

## Silicon and silicon-boron alloys as phase change materials in thermal energy storage units

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### Abstract

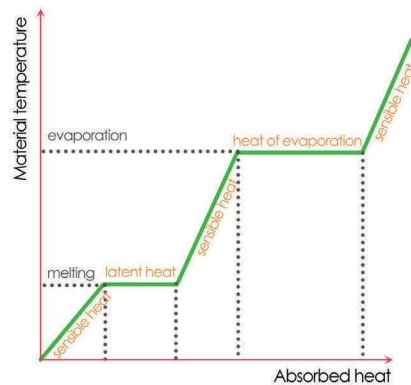
Although Si and Si-B alloys seem to be perfect candidates as high temperature PCMs (phase change materials) (in terms of their high melting points and latent heat values), a number of scientific/technical challenges have to be faced before introducing them into real Latent heat thermal energy storage (LHTES) devices. A special attention should be paid to a proper selection of reliable refractories for building the PCM container. For this purpose, the involved teams from the Foundry Research Institute in Krakow (FRI) and Norwegian University of Science and Technology (NTNU), have agreed to explore two distinctly different approaches: (i) “Non-wetting + negligible reactivity concept” and (ii) “Self-crucible concept”, respectively. In the former attempt, it has been assumed that the selected refractory material will be inert towards the contacting PCMs, i.e. the system will be characterized by non-wetting behaviour and a lack of significant dissolution of the refractory in molten PCM as well as no new reactively formed products. On the contrary, the principles of the latter approach include a reactive formation of continuous interfacial product layer at initial stages of PCM/refractory interaction. The reactively formed layer should play a role of barrier coating, and thus forms a “self-crucible” inside the PCM vessel.

In order to accomplish the Project’s goals, Si and Si-B alloys were fabricated by using various metallurgical routes (including an induction melting or an electric arc melting). After that, their high temperature solid/liquid state interaction with refractories selected in accordance to both proposed approaches, was examined. Additionally, a proper thermodynamic assessment of phase equilibria was performed in order to support obtained experimental data.

### Introduction

A thermal energy storage (TES) is considered as a good way to overcome drawbacks of concentrated solar power (CSP) systems regarding an energy availability during sunless periods. Among physical TES methods, those involving a latent heat evolution have gather a special attention. The latent heat thermal energy storage (LHTES) is based on absorption or release of heat from a material when it undergoes a *solid*↔*solid*, *solid*↔*liquid* or *liquid*↔*gas* phase change processes (Fig. 1) [2]. Such material is referred to as phase change material (PCM) and is the main player determining the overall output of the TES system. When a PCM is subjected to heating it absorbs the energy which amount is proportional to applied temperature (called as a sensible heat).

However, when temperature reaches a corresponding threshold of phase change, the PCM takes up a large amount of energy (so called latent heat or heat of fusion  $\Delta H$ ) to cover the transformation process. During a subsequent cooling step, the process goes in a reverse way and the absorbed energy is released. This effect has become a basis for the development of new energy storage and conversion units.



**Figure 1:** The heat evolution during heating of phase change material (based on [2]).

As it was already mentioned, the most important feature of every LHTES system is a PCM determining its performance, while temperature is the crucial operating parameter (generally, the higher the temperature the higher the unit potential). Unfortunately, current state-of-the-art systems are limited to temperatures around 800°C, due to a lack of suitable materials and devices. In this regard, the following main requirements should be fulfilled by PCM candidates [3, 4]:

