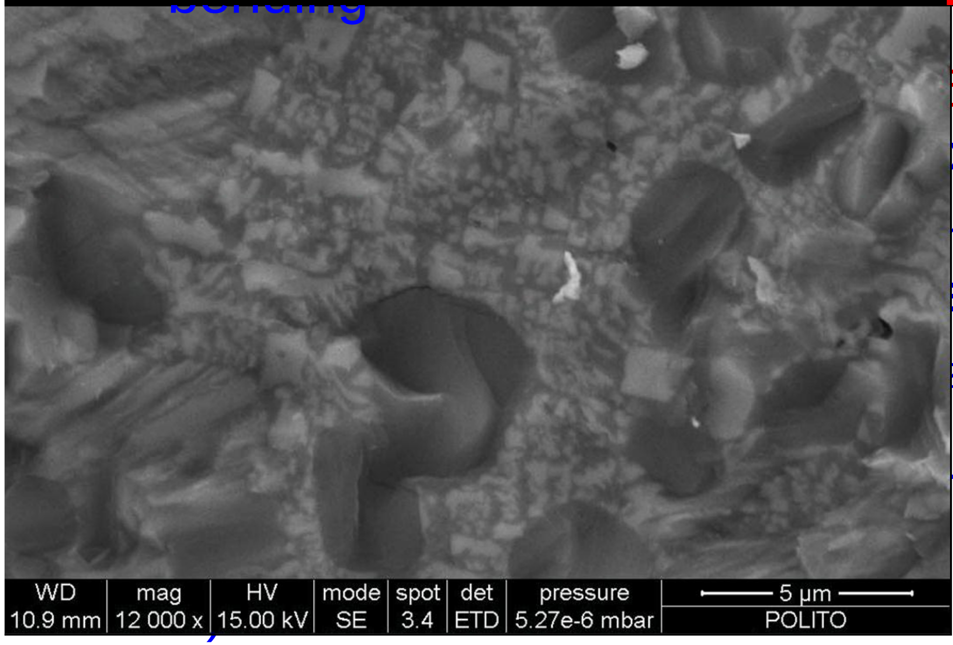

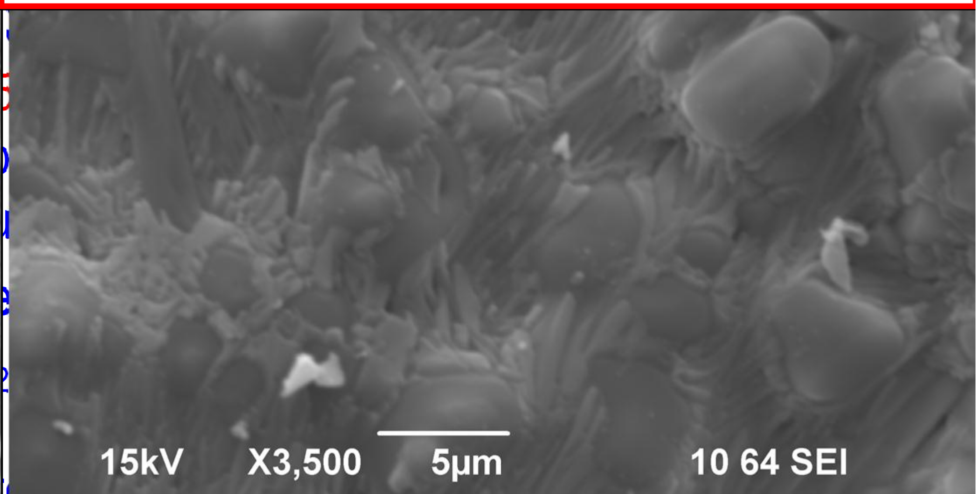

osilicate

0-1x10^{22}

SiC/SiC without using pressure

- Porter et al. 1981, (400-500 °C, fluence=2.4x10^{22} n/m^2) eight **glass-ceramics containg** $KMg_3AlSi_3O_{10}F_2$ or $Li_2Si_2O_5$ in a glass matrix
- Coghlan et al. 1991, **MACOR**)(room T, fluence=1x10^{23} n/m^2): expansion of mica phase and contraction of glass matrix

SiO2 - Al2O3 - Y2O3;

SiO2 - Al2O3 - MgO ;

CaO - Al2O3

Thanks !

