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Glass as a joining material for ceramic matrix composites: 25 years of research at Politecnico di Torino

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Abstract

A revision and update about the research activity done in the past 25 years at Politecnico di Torino (Italy) on design and testing of glasses and glass-ceramics for the joining of Ceramic Matrix Composites (CMC) will be presented and discussed. The “glass-ceramic method” to obtain a thermo-mechanically stable joint starting from a glass will be detailed with some examples. Several new glass compositions will be presented together with their characterization and behavior as joining materials for oxide-based and non-oxide based CMC.

Keywords: glass-ceramics, joining, CMC

Introduction

The Ceramic Matrix Composites (CMC) considered in this paper are long fiber reinforced non-oxide based composites, such as carbon fiber- or silicon carbide fiber- reinforced carbon matrices or silicon carbide matrices (C/C, C/SiC, SiC/SiC), and oxide based composites, such as alumina fiber reinforced oxide matrices ($Al_2O_3/Al_2O_3-ZrO_2$, Al_2O_3/Al_2O_3), usually defined as Ox/Ox or Ox-CMC.

Due to their excellent thermo-mechanical properties and low density, CMCs are considered the most suitable candidate to gradually replace metallic alloys in a wide range of applications [1]. However, CMCs present two main problems; since they do not melt, they cannot be welded by ordinary methods; in addition, the mechanical joints produced using ceramic rivets, bolts, etc. are not always tight. Reliable joining of CMCs is considered a key technology to enable their use in several applications characterized by harsh environmental conditions, such as energy production, aeronautics, aerospace, etc. where large and complex geometries are needed [2].

Ideally, CMCs should be joined by localized heating of the area to be joined and no pressure should be necessary to obtain the joint. The characteristics which the joining material should have are: tightness, resistance to oxidation, tailorabity in terms of thermo-mechanical and physical properties such as the coefficient of thermal expansion (CTE), wettability toward CMC, thermal stability, etc.

Glasses and glass ceramics can fulfil most of the requirements listed above to join CMCs to themselves and to other materials. The “glass-ceramic method” is unique to materials science [3] and with this method glasses can be used as pressure-less “*adhesives*” for high temperature applications. The glasses are first softened, and then crystallized to obtain stable and thermo-mechanically resistant glass-ceramic joints. Due to their high viscous flow rates, glass-ceramics can successfully be used as joining materials for CMCs. They can be processed through sintering and crystallization of fine powdered glasses. It is possible to develop advanced glass-ceramics materials with unique features, thanks to the great versatility of parent glass compositions and different sinter-crystallization processes. The development of glass-ceramic joining materials finally demonstrates the advantage of combining various extraordinary properties that can be resumed and found in one material.

When considering the whole process, sintering and crystallization processes can interact, thus affecting the final density of glass-ceramics materials. The precise order of events in the sinter-crystallization process is a critical step which enables the formation of dense glass-ceramic joining materials, with desired thermomechanical properties and microstructures. In addition, glasses and glass-ceramics are “self-healing” joining materials, i.e. they can be heated above their softening temperature to heal cracks accidentally formed in working conditions [4]; last but not least, in case of nuclear energy production, glasses and glass ceramic joints can be designed to be neutron and low-activation resistant, in order to maintain their characteristics in working conditions and to allow repairing and maintenance after use in a reactor [5, 6]. Finally, the chemical composition and crystalline phases in a glass ceramic can be tailored in order to achieve a good thermo-mechanical and thermo-chemical compatibility with the materials to be joined. In particular, the coefficient of thermal expansion (CTE) must be as close as possible to that of the materials to be joined, thus avoiding residual stresses at the joint interface.

The aim of this work is to revise and update the research activity done in the past 25 years at the Politecnico di Torino on glass and glass-ceramic joining of oxide and non-oxide CMC for several applications ranging from aerospace to energy conversion. The overview presented in this paper provides insights for the use of glass-ceramics, thanks to their versatility in compositions, properties and microstructures, as joining material for ceramic matrix composites.

Experimental procedure

The glasses used as joining materials have been designed by the authors: the composition range was selected by using Sciglass 6.6 software database. The main criterion was the CTE compatibility with CMC to be joined. All glasses ("*parent glasses*") were prepared by weighting and mixing the starting products (oxides or carbonates) according to the weight or molar percentage as in Table I and Table II. The powders were carefully mixed to guarantee homogeneity, then heated in chamber furnaces in air, in platinum crucibles. The parent glasses were obtained by melt-quenching and pouring on a suitable substrate or drilled from the crucible when the melt viscosity was too high. They were then grinded and sieved to less than 40 microns and analyzed by DSC (DSC 404 F3 Pegasus®, Netzsch, Germany) DTA (DTA 404PC, Netzsch, Germany), Heating Stage Microscopy (HSM, MISURA HSML 1600-3002, Expert system solution, Italy), XRD (XRD, X'Pert Pro MRD Panalytical, Cu Kα radiation, with the aid of the X-Pert HighScore software and identified with JCPDSfiles) and dilatometry (dilatometer DIL402PC, Netzsch, Germany) to measure their characteristic temperatures and CTE; all the analyses were carried out on parent glasses and on glass-ceramics deriving from parent glass after crystallization.