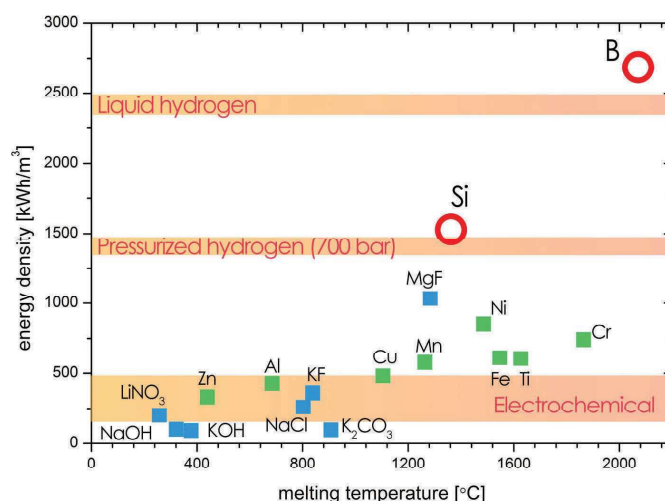
- 1) a high melting point near the required operational temperature range of the TES system;
- 2) a high latent heat per volume to improve energy storage density of the system;
- 3) a high thermal conductivity and heat capacity in both solid and liquid states to provide an additional sensible energy storage and lower charging and discharging times;
- 4) a high chemical and thermal stability of structure and properties upon thermal cycling operations.

So far, a wide range of various organic and inorganic materials have been proposed and examined as potential PCMs (e.g. [4-6]). Metals constitute a class of PCMs with a potentially higher energy density, thermal conductivity and possible operation temperatures than their organic counterparts (that are mostly based on molten salts) (Table 1).

**Table 1:** A comparison of selected thermophysical properties of organic and inorganic PCM candidates ( $T_m$  – melting point,  $\Delta H^\circ$  – latent heat,  $k$  – thermal conductivity) [4,7].

Class	Material	$T_m$ [°C]	$\Delta H^\circ$ [MJ/kg]	$k$ [W/mK]	Ref.
Organic	Paraffin	317	–		[7]
	Wax	379	0.07 – 0.21	0.19 – 0.75	
	Fatty	268	–		
	Acids	344	0.05 – 0.21	0.14 – 0.17	
	Hydrated Salts	281	–		
low melting point metals		1170	0.12 – 0.49	0.46 – 5.0	[4]
	Mercury	234	0.01	8	
	Cesium	302	0.02	17	
	Rubidium	312	0.03	29	
	Indium	430	0.03	36	
	Bismuth	544	0.05	8	
	Tin	505	0.06	15	
	Gallium	303	0.08	29	
	Lithium	459	0.43	41	
high melting point metals	Copper	1356	0.193	350	[7]
	Niobium	2750	0.29	82	
	Nickel	1728	0.3	83	
	Scandium	1814	0.31	16	
	Ruthenium	2607	0.38	80	
	Molybdenum	2896	0.38	84	
	Chromium	2180	0.4	48	
	Vanadium	2183	0.45	51	
	Beryllium	1560	1.31	69	
	<b>Silicon</b>	<b>1687</b>	<b>1.79</b>	<b>20</b>	
	<b>Boron</b>	<b>2350</b>	<b>4.6</b>	<b>10</b>	

As it comes from the comparison shown in Table 1, silicon and boron exhibit significantly greater  $\Delta H^\circ$  than any other materials, showing potentially available volumetric energy storage capacities an order of magnitude greater than typical salts used in CSP such as  $\text{NaNO}_3$  (Fig. 2). Thus, it has been assumed that introduction of Si-B alloys as PCMs should allow overcoming already existed limitations of TES systems as well as will also allow reaching energy densities similar to that available for other storage technologies (e.g. those based on pressurized or liquid nitrogen) (Fig. 2).



**Figure 2:** A possible energy density coming from latent heat of different materials as a function of melting temperature compared with the energy density of other storage technologies (based on [1]).

