Wetting behavior and reactivity of liquid Si-10Zr alloy in contact with Glassy Carbon and SiC

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Introduction

Goal

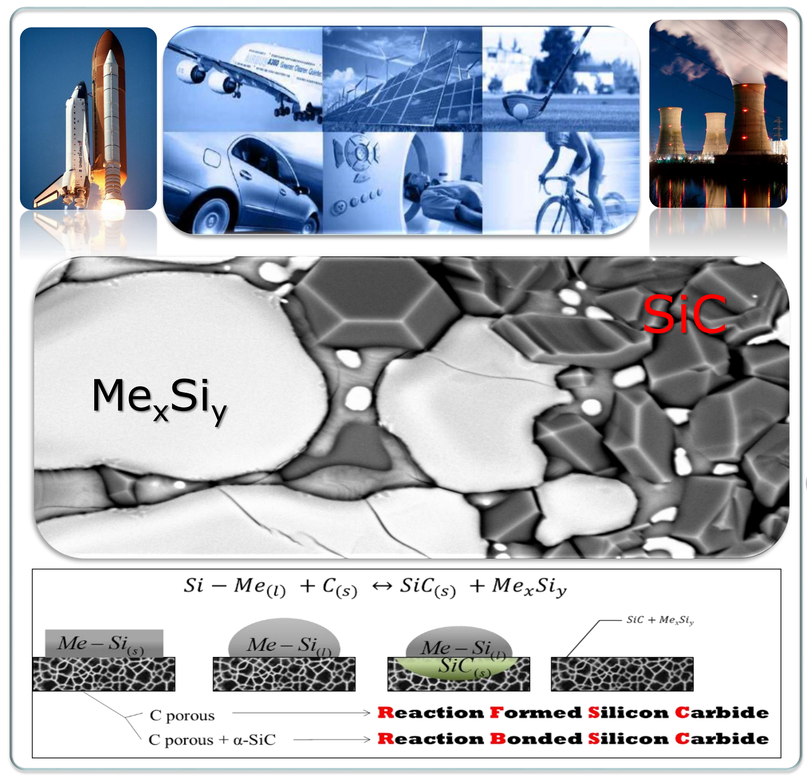
Aiming to "**mimic**" the conventional operating conditions imposed in fabricating **MMCs** via liquid assisted processes such as **reactive infiltration**, the contact heating sessile drop method (CHSD) was applied for better understanding the interaction phenomena occurring between the liquid **Si-10at%Zr** alloy in contact with **Glassy Carbon** (GC) and **SiC** substrates. Specifically, the **contact angles** behaviors as a function of time were obtained over the temperature range of **T = 1354-1500° C** under an Ar atmosphere.

Why?

Fundamental investigations performed under **well-defined operating conditions** on the occurring **interfacial phenomena** between liquid Si-Zr alloys in contact with C and SiC substrates, are **key steps** for optimizing liquid assisted processes (reactive infiltration). Targeted **wettability and reactivity studies** can easily provide useful indications for solving many technological problems affecting the reactive infiltration mechanisms, such as **pore closure/narrowing phenomena**.

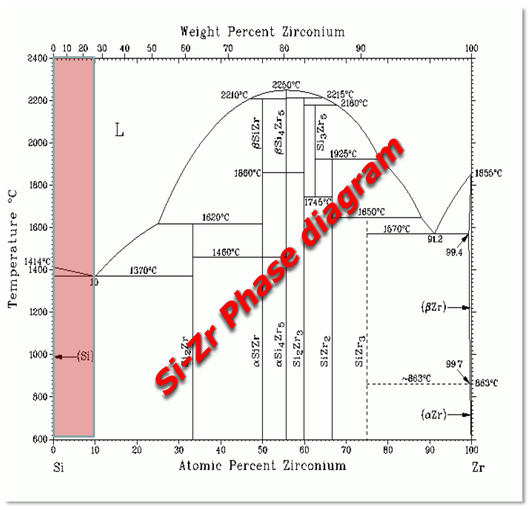
Impact

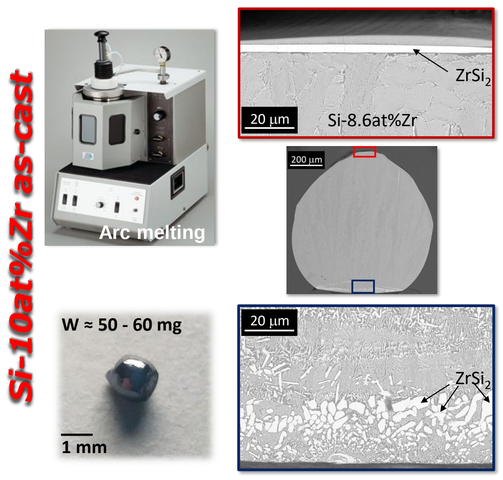
Currently, the main **challenges** facing the development of **advanced refractory MMCs (SiC-$ZrSi_2$) and CMCs (C/C-ZrC-SiC)** are related to the **costly fabrication** of tailored and performant interfaces microstructures for highly demanding applications, to **optimize** the fabrication process and to **identify** ad-hoc the metal matrix for each application.