Parent glass powders were mixed with isopropyl alcohol to obtain slurries to apply with a spatula between the two CMC parts to be joined. The joining of Ox-CMC was done by heating the CMC-slurry-CMC sandwich in furnaces in air, while an Ar flow was necessary to join non-oxide CMC, in order to avoid their oxidation.

Some joined samples were then cross sectioned and prepared for SEM and TEM investigation, while others were tested mechanically at room or at working temperatures.

Glass-ceramics for nuclear applications were also characterized under neutron and ion irradiation. Low fluence fission neutron irradiation (irradiation for approximately 1 year in the reactor core of the LVR-15 at Nuclear Research Institute Rez, Czech Republic and at High Flux Reactor (HFR), Petten, The Netherlands) on two glass-ceramics (referred to SAY and SAMg in Table II) and on joined samples was performed. The joints obtained with the glass-ceramic referred to CA in Table II were irradiated in the Oak Ridge National Laboratory High Flux Isotope Reactor at 500°C (~ 3 dpa) and 800°C (~ 5 dpa). The same glass-ceramic was irradiated at room temperature with 5.5 MeV $4He^+$ and with 5.1 MeV Si^{2+} ions to 3.3×10^{20} ions/m^2 at temperatures of 400 and 800°C with the aim of reproducing a damage scenario similar to that in nuclear plants.

In the case of one glass-ceramic for nuclear applications (referred to as SAY in Table II), two SiC/SiC tubes were joined and the joined volume analyzed by CT-scan (at Oxford University, UK); the glass-ceramic behavior in working conditions (autoclave, PWR water chemistry 330°C, 18 MPa, at SCK-CEN, Leuven, Belgium) was also investigated.

Results and discussion

Table I is a summary of some glasses initially designed and tested as joining materials for SiC/SiC and C/C composites: the first three (SB, BAS and SABB) are borosilicate based compositions selected because of their low CTE, ranging between 3.5 and 4.5×10^{-6} °C^{-1} and compatible with those of SiC/SiC (4×10^{-6} °C^{-1}) and C/C (2.5×10^{-6} °C^{-1}). All of them gave sound interfaces and good wettability on SiC-based CMC, while they were unsuitable to join C/C. This behavior will be discussed in the following section. The last one (ZBM) was designed to lower the joining process temperature, while keeping CTE compatible for CMC to be joined: both the multicomponent ZnO-rich ZBM glass and the BaO-Al_2O_3 borosilicate (SABB) were successfully used to join SiC/SiC.

These very preliminary results showed that glasses of suitable composition can be used as “adhesives” for high temperature applications: their wettability on SiC/SiC was excellent, while every attempt to use them to join C/C failed, due to the completely different reactivity of oxide glasses towards SiC and C. In order to overcome this problem, the C/C surface was subsequently modified by the formation of a β-SiC layer [14].

Five other successful glass compositions were then designed and tested for the joining of SiC/SiC and Ox/Ox composites: Table II summarizes composition, characteristic temperatures, CTE, crystalline phases and joining process temperature for parent glasses and their derived glass-ceramics. The parent glass properties, such as softening and viscosity decrease vs temperature, were exploited to join CMC by a pressure-less process, followed by the parent glass crystallization to obtain a glass-ceramic joint, with none or negligible residual amorphous phase. The parent glass crystallization was done directly by cooling down the joined CMC or by a suitable thermal treatment following the joining process; this way was used for SAY joints, where 20 minutes at 1375 °C were sufficient for the pressure-less joining process, followed by a crystallization process at 1235 °C, one hour. This method is known as the “glass-ceramic method”, and it was fully exploited with these five compositions to join oxide and non-oxide CMC. Figure 1 shows how it works: curve (a) is the differential thermal analysis of the SiO_2-Al_2O_3-Y_2O_3 (SAY) parent glass, showing its glass transition, crystallization and melting temperatures; curve (b) refers to the SAY glass–ceramic obtained by heat treatment of the SAY parent glass at 1375 °C for 20 min and at 1235 °C for 1 h (the same thermal treatment used during the joining process). The absence of crystallization peaks in curve b is an evidence of the almost complete crystallization of the SAY parent glass during the joining process.