Of course, one of the main questions summarizing technological challenges related to the assumptions of AMADEUS Project is: *What kind of refractory material should be used for building a container dedicated for a long-term storage of solid/liquid Si and Si-B alloys at ultra-high temperatures (up to 2000°C)?*

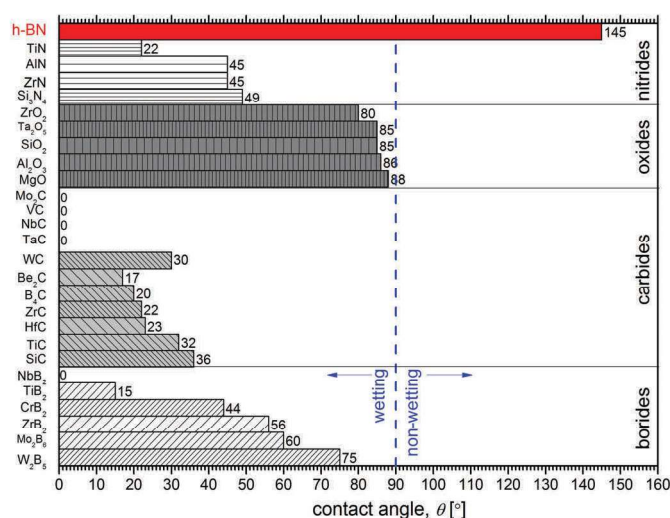
Giving the right answer on this question seems to be a not very easy task, since there is a prominent lack of reported data on (i) interaction of pure Si with refractories at very high temperatures ( $T > 1500^\circ\text{C}$ ); (ii) interaction of Si-B alloys with refractories at any temperature. For this reason, there is a strong need to evaluate basic high temperature capillarity phenomena (wetting, spreading, infiltration) and interfacial reactivity in Si(-B)/ceramic systems at temperatures higher than corresponding melting points of selected PCM candidates.

Consequently, in order to solve this problem and provide technological recommendations for a selection of container materials, the involved teams from the *Foundry Research Institute (FRI)* and *Norwegian University of Science and Technology (NTNU)*, have agreed to explore two distinctly different approaches.

#### “Non-wetting and negligible reactivity” approach (FRI)

In this attempt, it has been assumed that the selected refractory material will be inert towards the contacting PCMs, i.e. the system will be characterized by non-wetting behavior and a lack of significant dissolution of the refractory in molten PCM as well as no new formed products under conditions corresponding to those predicted for the LHTES system. It was decided to start with evaluation of pure Si/ceramic systems, and after that to examine the effect of boron addition on behavior of Si-B/ceramic couples. High temperature capillarity phenomena in Si/ceramic systems have been so far investigated mostly concerning manufacturing and processing of photovoltaic Si. In this matter, it is worth to recommend comprehensive review papers by Yuan et al. [8] and

Drevet and Eustathopoulos [9]. A wide range of refractories has been examined including various oxides, carbides, borides and nitrides. It has been documented that due to a high chemical affinity of silicon to oxygen, nitrogen and carbon most of ceramics show the contact angle  $\theta$  values close to or lower than  $90^\circ$  at temperatures  $<1500^\circ\text{C}$ , i.e. almost all of them are well-wetted by molten silicon, except hexagonal boron nitride (h-BN) that exhibits an unique ability to be not wetted (Fig. 3).



**Figure 3:** A brief overview on contact angle values reported in the literature for various Si/ceramic systems at  $T < 1500^\circ\text{C}$ .

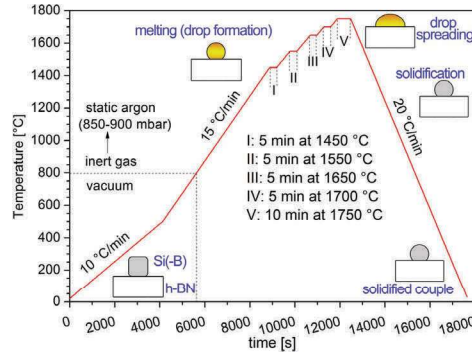
As it was discussed by Yasinskaya [10], the  $\theta$  value in Si/nitrides, Si/carbides and Si/borides systems is affected by the character of chemical reaction of the contacting materials. When, a chemical reaction is either absent or negligible (only for Si/h-BN), the surface of ceramics is non-wetted or slightly wetted by Si. In the case of reaction taking place between a ceramic and liquid Si leading to the formation of a wettable product, a good or even complete wetting takes place. The effect of interfacial product formation on wetting behavior might be well presented on example of Si/oxide ceramic systems. It was reported that an involved reaction mechanism in Si/Al<sub>2</sub>O<sub>3</sub>, Si/ZrO<sub>2</sub>, Si/SiO<sub>2</sub> or Si/MgO systems at temperatures  $<1500^\circ\text{C}$  [9] is mostly based on the dissolution of a ceramic substrate followed by the reprecipitation of SiO<sub>2</sub> product layer at the interface. Since the equilibrium contact angle of molten silicon on silica is around  $80\text{--}90^\circ$ , the behavior of all oxide ceramics is very similar. For carbides and nitrides, the situation is analogous since in contact with molten silicon they form well-wettable carbides, nitrides or silicides based interfacial products.