Computational models are enabling the design and manufacture of **costless and high quality advanced complex metal-based materials** such as **SiC-based composites**, since longer gaining interest for the use in aerospace, automotive, defense, nuclear reactors and various other engineering applications. In particular, the production of **tailored SiC-based composites** via **reactive infiltration of Si-based alloys into C-and SiC-based preforms**, mainly by assisting computational simulations, is currently one of the main goals of **materials design**.

Materials & Method

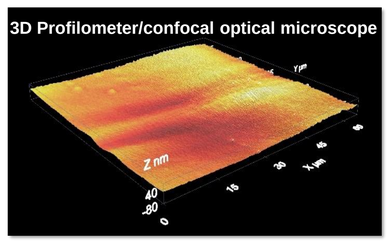


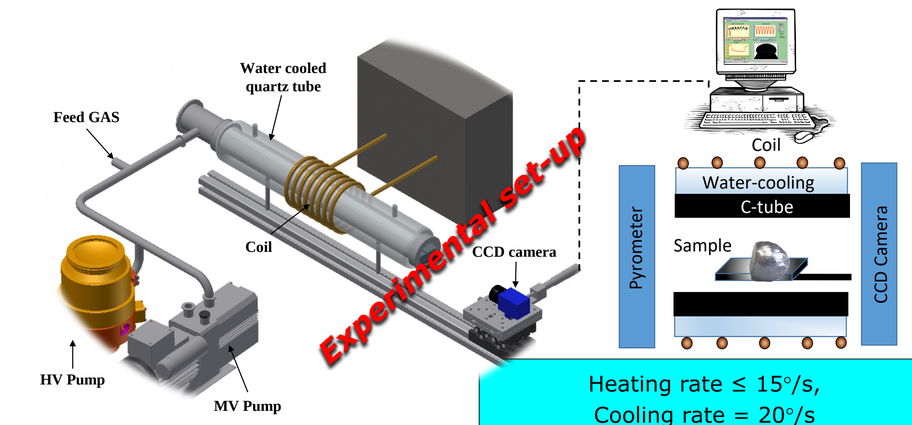


Substrates

Glassy Carbon (**GC**) Alfa Aesar,
Ra ≈ 20 nm

HP SiC (**SiC**) Goodfellow,
Ra ≈ 1µm



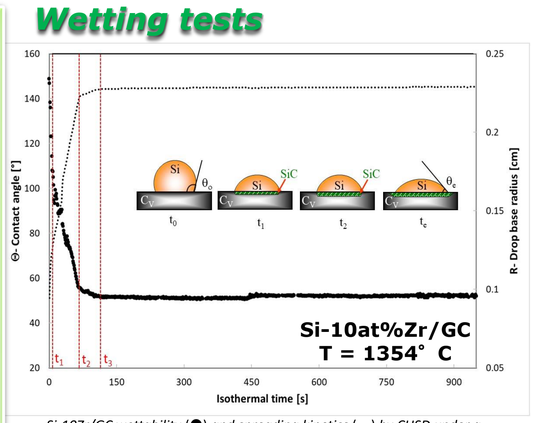


T ≤ 1700°C, 1 < P_{tot} < 10^{-6} mbar
C-heater acting as O_2-getter

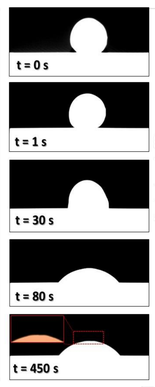
Heating rate ≤ 15°/s,
Cooling rate = 20°/s
Thermocouples/Pyrometer
Real-time residual gas analysis
Real-time acquisition data (ASTRAview®)

