Effect of h-BN spray coatings on wetting and reactivity in Si/SiC system

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Motivation

A successful implementation of highly efficient latent heat thermal energy storage systems working at ultra high temperatures (up to 2000 °C) requires a proper selection of both phase change materials (PCM) and refractories that would withstand a long-term contact heating under such extreme conditions. Silicon based materials have been pointed out as very interesting candidates for PCMs applications.

Regarding an achievement of high reliability and long lifetime of the LHTES device, the most important conditions are a lack of wettability and negligible reactivity between the PCM/refractory couples. However,almost all of commonly applied ceramics are well wetted by molten Si at temperatures around its melting point. The only one exception is hexagonal boron nitride that shows non-wetting behavior at temperatures up to 1500°C, while there is no available information on the couple behavior at higher temperatures. Furthermore, the performance of the boron nitride coatings has not been so far evaluated under conditions similar to that predicted in the Project.

In this work, we experimentally examined the wettability and reactivity behavior of silicon on commercially available polycrystalline SiC substrates coated by two types of h-BN spray coatings. The Si/h-BN coating/SiC samples were subjected to sessile drop experiments at temperatures up to 1750 °C.

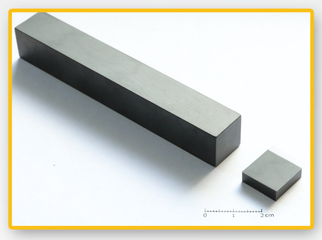
Materials

Polycrystalline Si
ultra high purity Si (7N)

+

h-BN spray coatings:
A: Henze HeBoCoat® 21E - polymer slurry
B: Henze HeBoCoat® 401E - ceramic slurry

+



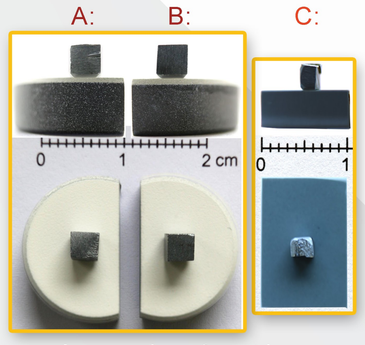
Polycrystalline SiC substrates
FRIALIT® SiC 198D

Samples preparation

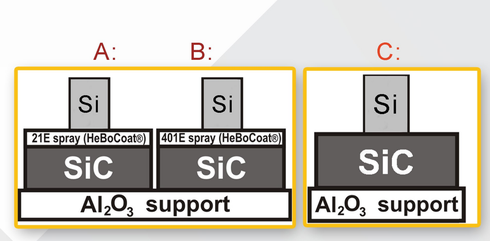
Polycrystalline SiC substrates (FRIALIT® SiC 198D*) and ultra high purity Si (7N) pieces were cut by diamond discs on a precise cutting machine; and then subjected to grinding and polishing with diamond paste (the SPM measured roughness of SiC surface was ~100 nm). Such prepared substrates were coated by two types of h-BN sprays:

A: Henze HeBoCoat® 21E - polymer slurry
B: Henze HeBoCoat® 401E - ceramic slurry

Before the wetting tests each substrate (coated or not) was subjected to vacuum preaheating treatment (at 150°C/10^{-5} bar/45 min) in order to remove residual gases.



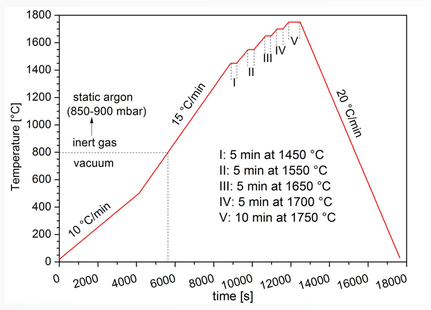
A macroview of samples
before the wettability test



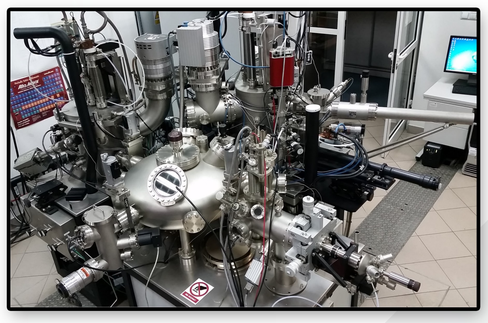
couple A: Si/h-BN spray (HeBoCoat 21E) coating/polycrystalline SiC
couple B: Si/h-BN spray (HeBoCoat 401E) coating/polycrystalline SiC
couple C: Si/polycrystalline SiC (REFERENCE non-coated sample)