Thus, by taking into account the “non-wettability and non-reactivity” criteria, h-BN has been selected as the material of the first choice. However, it should be noted that although the contact angle values reported in the literature for Si/h-BN couples tested by using various materials and conditions are within the non-wetting regime ( $\theta > 90^\circ$ ) (Table 2), all available data are limited to  $1500^\circ\text{C}$ , and in fact – do not allow fully predicting the materials performance at higher temperatures.

**Table 2:** Contact angle  $\theta$  values reported in the literature for the Si/h-BN system [4,7].

Substrate	Substrate remarks	Testing conditions	Contact angle, $\theta$ [°]	Ref.
h-BN		Vacuum; <b>1500°C</b>	110	[12]
c-BN			95	
BN		Argon; <b>1420°C</b> ; 1200 s	145	[8]
p-BN	Pyrolytic BN		144	
p-BN	Pyrolytic BN	Vacuum; <b>1500°C</b>	105	[13]
BN	Hot pressed, no binder	Inert gas; <b>1412°C</b> ; 300 s	122	[14]
p-BN	Pyrolytic BN; $R_a=30$ nm	Argon; low $p_{O_2}$ ; <b>1430°C</b> ; 600 s	117	[15]

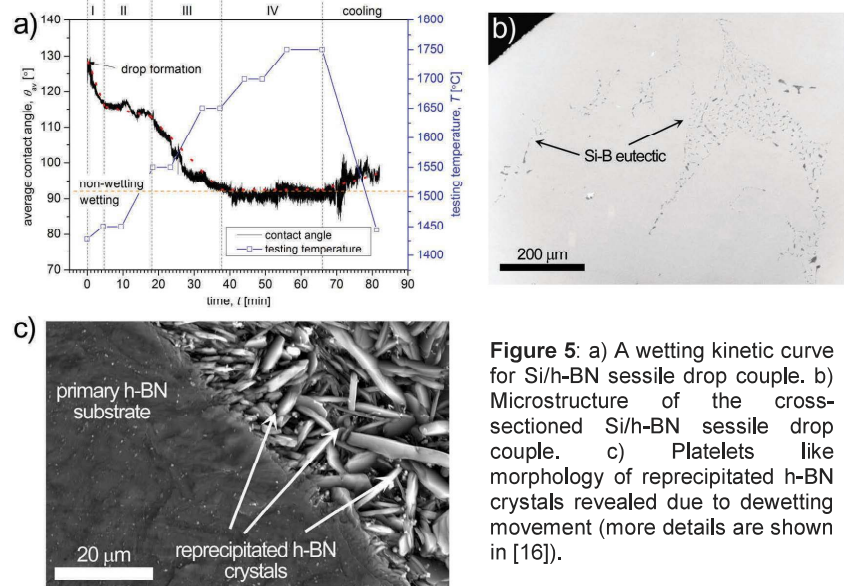
Therefore, in order to fill the existed gap and to verify the limits of materials applicability, a sessile drop experiments (Fig. 4) were performed by using dedicated complex for examining high temperature phenomena [11]. Ultra-high purity silicon (7N) pieces were placed on commercially available h-BN sintered substrates, and then subjected to heating to 1750°C in static argon atmosphere (850-900 mbar) in accordance to the temperature profile shown in Fig. 4c. During the tests, images of sessile drop couples were *in-situ* recorded by a CCD high speed camera, and then used for a calculation of wetting kinetics. After the tests, solidified couples were subjected to a detailed structural characterization by means of light microscopy (LM) and scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS).