Figure 2 (a) shows the presence of three crystalline phases ($Y_2Si_2O_7$, Al_2SiO_5 and SiO_2) in the SAY glass-ceramic, as detected by XRD (data not reported here), but not detectable in the DTA curve in Figure 1 (a), where only one broad crystallization peak is visible. An interesting (and unexpected) feature of this SAY glass-ceramic was its unchanged morphology and mechanical strength after neutron irradiation (Figure 2 (b)); these results made the SAY glass-ceramic one of the very first neutron resistant material suitable to join SiC/SiC in a pressure-less mode, feature of interest for nuclear energy production plant component [6].

Together with SAY, also a second glass–ceramic based on SiO_2–Al_2O_3–MgO (SAMg), designed to be used as pressure-less joining materials for SiC/SiC, gave excellent results potentially exploitable to build components for nuclear applications. Glass–ceramic pellets made of SAY and SAMg glass-ceramics were irradiated for approximately one year in the reactor core of the LVR-15 research reactor at Nuclear Research Institute Rez, Czech Republic, (neutron irradiation of about 1 dpa) and SiC/SiC composites joined by SAY glass-ceramic were irradiated about 1 year at High Flux Reactor (HFR), Petten, The Netherlands, (neutron irradiation of ~ 5 dpa). Optical microscopy with image analysis and scanning electron microscopy with X-ray microanalysis (EDS) were used to investigate the glass–ceramic morphology and composition, showing a remarkable similarity before and after neutron irradiation for both glass–ceramics. Comparison of bending strength for irradiated and non-irradiated SAY glass-ceramic joined SiC/SiC indicated that the mechanical strength was unaffected by neutron irradiation at these conditions [6].

Due to these promising results, SAY is being used within the EU project Il Trovatore, https://www.iltrovatore-h2020.eu/) to join SiC/SiC tubes produced by CEA (Commissariat à l'énergie atomique et aux énergies alternatives) (Figure 3 (a)). It can be difficult to join very thin samples (SiC/SiC tube thickness = 0.95 mm) using a slurry process, as a CT-scan of the joined region (Figure 3 (b) shows. Most of the joining material was squeezed outside the joined region and an alternative joining method should be tested in order to fully exploit the advantages of SAY as joining material for SiC/SiC. As it can be seen in Figure 3 (c), the SEM on a cross-sectioned joined area shows an excellent adhesion of SAY glass-ceramic; however, after testing these joints in working conditions (autoclave, water 330 °C, 18 MPa), a certain corrosion of the joining material is evident, Figure 3 (d). Some investigations now ongoing seem to suggest the dissolution of silica, one of the three crystalline phases found in SAY glass-ceramic. If this is the case, a change in SAY composition must be considered.

Another very promising glass-ceramic for joining of CMC is a *non-silica* based composition labelled CA after its constituents, CaO and Al_2O_3, (Table II). The "glass-ceramic method" was successfully used to join several SiC/SiC by the CA parent glass: also for this non-silica based parent glass, the joint was obtained by a pressure-less process, followed by parent glass crystallization of to obtain a glass-ceramic joint. The absence of crystallization peaks on the DTA curve a1, Figure 4 (a), relative to the CA glass-ceramic, is due to the complete crystallization of the parent CA glass (curve a2, Figure 4) during the joining process. Figure 4 (b) shows the XRD of CA as prepared (b1) and as after the thermal treatment used for the joining process (b2): the as obtained CA already contained some crystalline phases (curve b1), which grow to give a joint composed by $3CaOAl_2O_3$ and $12CaO_7Al_2O_3$, without any detectable sign of residual amorphous phases (curve b2) [22]. The mechanical strength of CA joined SiC-based components is still subject of investigation within a common research program involving ORNL (USA) and Politecnico di Torino (Italy): torsion tests on CA joined SiC-based components were tested before and after neutron irradiation 350 days (20 dpa) at 530°C. CA glass ceramic joint retained their strength, of about 100 MPa in torsional shear, after exposure to neutron irradiation at ORNL: this additional demonstration encourages further development of glass-ceramics as joining materials for SiC/SiC for nuclear applications [26].

Now focusing oxide/oxide CMC, namely alumina fiber reinforced oxide matrix composites, two silica-based glass compositions (SACM and GOX) were designed and successfully tested to join different Ox/Ox by the glass-ceramic method [24,25]. Table II summarizes SACM and GOX parent glasses and derived glass-ceramics compositions, characteristic temperatures, CTE, crystalline phases and joining process temperatures. Also in these cases, the CTE compatibility with the Ox/Ox to be joined was the main concern for the design of the glass composition.

