Micro-encapsulation of Fe-Si ultra-high temperature phase change material

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Abstract — **In this work, a micro-encapsulation approach was introduced for the first time to a binary Fe-Si phase change material. The alloy composition was developed through a thermodynamic modelling. After that, a multistep processing was designed and applied to fabricate spherical microcapsules having a structure of SiO_2 shell and the Fe-Si alloy core. Finally, basic thermophysical properties of encapsulated Fe-Si powders were experimentally validated in DSC studies.**

I. Introduction

Latent heat thermal energy storage (LHTES) has been recognized as an attractive method for supporting renewable energy sources in terms of energy availability during blackout periods [1]. One of the main features of each LHTES system is a phase change material (PCM), i.e. a substance that can absorb and release energy (as a heat) on demand. Thermophysical properties of PCM (including heat of fusion, thermal conductivity and volumetric expansion) determine the overall performance of the energy storing unit [2].

In our recent projects, we have developed a ternary eutectic Fe-26Si-9B (wt%) alloy as a new candidate to be applied in ultra-high temperature LHTES system [3]. The alloy utilizes a concept of using very high latent heat of both pure Si and B, while Fe is added to ensure a low volumetric thermal expansion upon solid↔liquid phase changes. Nevertheless, as application of PCMs at ultra-high temperatures brings several intrinsic challenges, some new material design approaches ought to be taken into consideration, in order to further increase durability and accessibility of LHTES devices.

A. *From Fe-Si-B to Fe-Si based PCMs*

Although the Fe-26Si-9B alloy has proved its high applicability as high temperature PCM, there are still some economic and technological issues that ought to be solved. One of them is related with a very high price and geographically limited availability of boron [4]. Furthermore, addition of boron increases a reactivity with most of carbon-, oxide- or nitride-based refractory ceramics, what makes a selection of materials for PCM container quite limited. Therefore, in this work we propose to impose the following new strategies:

- A transition from ternary eutectic Fe-Si-B alloy towards B-free binary eutectic Fe-Si alloys, to decrease potential costs and technical difficulties coming from using of boron.
- An implementation of “micro-encapsulation” approach in order to provide a possibility for using various containers materials and to increase a lifetime of high temperature PCMs.

B. *Micro-encapsulation concept*

A widely documented high reactivity of Si-containing melts should be considered as detrimental in terms of potential premature degradation of the PCM’s as well other components of the LHTES devices. In this regard, a concept of “micro-encapsulation” of a PCM through the pre-oxidation treatment appears as a promising path to develop highly durable LHTES solutions. The basic concept behind this process consists of at least two steps:

(i) a fabrication of the alloy in the form of spherical powders,

(ii) a thermochemical heat-treatment focused on a development of continuous and stable shell/core like structure.

Indeed, it has been recently documented by Han et al. [5] that the micro-encapsulation of the Al-based PCMs can provide excellent cyclability in terms of both the latent heat and melting point values in consecutive melting/solidification phase changes, even up to 3000 operational cycles.

In this work, we propose for the first time to use this approach to ultra-high temperature PCMs from the Fe-Si system.

II. Materials and methods

A. *Material selection and processing*

The material investigated was a Fe-Si alloy developed and selected by using thermodynamic modelling in FactSage 8.1. The alloy was produced by an induction melting of a mixture of commercially available ferrosilicon and metallurgical grade Si (Elkem, Norway). The materials processing route is schematically shown in Fig. 1. Firstly, the alloy ingots were transformed into spherical powders by using novel ultrasonic assisted atomization technique provided by the AMAZEMET rePowder system (Warsaw, Poland). After that, the powders were subjected to a two-step thermo-chemical treatment that included:

(i) an impregnation of powders with silica suspension and;

(ii) a high temperature heat treatment (annealing in air).

The main purpose of both steps was to activate the surface of particles and then to build up continuous stable SiO_2 layer protecting the PCM alloy against structure/properties degradation.

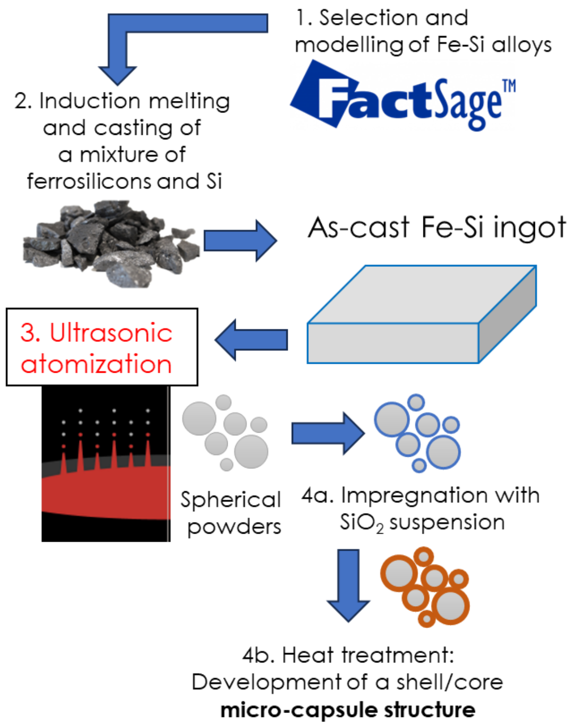

Fig. 1. A scheme of the applied materials processing.

B. Materials characterization

At every stage of the process, microstructure of involved materials was characterized by using light and scanning electron microscopy coupled with the X-ray Energy Dispersive Spectroscopy (SEM/EDS) method. A special attention was given to qualitative and quantitative analysis of the effect of processing on the chemical composition and morphology of the developed oxide shell.

Thermophysical properties of the as-cast Fe-Si alloy, atomized and encapsulated powders, were examined and compared in differential scanning calorimetry (DSC) experiments. The DSC technique was used to evaluate melting /solidification behavior of the materials in terms of temperature ranges, as well as to quantify the latent heat of fusion.

Furthermore, thermocycling experiments, i.e. consecutive heating/cooling cycles, were performed for the sake of assessing cyclability and stability of the developed PCM microcapsules.

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