**Figure 4:** An experimental procedure used for sessile drop method examinations of ultra-high temperature phenomena in Si/h-BN system.

The results of our research (more details are presented elsewhere [16]) demonstrated that increase in temperature improves wetting in the Si/h-BN system, and consequently, non-wetting-to-wetting transition takes place at around 1650°C (Fig. 5a). However, the contact angle of  $\theta = 90 \pm 5^\circ$  is maintained at temperatures up to 1750°C. Based on the results of conducted sessile drop experiments and structural characterization, it was documented that the high-temperature interaction between molten Si and h-BN is mainly controlled by a dissolution/precipitation mechanism. It means that h-BN is slightly dissolved (the dissolution rate depends on applied temperature) during a contact with molten Si at high temperature, what results in (i) a modification of chemistry of initially pure Si towards Si-B alloys (Fig. 5b) and (ii) a formation of gaseous nitrogen. Subsequently, new h-BN crystals are precipitated at the interface during cooling. What

is important, any wettable interfacial products were formed upon the ultrahigh temperature interaction. In a combination with a platelets-like morphology of reprecipitated h-BN crystals (Fig. 5c), it was reflected by a dewetting effect (i.e. a substantial increasing of contact angle) (Fig. 5a) during cooling from 1750°C. Since, overall behaviour allows for a preliminary assessment of h-BN as a good candidate for application in the Si-based LHTES system working at temperatures up to 1650°C, its performance upon a thermal cycling operation, has to be further examined.



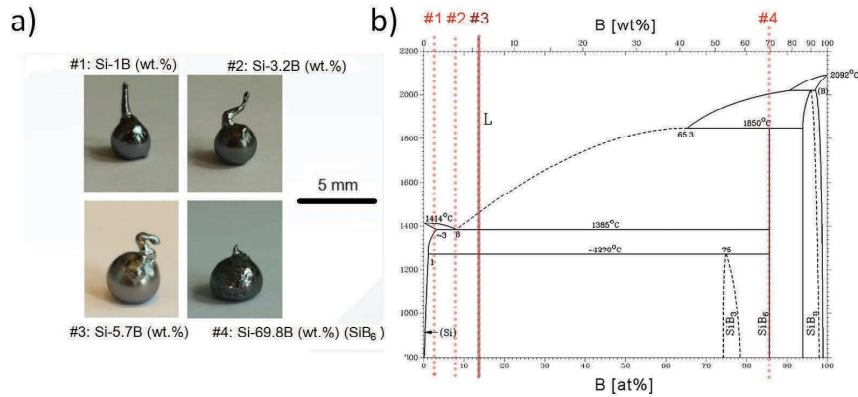
**Figure 5:** a) A wetting kinetic curve for Si/h-BN sessile drop couple. b) Microstructure of the cross-sectioned Si/h-BN sessile drop couple. c) Platelets like morphology of reprecipitated h-BN crystals revealed due to dewetting movement (more details are shown in [16]).

Subsequently, in order to evaluate the effect of boron addition on wettability in Si-B/h-BN system, a series of binary Si-B alloys were produced by a crucible-less electric arc melting method (in *IFW Dresden, Institute for Complex Materials, Germany*). Small specimens with a mass varying between 0.2 and 0.6 g (Fig. 6a), were fabricated from pure elements by melting pre-prepared mixtures having hypoeutectic, eutectic and hyper eutectic compositions, respectively (in accordance to the Si-B binary phase diagram [17]) (Fig. 6b).