Figure 5 shows DTA and Heating Stage Microscopy (HSM) results of SACM parent glass (a) with temperature of first shrinkage (TFS), temperature of maximum shrinkage (TMS), on-set crystallization temperature (T_X), crystallization end temperature (T_Y), peak crystallization temperature (T_P) and glass transition temperature (T_g). Figure 5 (b) is the SEM cross section of SACM glass-ceramic joined Ox/Ox: the interface is continuous and cracks free. The presence of a few closed porosities is due to the slurry method used to obtain the joints and, also in this case, an alternative, better joining technology should be developed. However, both SACM and GOX gave excellent mechanical strength for the joined Ox/Ox. In the case of GOX, the mechanical strength at 850 °C was about 80% of the un-joined composite tested for reference [24, 25].

Conclusions

Main results on research activity done in the past 25 years at the Politecnico di Torino on joining of oxide and non-oxide CMCs have been discussed for applications ranging from aerospace to energy production. Several new glass compositions have been designed and successfully tested, thus demonstrating that glasses and glass-ceramics are suitable materials to obtain sound joints with pressure-less processes.

Further activity is now focused on glass-ceramics localized heating and joining.

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List of Table and Figure Captions

Table I. Summary of glass composition, preparation and joining temperature initially (1994-2004) used to join SiC/SiC and C/C composites.

Table II Summary of parent glasses and derived glass-ceramics used to join oxide and non-oxide based Ceramic Matrix Composites (CMC): composition, characteristic temperatures, CTE, crystalline phases and joining process temperature.

Figure 1. Differential thermal analysis of a SiO_2-Al_2O_3-Y_2O_3 based (SAY) parent glass (a) and SAY glass–ceramic (b) obtained by heat treatment of the SAY parent glass at 1375 °C for 20 min and at 1235 °C for 1 h. The absence of crystallization peaks on curve (b) relative to the SAY glass-ceramic is due to the crystallization of the parent SAY glass during the joining process. The graph is reprinted from Ref. [5].

Figure 2. SAY glass-ceramic as neutron resistant joining material for CMC: SEM microscopy of SAY glass-ceramic before (a) and after (b) neutron irradiation: SEM-EDS on

irradiated and non-irradiated SAY confirms the presence of three phases white phase ($Y_2Si_2O_7$), gray phase (Al_2SiO_5) and darker phase (SiO_2). The images are reprinted from Ref. [6].

Figure 3. SAY joined SiC/SiC tubes (a), CT-scan (b) and SEM on the joined area before (c) and after (d) autoclave (water 330 °C, 18 MPa) (courtesy of CEA, University of Oxford and SCK-CEN)

Figure 4 (a) Differential thermal analysis of: (a1) as-cast CaO-Al_2O_3 based parent glass (CA) (curve a2) and CA glass-ceramic after heat treatment at 1480°C for 10 min (20°C/min) (curve a1); (b) XRD of CA as obtained (b1) and after the thermal treatment used for the joining process (b2). The images are reprinted from Ref. [22].

Figure 5. DTA and Heating Stage Microscopy (HSM) of SACM parent glass (a) showing temperature of first shrinkage (T_{FS}), Temperature of maximum shrinkage (T_{MS}), on-set crystallization temperature (T_X), crystallization end temperature (T_Y), peak crystallization temperature (T_P) and glass transition temperature (T_g); (b) SEM cross section of SACM glass-ceramic joined Ox/Ox. The images are reprinted from Ref. [24].