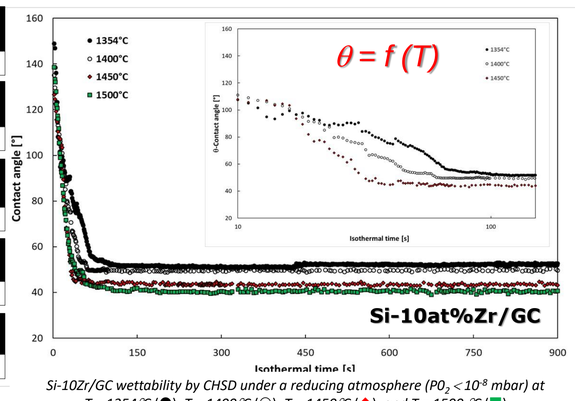
Results

Wetting tests

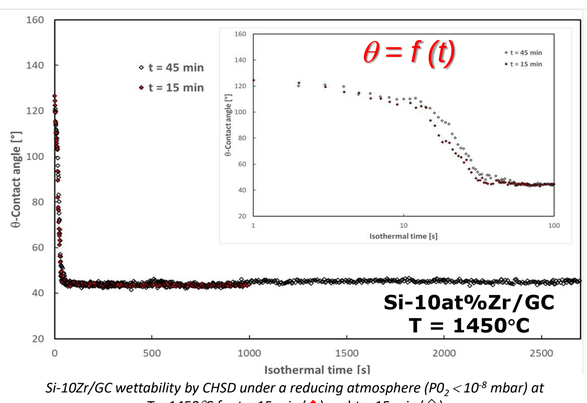


Si-10Zr/GC wettability (●) and spreading kinetics (….) by CHSD under a reducing atmosphere ($PO_2 < 10^{-8}$ mbar) at T = 1354 ℃

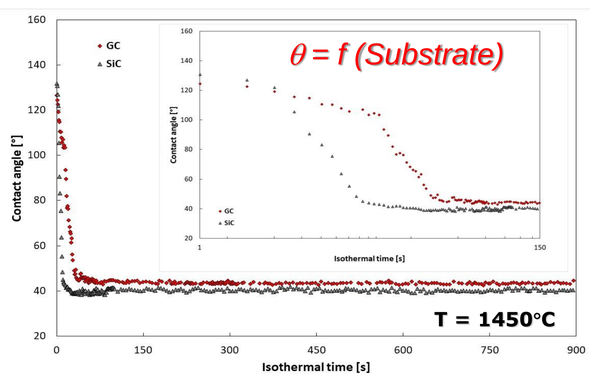




Si-10Zr/GC wettability by CHSD under a reducing atmosphere ($PO_2 < 10^{-8}$ mbar) at T = 1354 ℃ (●), T = 1400 ℃ (○), T = 1450 ℃ (◆), and T = 1500 ℃ (■)

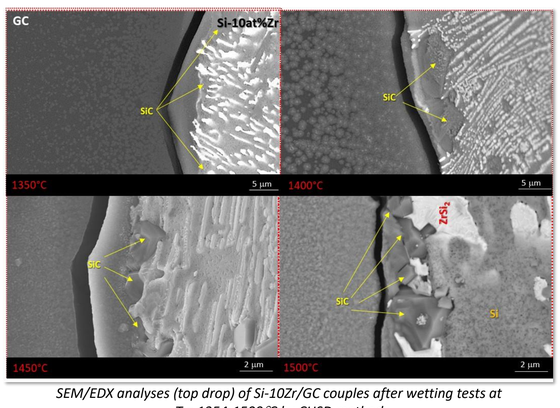


Si-10Zr/GC wettability by CHSD under a reducing atmosphere ($PO_2 < 10^{-8}$ mbar) at T = 1450 ℃ for t = 15 min (◆) and t = 15 min (◇)

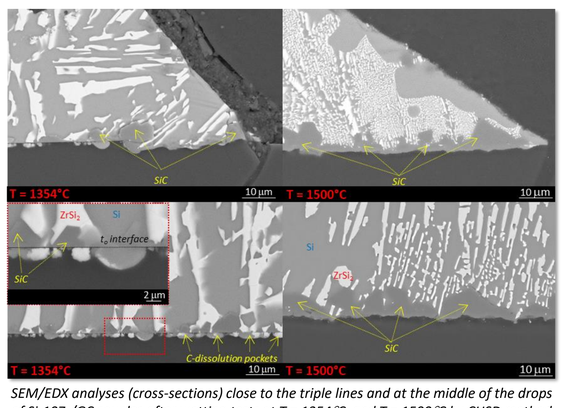


Si-10Zr/GC (◆) and Si-10Zr/HP SiC (▲) wettability by CHSD under a reducing atmosphere ($PO_2 < 10^{-8}$ mbar) at T = 1450 ℃