The Si-B alloys were subjected to the same testing procedure as the pure silicon previously. The contact angle values obtained for Si, Si-1B, Si-3.2B and Si-5.7B (wt%) alloys on h-BN substrates at various testing temperatures are listed in Table 3 and compared with other reference Si/ceramics couples tested under the same conditions. What is clearly found is that the addition of boron to silicon lowers the wettability with h-BN – for each Si-B alloy the contact angle is stabilized at a very high level of ~130–150° in the whole temperature range (up to 1750°C). Furthermore, it should be noted that the highest values (around  $\theta=150^\circ$ ) were measured for hypo- and eutectic Si-B alloys. Therefore, it might be recommended that for the sake of ensuring a lack of wetting in Si-B/h-BN system it is beneficial to use near eutectic concentration of boron. Since, we have already discussed that the mechanism of high temperature interaction in Si/h-BN system is dominated by the dissolution/reprecipitation of h-BN platelets, the results obtained for Si-B alloys clearly point towards a hindering of dissolution of h-BN



ceramic in molten Si through additions of boron. Additionally, during cooling the Si-B alloys droplets were spontaneously detached from h-BN substrates, confirming a very low reactivity in the system.



**Figure 6:** a) Macroviews of exemplary Si-B alloys fabricated by electric arc melting. The Si-B binary phase diagram [17] with marked nominal compositions of fabricated alloys.

**Table 3:** Contact angle  $\theta$  values measured by sessile drop method for Si and Si-B alloys on h-BN substrates and the other reference ceramic materials.

	Si/h-BN	Si-1B/h-BN	Si-3.2B/h-BN	Si-5.7B/h-BN	Si/SiC	Si/ZrO <sub>2</sub> *
T [°C]	contact angle values after 5 min test, $\theta$ [°]					
1450	115	147	146	145	43	100
1550	97	150	144	143	45	96
1650	94	150	147	136	44	96
1700	92	152	150	134	44	92
1750	90	151	151	131	0	92

\* During the test, a substantial drop movement (“a dancing drop” effect) was observed as the direct effect of the formation of volatile oxide products.

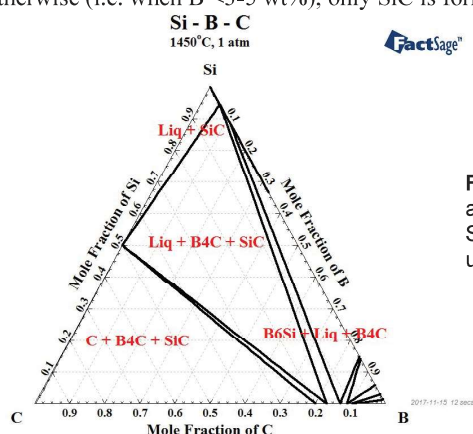
### “Self-crucible” approach (NTNU)

As opposite to the assumptions described above of negligible reactivity between contacting materials, principles of the latter approach include a reactive formation of continuous interfacial product layer at initial stages of PCM/refractory interaction. The reactively formed layer should play a role of barrier coating, and thus presents a “self-crucible” inside the PCM vessel.

In order to realize this idea, the NTNU team has decided to choose graphite ( $C_{gr}$ ) as the potential refractory material for PCM container. Graphite is widely used in the fabrication and processing of photovoltaic grade silicon due to its favorable set of thermophysical properties including compatible thermal expansion with Si, good thermal shock resistance, high thermal conductivity, and excellent machinability [18]. Nevertheless, a present selection of this material is justified mostly by a well-recognized behavior in molten Si/ $C_{gr}$  system leading to the formation of SiC interfacial continuous product layer [18]. The involved reaction between molten Si and  $C_{gr}$  originates from a lack of chemical equilibrium in the system – the interaction is not terminated until either