Experimental procedure

SiC substrates (h-BN coated and non-coated) were subjected to testing of wettability in contact with Si by sessile drop method combined with contact heating procedure at T up to 1750°C.
five intervals:
1450°C/5 min (I);
1550°C/5 min (II);
1650°C/5 min (III);
1700°C/5 min (IV);
1750°C/10 min (V)

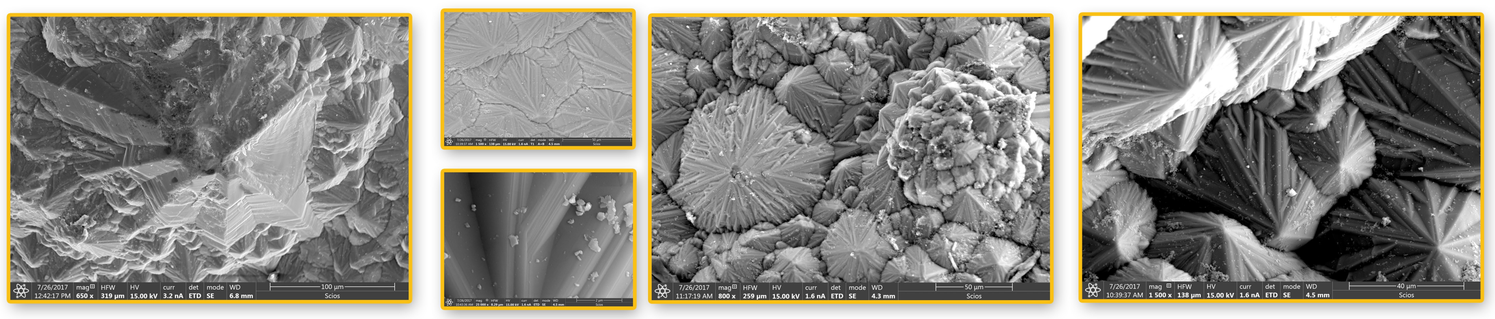


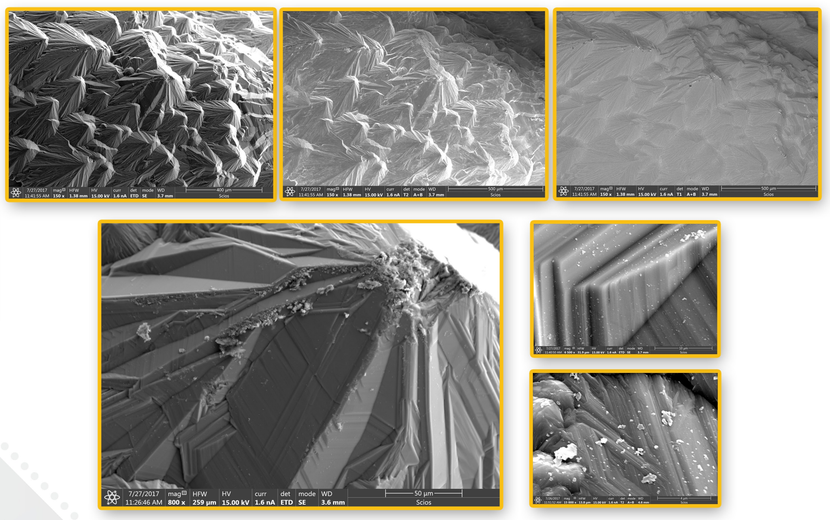
Experimental complex for investigation of molten metal/ceramics interactions