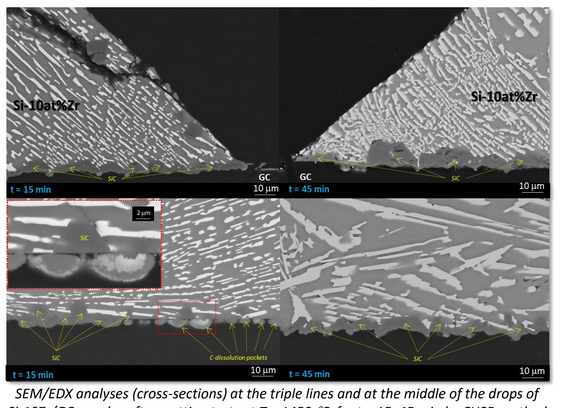
Microstructural characterization



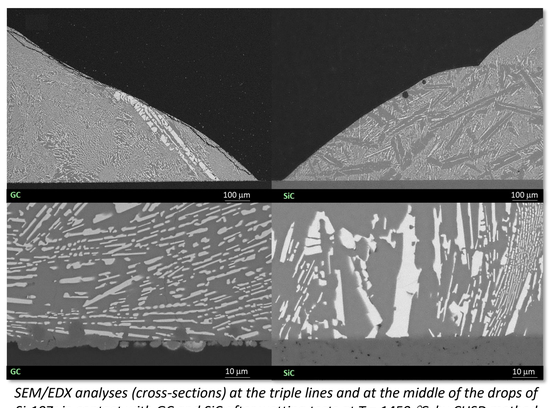
SEM/EDX analyses (top drop) of Si-10Zr/GC couples after wetting tests at T = 1354-1500 ℃ by CHSD method



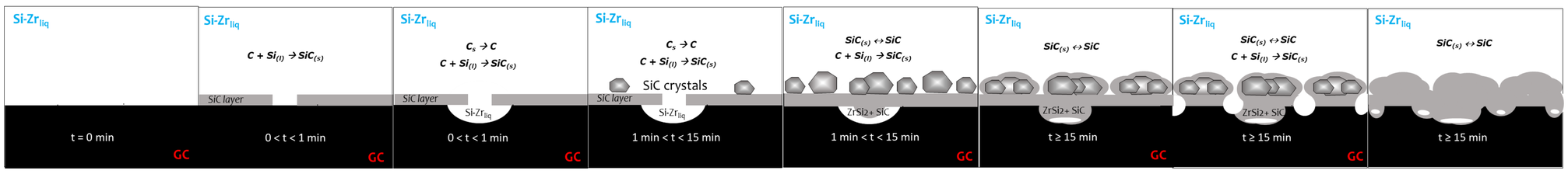
SEM/EDX analyses (cross-sections) close to the triple lines and at the middle of the drops of Si-10Zr/GC couples after wetting tests at T = 1354 ℃ and T = 1500 ℃ by CHSD method



SEM/EDX analyses (cross-sections) at the triple lines and at the middle of the drops of Si-10Zr/GC couples after wetting tests at T = 1450 ℃ for t = 15, 45 min by CHSD method



SEM/EDX analyses (cross-sections) at the triple lines and at the middle of the drops of Si-10Zr in contact with GC and SiC after wetting tests at T = 1450 ℃ by CHSD method



Schematic of all the possible interfacial phenomena occurring at the Si-10at%Zr/GC interface and their evolution over time [1]

Conclusions

- For the first time, the **interfacial phenomena** observed in terms of **contact angle values**, **wettability** and **spreading kinetics** at the liquid **Si-10%Zr alloy/GC** interfaces are analyzed and related to the temperature and time under an atmosphere with reduced oxygen content.
- The **final contact angle values** measured at the Si-10at%Zr/GC interfaces over the temperature range of **T = 1354-1500°C**, were decreasing from **52° to 40°**.
- **Reactive wetting** mechanism was mostly driving all the interfacial phenomena observed.
- The **growth of the SiC-reaction layer** at the interface and at the triple line is a **time** and **temperature**-dependent phenomenon.
- The contact angle values measured at the Si-10%Zr/GC are **comparable** and in **very good agreement** with the values reported in literature [2,3].
- The results obtained can be helpful to identify the **key parameters** affecting the manufacturing of SiC/$ZrSi_2$ and C/C-ZrC-SiC composite materials via **reactive infiltration**.

Temperature [°C]	time [min]	θ_f [°]	SiC-thickness [µm] TL	SiC-thickness [µm] MD
1354	15	51÷52	4.5	5
1400	15	49	5	5
1450	15	43	4.5	5
1450	45	45	8	5
1500	15	40	6	6

Contact angle values and SiC-layer average thickness both at at the triple lines (TL) and at the middle of the drop (MD) as a function of temperature and time.

[1] R. Voytovych, V. Bougiouri, N. R. Calderon, J. Narciso, N. Eustathopoulos, Reactive infiltration of porous graphite by NiSi alloys, Acta Mater. 56 (2008) 2237–2246.
[2] B. Drevet, N. Eustathopoulos, Wetting of ceramics by molten silicon and silicon alloys: a review, Journal of Materials Science 47 (2012) 8247–8260.
[3] M. Naikadea, B. Fankhänel, L. Weber, A. Ortona, M. Stelter, T. Graule, Studying the wettability of Si and eutectic Si-Zr alloy on carbon and silicon carbide by sessile drop experiments, J- Eur. Ceram. Soc., 39 (2019) 735-742.

Acknowledgements

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