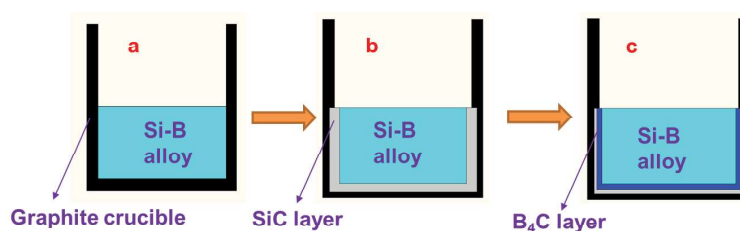


the melt completely infiltrates into the porous graphite substrate, or the SiC layer is produced. The competition between infiltration and formation of the product layer is governed by many factors, e.g. a size and shape of pores, surface roughness, temperature and contact time [19]. Furthermore, as it comes from our thermodynamic assessment of phase equilibria in the Si-C-B ternary system (performed by using FactSage database - Fig. 7), addition of boron strongly contributes to a type of products formed at the interface. It was estimated that when the content of boron in molten Si-B alloy is higher than 3-5 wt%, the interaction with graphite goes in two stages including the formation of product layer composed of (i) SiC and (ii) B<sub>4</sub>C layers (Fig. 8). Otherwise (i.e. when B < 3-5 wt%), only SiC is formed at the interface.



**Figure 7:** A thermodynamic assessment of phase equilibria in the Si-C-B ternary system (performed by using FactSage database).

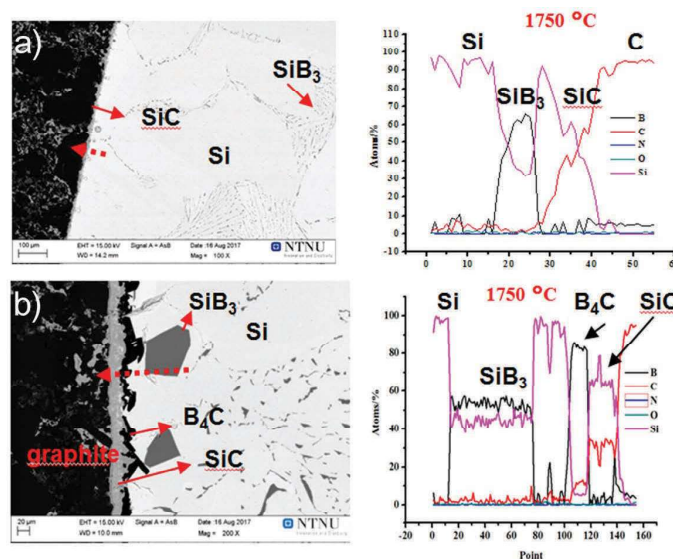
In order to validate these thermodynamic calculations, a number of Si-B alloys having the nominal boron content of 2, 3, 5, 8, 11, 15 and 25 (wt%) were produced by induction melting of pure elements inside graphite crucibles, in argon atmosphere, at temperatures between 1450 and 1750°C. The molten Si-B mixtures were held for 2 hours at selected processing temperatures, then cooled down to room temperature and subjected to microstructural evaluation (Fig. 9).



**Figure 8:** The interaction stages between Si-B alloys and a graphite crucible. When the content of boron in molten Si-B alloy is higher than 3-5 wt %, the interaction with graphite goes in two stages (a→b→c) [20].

Regarding the interface between the alloy and the crucible, it is found that a thin SiC layer is created (Fig. 9a). At high B contents, B<sub>4</sub>C layer was also produced at the surface of the crucible (Fig. 9b). This layer probably originates from the reaction of SiC with B near the crucible surface. When B<sub>4</sub>C is produced it consumes a lot of B in the alloy and

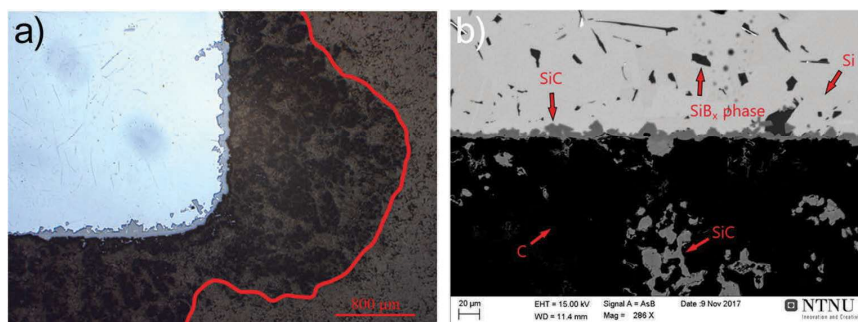
the B content in the alloy is decreased. It should be noted that some of the  $B_4C$  was found also in the alloy phase. At low B contents (less than 5 wt%),  $SiB_6$  and  $B_4C$  are not present and the alloy is relatively clean.