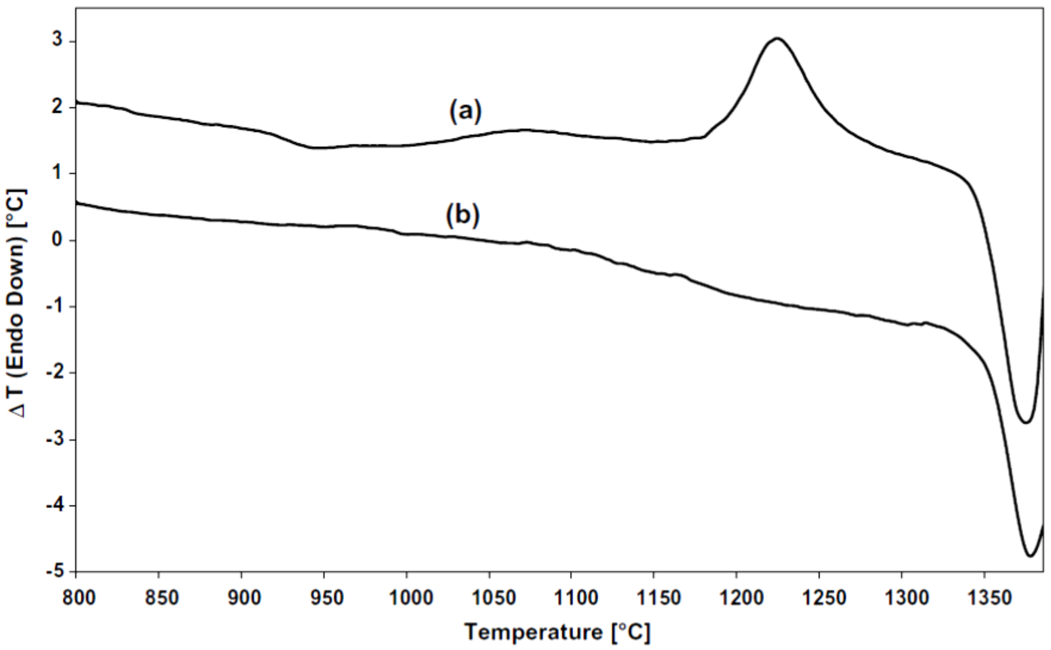


Figure 1

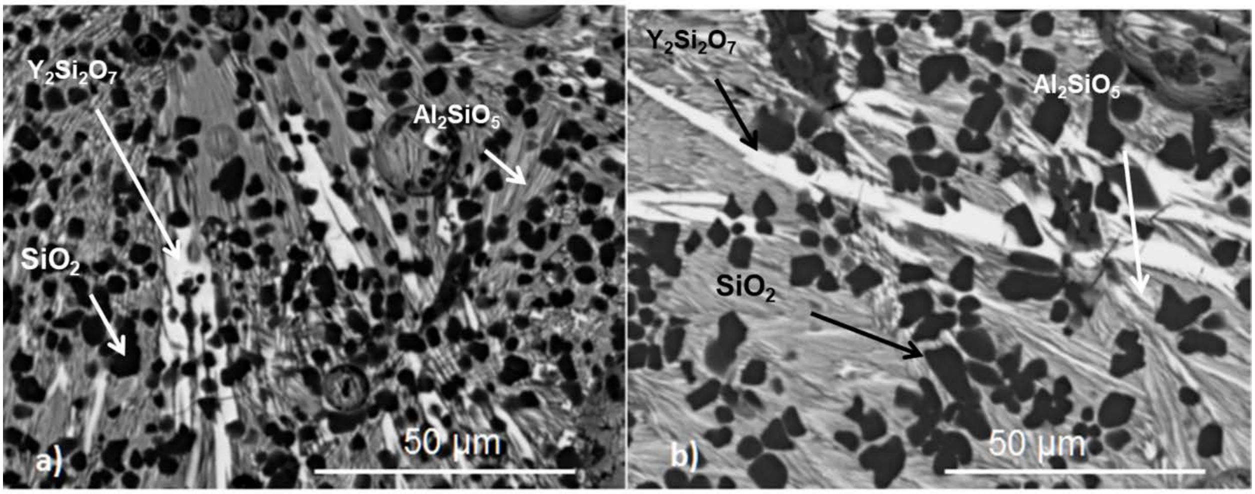
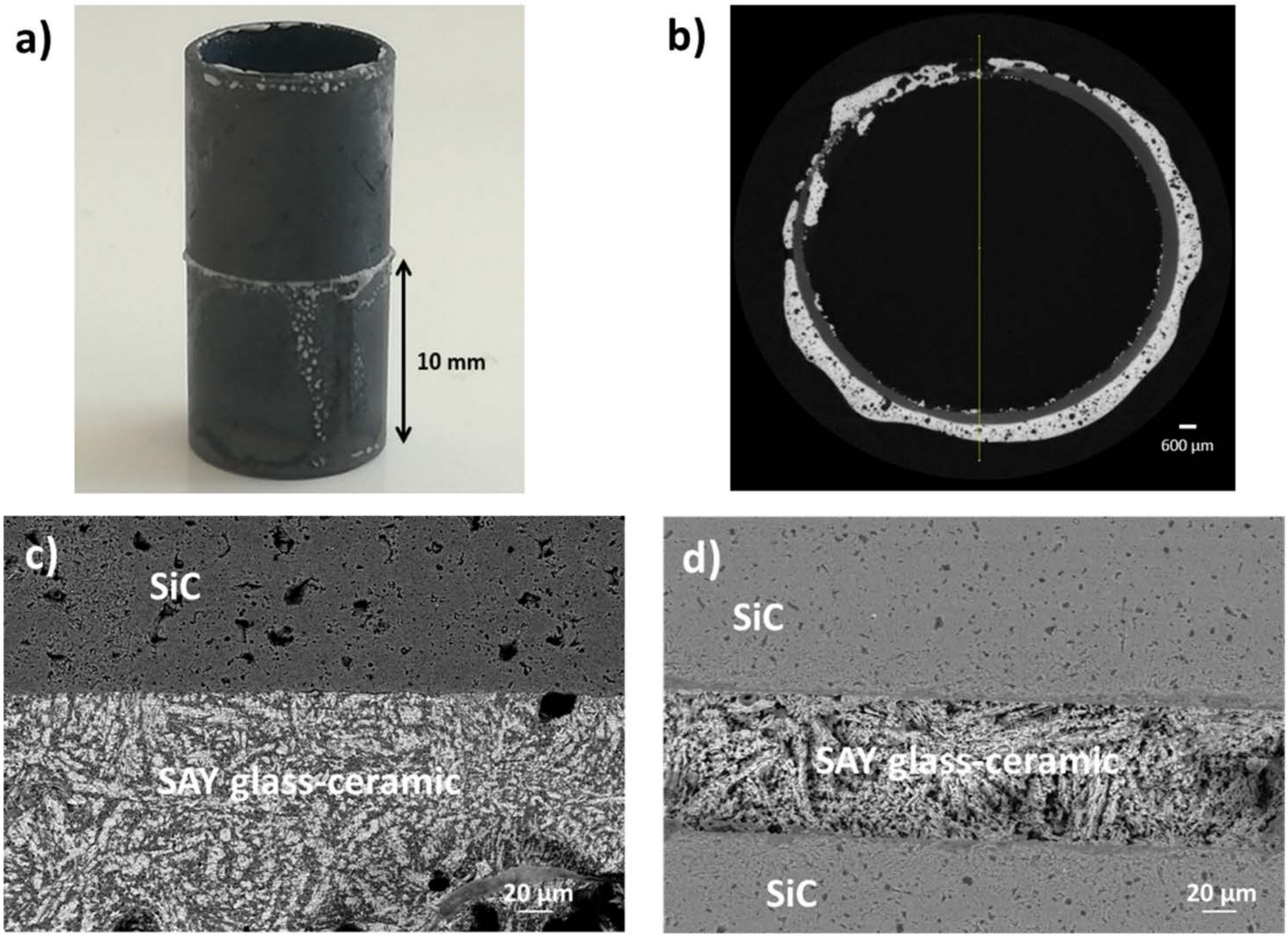


