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Processing of open-pore silicon foams using graphite composite as space holder

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Abstract

In this study, a novel space holder is used for the fabrication of Si foams. The space holder is a graphite composite (GC) with regular spheroidal cells. Conventional space holder materials are not suitable for casting Si foams. The main issue in this context is the differences between the melting point of the foam's base material and the one of common space holders. GC offers many advantages as e.g. low cost, good dissolution by oxidation, high melting point and non-toxicity. This type of space holder is chemically stable when being in contact with liquid and solid Si, and it is, hence, suitable for a melt metallurgical processing route. Si foams are manufactured by applying replication casting. The resulting foam geometry and its surface are analyzed using LM and SEM. XRD measurements are performed to investigate the quality of the Si foam in terms of contamination by graphite or oxygen.

Keywords: Metal foam; Space holder; Silicon; Graphite; Infiltration

1. Introduction

Metal foams are a special type of engineering materials. The combination of base metal and cellular structure shows great potential for a wide range of applications in energy absorption [1], thermal engineering [2] and lightweight constructions [3]. Common manufacturing techniques for metal foams are: sintering, using a blowing agent, investment casting and replication casting [4–6]. A melt metallurgical processing route is usually used for fabrication of open-pore metal foams. Primarily the porosity and morphology of the open-pore foam is defined by a perform. In the case of investment casting, typically a reticulated polymer foam is used as precursor. It is embedded in a ceramic slurry, burned out and infiltrated with molten metal [7–10]. However, this method requires a high effort in time and material. Another melt metallurgical processing route is the replication casting technique. A space holder material is placed in a mold that gets infiltrated with the molten metal. In a next step, the space holder is removed leaving an open-pore metal foam. The space holder has to be insoluble and unreactive with the molten and solid metal while showing good solubility in a solvent or acid that does not react with the metal [10–12].

Energy-efficiency and energy-storage are mayor research fields in science, engineering, and technology in the future [13]. Si has been the most promising candidate for the next-generation anode materials, expressing a theoretical specific capacity of 4200 mAh g^{-1}, which is much higher than the commercial graphite anode. Hence, there are currently many developments using Si foams for energy applications [14–16]. Another very promising application for Si foams is the thermoelectricity. Thermoelectric materials show outstanding properties due to their ability to convert waste heat into electricity [17–19]. Of many different alloys and compounds belonging to this class of materials, Mg_2Si is a promising candidate. A new manufacturing approach requires Si foam as a precursor that is infiltrated by molten Mg followed by a thermal treatment to form the thermoelectric material Mg_2Si [20]. These examples in application show the need of the porous Si structures. The aim of the present study is to investigate the manufacturing process of open-pore Si foams by the melt metallurgical route. For this purpose, we

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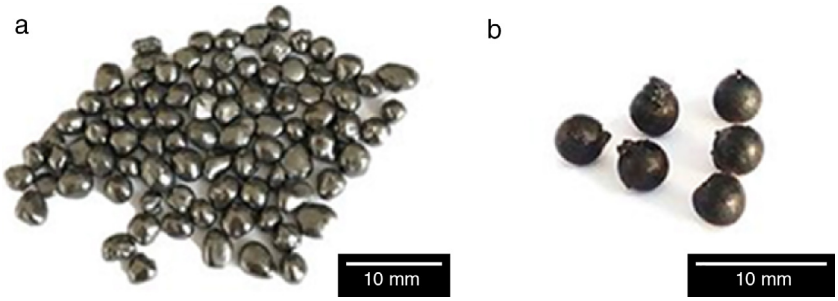

Fig. 1. Space holder spheres made of (a) graphite particles, (b) graphite composite.