**Figure 9:** SEM images and EDS profiles of the interface between Si-B and  $C_{gr}$  for two Si-B compositions: (a) Si-2wt% B and (b) Si-8wt%B. Experiments were carried out up to 1750°C. For the larger B content (bottom), a double layer of SiC/ $B_4C$  is formed at the interface. For the lower amount of B, only SiC is formed [20].

Subsequently, long-term high temperature annealing experiments (up to 48 h) were performed to examine thermal stability of Si-B alloys and interfacial products. Near eutectic compositions of Si-B alloys were selected for these tests, since they are also beneficial in terms of relatively lower melting temperatures combined with even higher latent heat than that of pure Si. It was documented that molten Si(B) penetrates the crucible and produce SiC quite fast (Fig. 10), and after that the reaction is very slow. By taking into account that the Si(B) penetration strongly depends on the porosity of graphite, a dense crucible is needed. Besides, as observed during the short term experiments, a dense SiC barrier layer is produced on the surface of the crucible, which decreases the further reactions. It was found that, this layer may rarely leave the crucible surface and be found in the alloy. Nevertheless, during the experiments the eutectic alloy remained quite clean from SiC at the eutectic composition. Additionally, we observed that cycling of temperature, above the melting temperature, does not affect the alloy composition. Therefore, when  $C_{gr}$  crucibles are applied, the Si-B alloy composition should be within the area of the eutectic composition (3 wt% B), e.g. between 2-5 wt% B, due to a suppressed formation of  $SiB_6$  and  $B_4C$  solid particles. A lack of solid particles should be reflected in the following beneficial effects:

- 1) an increase of latent heat of fusion per volume unit;
- 2) no boron depletion of Si-B alloy;
- 3) a more controlled solidification process;
- 4) viscosity and other parameters are determined by the melt properties only.



**Figure 10:** Eutectic Si-B alloy/  $C_{gr}$  interface after 48h of interaction at 1550°C.

## Conclusions

In order to provide practical recommendations for the selection of crucible material for Si and Si-B based LHTES systems working at ultra-high temperatures, two distinctly different approaches have been explored: one assuming an inertness between contacting PCM and refractory, while the other was based on a reactively formed interfacial barrier layers. Regarding the former attempt, it was documented that h-BN is the only one ceramic fulfilling criteria of the non-wetting and negligible reactivity with molten silicon at temperatures up to 1650°C. Additionally, it was found that Si-B alloys exhibit even lower wetting with h-BN at temperatures such high as 1750°C, as compared to pure silicon. The decreased reactivity originates from hindering of dissolution of h-BN ceramic in boron enriched silicon alloys.

In the later approach, graphite proved its ability to form a barrier layer composed of SiC or  $(SiC+B_4C)$ , depending on boron content in PCM. The *in-situ* formed products showed also a high thermal stability upon short and long term (up to 48h) holding at temperatures up to 1750°C. Therefore, both dense graphite and SiC crucibles could be used as container material. As both carbon and SiC wet the alloy, over time a thin silicon layer should cover the crucible. In other processes, this has been stopped by adding a non-wetting band in the top of the crucible (e.g. h-BN).

Since, at the early stage of the AMADEUS project, both considered attempts seems to be reasonable (but each having some advantages and disadvantages), the final selection of materials requires further study on behavior of selected PCM/refractory couples upon cyclic melting/solidification steps.

## Acknowledgments

The project AMADEUS has received funds from the European Union's Horizon2020 research and innovation program, FET-OPEN action, under Grant agreement 737054. The sole responsibility for the content of this publication lies with the authors. It does not necessarily reflect the opinion of the European Union. Neither the REA nor the European Commission is responsible for any use that may be made of the information contained therein.

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