Figure 2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10 mm
b)
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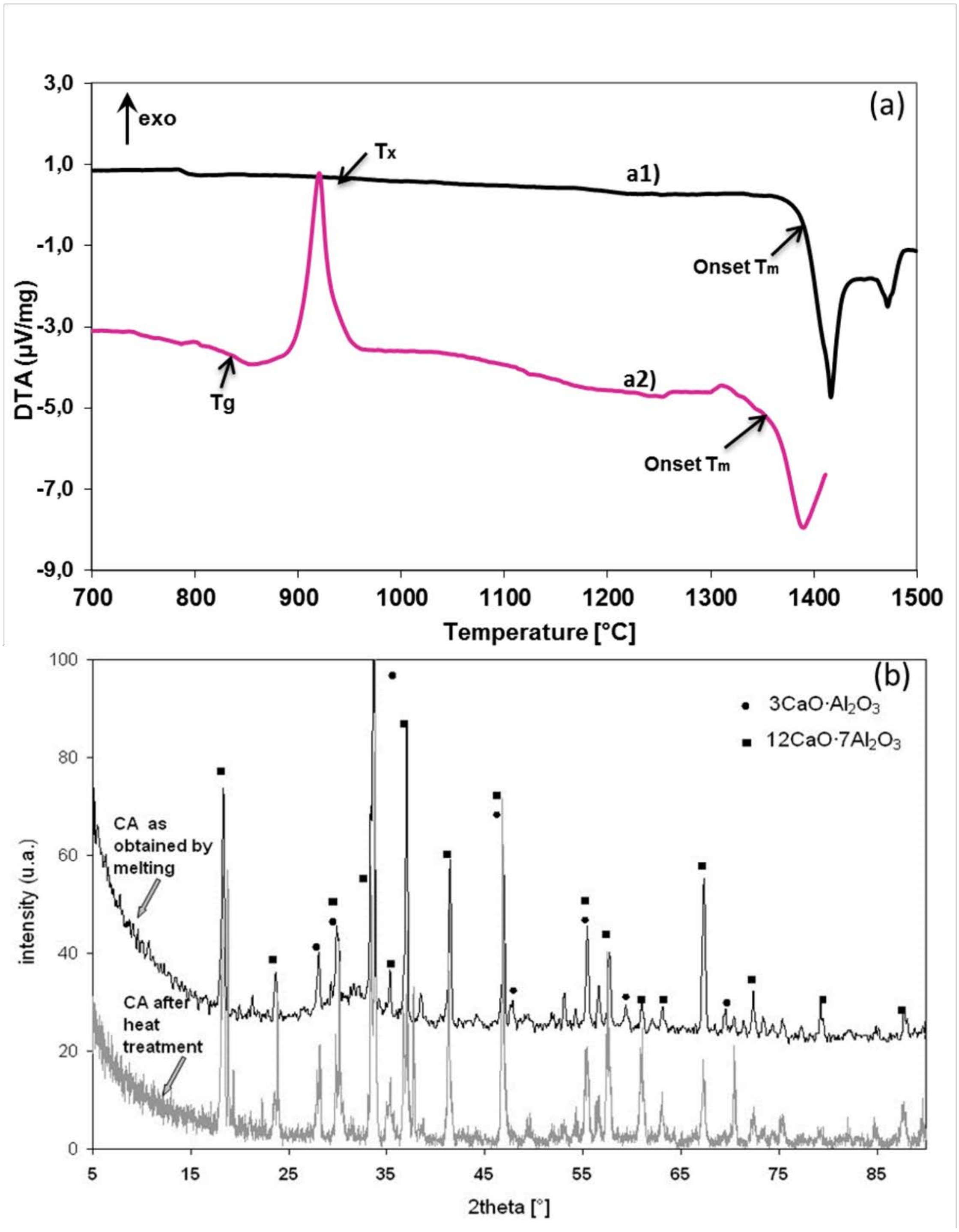
c)
SiC
SAY glass-ceramic
20 µm
d)
SiC
SAY glass-ceramic
SiC
20 µm