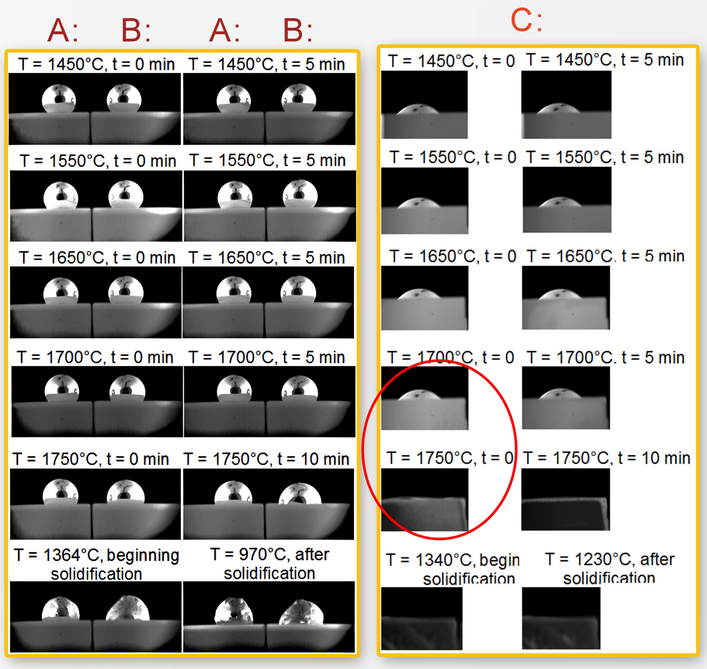
Results - SEM observations of Si in the as-received condition

a comparison of various SEM imaging modes
(ETD vs. in-lens detectors)





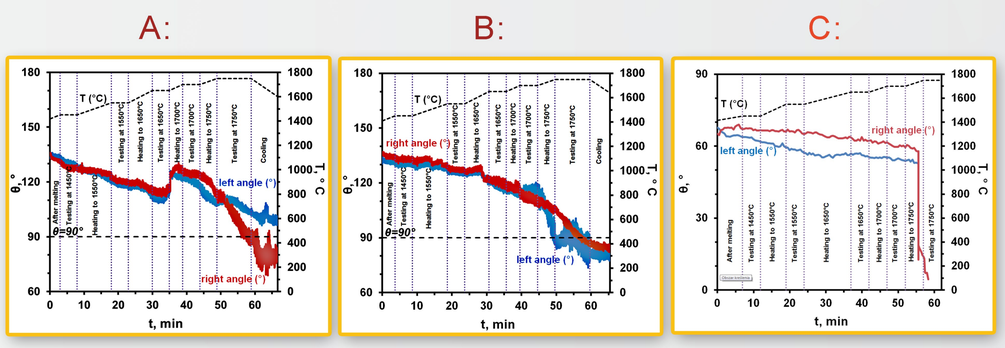
Results - The wettability test



Images of Si drops on coated and non-coated SiC substrates
in-situ recorded by high-speed camera (100 fps)
upon the wettability test at T up to 1750°C

Post-processing of collected data

The contact angle values vs. time plots were calculated by dedicated software (ASTRA2 (IENI-CNR, Italy)) based on in-situ recorded images of drops/substrates



couple A: Si/h-BN spray (HeBoCoat 21E) coating/polycrystalline SiC
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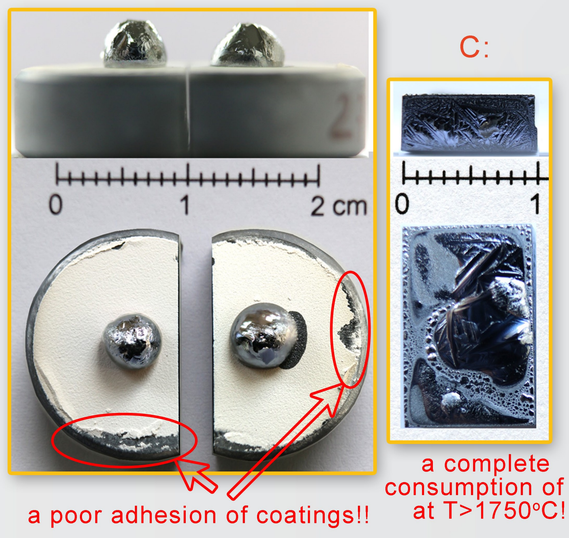

A macroview of solidified couples
after the wettability test

CONCLUSIONS

The application of h-BN spray coatings:
1) supresses a high reactivity of Si/SiC system at T up to 1750°C;
2) allows maintaining non-wetting behavior in this tempearture range.
However, low adhesion makes them rather unsuitable for thermal cycling applications.

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