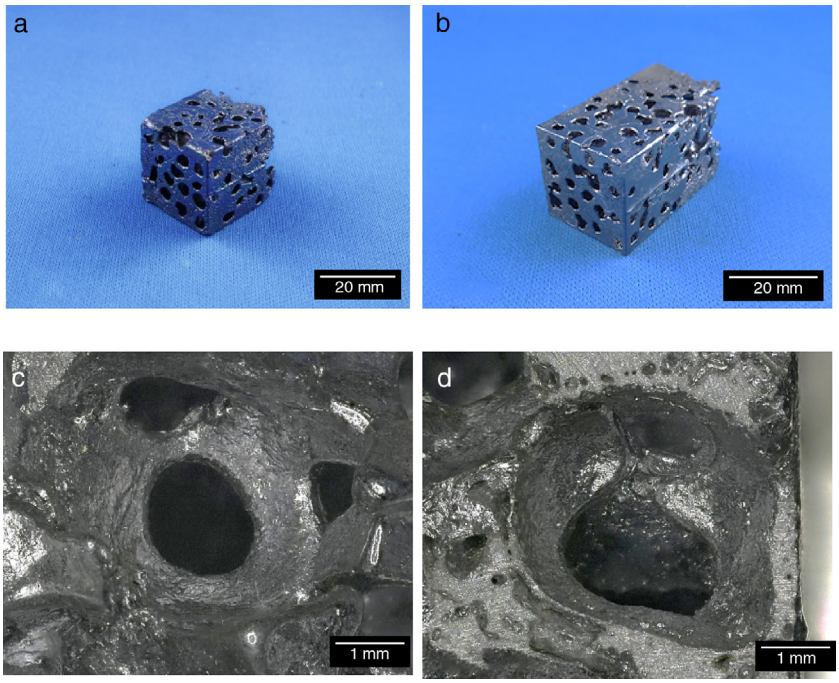

Fig. 2. Digital image of open-pore Si foams (a) using graphite particles and (b) graphit composite as space holder. Cross section of the produced foams showing the cell geometrie in (c) and (d).

investigate graphite as a space holder to produce replicated open-pore Si foams.

2. Material and methods

In this study, pure graphite (99.8%) spheres (Fig. 1(a)) and graphite based composite (GC) were used as space holder materials. Both types of space holder have a spheroidal shape and a size of 4 mm in diameter. The fabrication of GC was performed using graphite particles (purity of 99.8%) in a range of $0.2\,\text{mm} < d < 0.5\,\text{mm}$ (supplied by Thielmann Graphite GmbH & Co. KG, Germany) that were blended with an inorganic binder. This mixture was put in silicone spheroidal and 4 mm diametric molds to achieve the desired shape of the space holders. Subsequently, the mixture of graphite and inorganic binder was dried out. Fig. 1(b) shows the final GC after the removal of the mold in comparison to pure graphite spheres.

Open-pore Si foams were produced by centrifugal casting (Vacutherm-3,3-Titan from Linn High Therm GmbH, Germany). This is carried out in five steps: (1) melting of Si, (2) infiltration of the mold filled by the space holder, (3) cooling, (4) removal of the space holder and (5) a chemical aftertreatment. The material used for the infiltration procedure was Si with a purity of ≥99.99% (supplied by ALD Vacuum Technologies GmbH, Germany). Melting of Si was carried out inductively in argon inert gas atmosphere under 0.5 bar gauge pressure, after previous evacuation of the melting chamber to 0.1 mbar. Inert gas was used to avoid any reaction of graphite with ambient air. Si was placed in a preheated crucible and heated to a temperature of $\vartheta = 1600\,^{\circ}\text{C}$. Centrifugal casting velocity was 500 rpm, achieving a flow pressure of the melt of approx. 33.7 bar. Previous to infiltration, the space holder particles are placed into a two-piece mold made of graphite. This was pre-coated by boron nitride on the inner walls and preheated to $\vartheta = 200\,^{\circ}\text{C}$ for $t = 1$ h. After infiltration, cooling was carried out in 20 °C ambient temperature. In order to remove the space holder material, a heat treatment of $\vartheta = 800\,^{\circ}\text{C}$ for $t = 4$ h in normal atmosphere was applied. In a following step, the open-pore foam is treated by aqueous Hydrogen Fluoride (7%) at a temperature of $\vartheta = 35\,^{\circ}\text{C}$ for $t = 240$ min, this is to ensure the removal of SiO_2 layer on the