Figure 3



(a)
exo
Tx
a1)
Onset Tm
Tg
a2)
Onset Tm
DTA (µV/mg)
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900
1000
1100
1200
1300
1400
1500
Temperature [°C]
(b)
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■ 12CaO·7Al2O3
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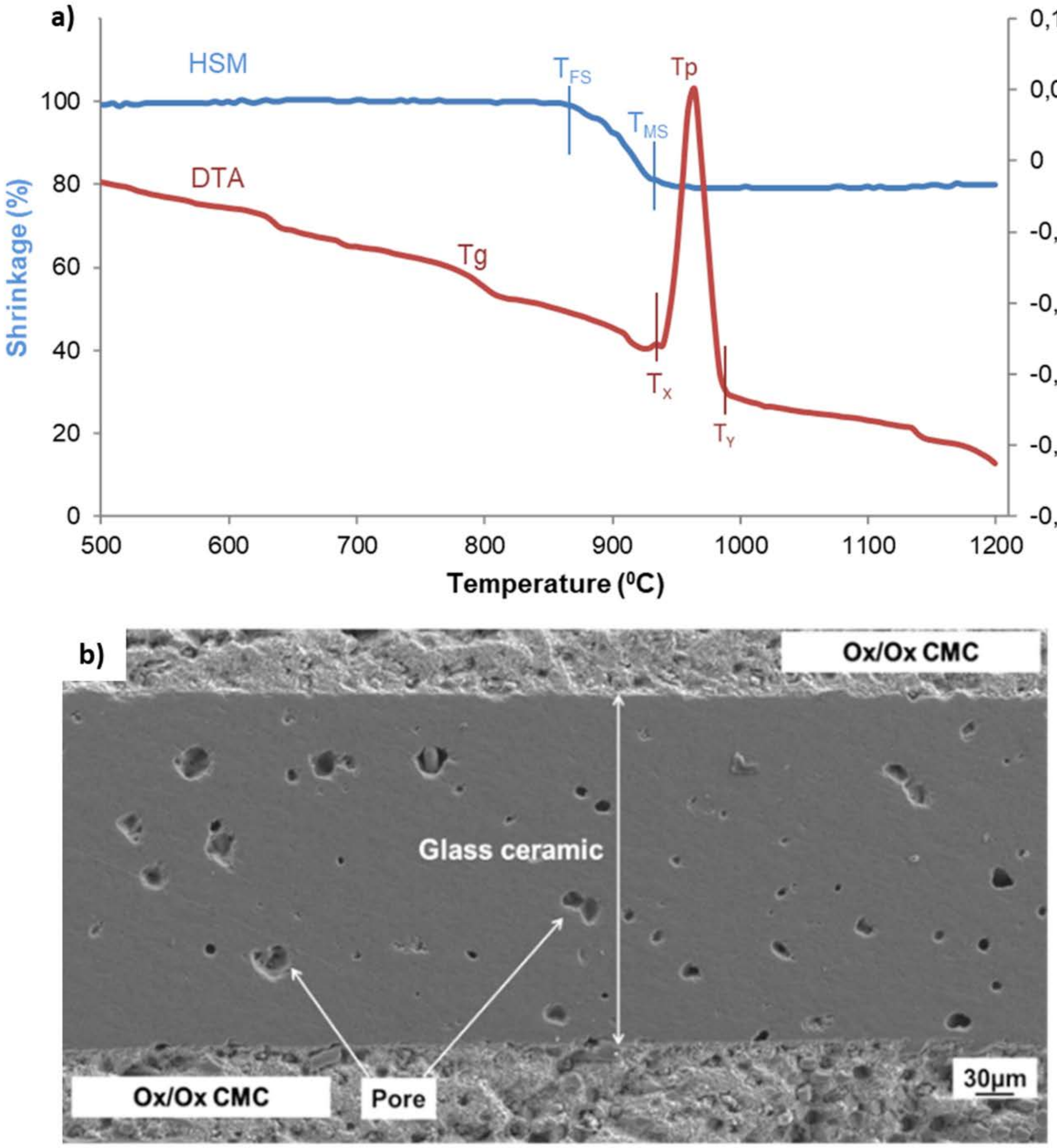
Figure 4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T_FS
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Tg
T_X
T_Y
Shrinkage (%)
DTA (µV/mg)
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60
80
100
0,1
0,05
0
-0,05
-0,1
-0,15
-0,2
-0,25
500
600
700
800
900
1000
1100
1200
b)
Ox/Ox CMC
Glass ceramic
Pore
Ox/Ox CMC
30µm