monica.ferraris@polito.it

http://www.composites.polito.it

M. Salvo,
V. Casalegno,
S. Han,
S. Rizzo,
A. Ventrella,
F. Smeacetto,
& students

P. van Idsert, T. van Staveren (NRG)

Table 4.3 Dose levels and irradiation temperatures Extremat II, sample holder 362-01

specimen drum	distance w.r.t. C_L spec. hold. [in mm]	'thermal' fluence Φ_{Co} [$10^{24}m^{-2}$]	fluence $\Phi_{E>1.0\,MeV}$ [$10^{24}m^{-2}$]	N_{dpa} 1) (C)	N_{dpa} 2) (Cu)	N_{dpa} 3) (W)	N_{dpa} 4) (steel)	specimen 4) temperature median/max. [°C]
TOP								
11	+232.0	7.56	16.3	2.57	3.00	0.623	2.36	**596/659**
10	+180.0	11.0	22.4	3.50	4.09	0.850	3.22	**610/669**
09	+135.0	13.3	26.5	4.12	4.82	1.000	3.81	**549/604**
08	+83.0	15.0	29.9	4.63	5.43	1.130	4.28	**753/902***
07	+31.0	15.8	31.8	4.92	5.77	1.200	4.55	**868/926***
06	-10.0	15.8	32.3	4.99	5.86	1.220	4.62	**816/883***
05	-58.0	15.0	31.6	4.90	5.75	1.190	4.53	**821/884***
04	-95.0	13.8	30.3	4.69	5.51	1.150	4.35	**840/899**
03	-153.0	11.1	26.6	4.15	4.87	1.010	3.84	**892/942**
02	-207.0	7.44	21.6	3.39	3.99	0.829	3.14	**589/617**
01	-245.0	4.29	17.1	2.71	3.19	0.664	2.51	**601/632**
BOTTOM								

* Irradiation temperatures have not been constant throughout the irradiation experiment for these drums, s

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Table 4.1 Neutron displacement damage in sample holder 363-01 (EXTREMAT-01)

specimen drum	distance w.r.t. C_L spec. hold. [in mm]	'thermal' fluence Φ_{Co} [$10^{24}m^{-2}$]	fluence $\Phi_{E>1.0\ MeV}$ [$10^{24}m^{-2}$]	N_{dpa} [1] (C)	N_{dpa} [2] (Cu)	N_{dpa} [3] (W)	N_{dpa} [4] Steel
TOP							
10	+234.0	4.33	7.27	1.13	1.33	0.278	0.95
09	+184.0	5.63	8.88	1.40	1.65	0.344	1.18
08	+131.0	6.63	10.2	1.62	1.91	0.397	1.36
07	+79.0	7.22	11.0	1.77	2.07	0.432	1.48
06	+28.0	7.42	11.4	1.85	2.16	0.449	1.54
05	-22.0	7.26	11.3	1.87	2.17	0.451	1.55
04	-71.0	6.77	10.9	1.83	2.11	0.437	1.51
HEAT SHIELD							
03	-137.0	5.56	9.75	1.69	1.92	0.395	1.37
02	-188.0	4.20	8.37	1.51	1.68	0.343	1.20
01	-220.0	3.17	7.29	1.37	1.49	0.303	1.06
BOTTOM							

[1] *number of displacements (N_{dpa}) produced in reference material Carbon (C)*

[2] *number of displacements (N_{dpa}) produced in reference material Copper (Cu)*

[3] *number of displacements (N_{dpa}) produced in reference material Tungsten (W)*

[4] *number of displacements (N_{dpa}) calculated by dividing the (Cu) dpa by 1.40*

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The average and median irradiation temperatures for the various drums are given in Table 4.2.

Table 4.2 Irradiation temperatures in sample holder 363-01 (EXTREMAT-01)

specimen drum	distance w.r.t. C_L spec. hold. [in mm]	average specimen temperature [°C]	specimen temperature median/max. [°C]
TOP			
10	+234.0	**552**	**552 / 583**
09	+184.0	**543**	**542 / 585**
08	+131.0	**553**	**552 / 579**
07	+79.0	**552**	**553 / 596**
06	+28.0	**548**	**548 / 582**
05	-22.0	**543**	**542 / 588**
04	-71.0	**554**	**554 / 600**
HEAT SHIELD-----			
03	-137.0	**285**	**286 / 306**
02	-188.0	**283**	**283 / 298**
01	-220.0	**298**	**298 / 322**
BOTTOM			