Figure 5

Table I. Summary of glass composition, preparation and joining temperature initially (1994-2004) used to join SiC/SiC and C/C composites.

Acronym	Composition (molar%)	Preparation T (°C)	Joining temperature (°C)	Reference
SB	80 SiO_2 20 B_2O_3	1600	1020-1200	7- 9
BAS	78.2 SiO_2 2.3 Al_2O_3 19.5 B_2O_3	1600	1300	7, 10
SABB	70.4 SiO_2 2.1 Al_2O_3 17.5 B_2O_3 10 BaO	1600	1310	7, 11-13
ZBM	50.46 ZnO 29.48 B_2O_3 9.12 MgO 4.53 SiO_2 4.04 Al_2O_3 2.37 Na_2O	1200	1200	8-10

Table II Summary of parent glasses and derived glass-ceramics used to join oxide and non-oxide based Ceramic Matrix Composites (CMC): composition, characteristic temperatures, CTE, crystalline phases and joining process temperature.

PARENT GLASS							**GLASS-CERAMIC**			**CMC JOINING**		**REF.**
ACRONYM	Composition (wt%)	Prepar. T (°C)	CTE (10^{-6} °C^{-1})	T_g (°C) ± 5	T_{soft} (°C) ± 10	Cryst. T (°C) ± 5	CTE (10^{-6} °C^{-1})	crystalline phases	Melting onset T (°C) ± 10	Joining T (°C) ± 10	Joined CMC	
SAY	54 SiO_2 18.07Al_2O_3 27.93 Y_2O_3	1750	3.8 (400-700°C)	910		1220	5.5 (400-700°C)	cristobalite, $Y_2Si_2O_7$ mullite	1377	1375 (20 min) 1235 (60 min)	SiC/SiC	5, 6, 15-18
SAMg	60 SiO_2 30Al_2O_3 10MgO	1700	3.1 (400-700°C)	867	985	1053 1151	3 (200-600°C)	Mullite cordierite	1240	1180	SiC/SiC	6, 11, 17, 19
CA	49.77 CaO 50.23 Al_2O_3	1750	7.43 (25°C) 9.42 (350°C)	850	1380	920 1050	5.2 (400-700°C)	$3CaOAl_2O_3$ $12CaO7Al_2O_3$	1380	1480	SiC/SiC	17, 18, 20-23
SACM	42 SiO_2 32 Al_2O_3 20 CaO 6 MgO	1600	5.53 (200-650°C)	790	873	945	7.40 (200-650°C)	Anorthite Gehlenite magnesian	1464	980	Ox/Ox	24
GOX	39.5 SiO_2 12.5 Al_2O_3 21.5 CaO 12.5 MgO 12.5 Y_2O_3 1.5 ZrO_2	1650	-	754	911	941 1010	8.93 (125-900°C)	$Ca_4Y_6O(SiO_4)_6$ $Ca_2(Mg_{0.5}Al_{0.5})(Si_{1.5}Al_{0.5}O_7)$ (Akermanite-Gehlenite) $CaMg(SiO_3)_2$ (diopside)	>1000	1010	Ox/Ox	25