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Structural characterization and strengthening mechanism of forsterite nanostructured scaffolds synthesized by multistep sintering method

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ABSTRACT

In this study, highly porous forsterite scaffolds with interconnected porosities were synthesized using multi-step sintering (MSS) method. The starting powder was nanosized forsterite, which was synthesized from talc and magnesium carbonate powders. The phase composition, average particle size and morphology of the produced forsterite powder were characterized by X-ray diffraction technique (XRD) and transition electron microscopy (TEM). Forsterite scaffolds were produced by foamy method using polymeric sponges. MSS process including three steps was used to efficiently sinter the forsterite nanopowders without destroying the initial porous structure of polymeric sponges. The results showed that MSS technique is an efficient and appropriate procedure to produce highly porous forsterite scaffolds with pore size in the range of 100–300 μm. The compressive strength, compressive modulus and porosity of C12 specimen (sintered at 1650 °C for 1 h with subsequent annealing at 1000 °C for 1000 min) was 1.88 MPa, 29.2 MPa, and 72.4%, respectively, which is very close to that of cancellous bone. The approach studied in this research can be developed for other nanostructure ceramics to produce highly porous scaffolds with interconnected porosities for load bearing applications.

1. Introduction

Different techniques have been developed to synthesize bone scaffolds for load bearing applications in bone defects [1]. Bioceramics, such as calcium phosphate ceramics, are an ideal choice due to the close chemical makeup of bone; however, there are several weaknesses that have to be addressed [2]. A common problem is the lack of strength as a result of grain growth, which happens during the sintering process of scaffolds at high temperatures for a long period of time [3].

The overall objective of the sintering process is to increase the strength of the scaffolds [4]. The high temperature of the sintering process causes the grains in the structure to grow, which then weakens the structure [5,6]. There are several different methods of sintering such as conventional sintering [7], spark plasma sintering [8], microwave radiation [9], hot pressing and sinter forging [10]. These sintering techniques either provide extensive grain growth due to the high temperatures, are expensive, or require expensive equipment [7–14]. A new technique that has been developed is two-step sintering, which starts by heating the sample at a high temperature, and then by rapidly decreasing the temperature to a lower temperature and holding at that temperature for a long period of time. The rapid decrease in temperature stops the grain growth while allowing the grain boundary diffusion to take place; however, the thermal shock introduced to the structure may initiate some micro-cracks which in turn deteriorates the final mechanical properties of the structure [15–28].

Forsterite, Mg_2SiO_4, is a compound that has high strength, is considered chemically stable, and has a low thermal expansion rate [29–33]. These characteristics, as well as the fact that it is biodegradable in the form of nanostructure, contributes to the good potential of this compound for bone tissue engineering applications [34–40]. The nanostructure allows for an increase in strength and hardness, as well as reduction in sintering times and a decrease in

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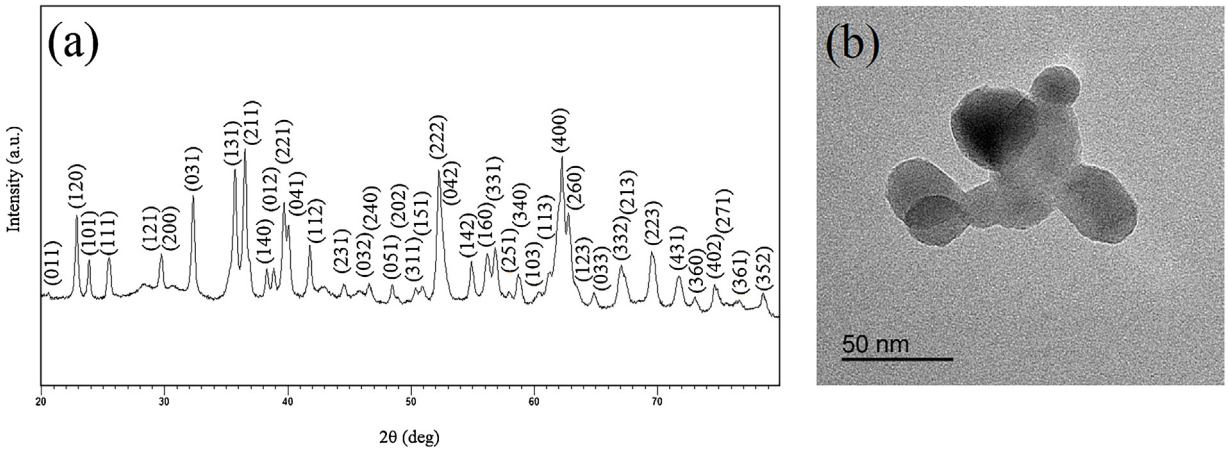

Fig. 1. (a) XRD pattern and (b) TEM micrograph of forsterite powder.

diffusion temperature [29–34]. On the other hand, nanostructure forsterite is bioactive and forms a bonelike apatite layer on its surface in simulated body fluid, while micron size forsterite is bio-inert and has a negligible interaction with physiological environment [41].

Synthesis of forsterite by two-step sintering method is reported previously by Mirhadi [38]; however, the porosity of the produced forsterite scaffolds was 58% which is not acceptable for biomedical applications. The aim of the present study is to fabricate highly porous forsterite scaffolds with interconnected porosities and desired strength for load bearing applications. Scaffold parameters such as pore size, porosity and pore distribution can be controlled in order to facilitate the mass transport of oxygen and nutrients into the interior of the scaffold which eventually supports cellular growth in that region [42]. An ideal scaffold for bone tissue engineering should have the pores size similar to trabecular bone with the mean pore diameter varying from 300 to 1500 μm. However studies have shown that pore size in a range of 50–400 μm is optimal for ceramic implants due to its higher mechanical stability [43]. Other studies reported better osteogenesis for scaffold with pore size > 300 μm [44]. Despite from different reported optimum pore size numbers in literature, the scaffold should sustain adequate mechanical properties. By using multi-step sintering (MSS) technique, a uniform scaffold with high strength and porosity can be created. MSS process with controlled heating/cooling rates would increase the strength of the scaffolds which allows the scaffold to work in areas of the body that undergo high stress conditions. On the other hand, the bioactivity and biodegradability of forsterite will provide a biological fixation in the body and simultaneously allow the material to be replaced by new bone growth.

2. Materials and methods

The initial substances used to produce forsterite powder were Talc ($(Mg_3Si_4O_{10})(OH)_2$) (99% purity, Sigma – Aldrich) and magnesium carbonate ($MgCO_3.xH_2O$) (99% purity, Bulk Supplements). Forsterite was produced based on the procedures described in [29]. A molar ratio of 1 mole of Talc for every 5 mol of magnesium carbonate was used. A vertical planetary ball mill (Tencan, Model: XQM-1-A) was utilized to combine the initial powders for 15 h in

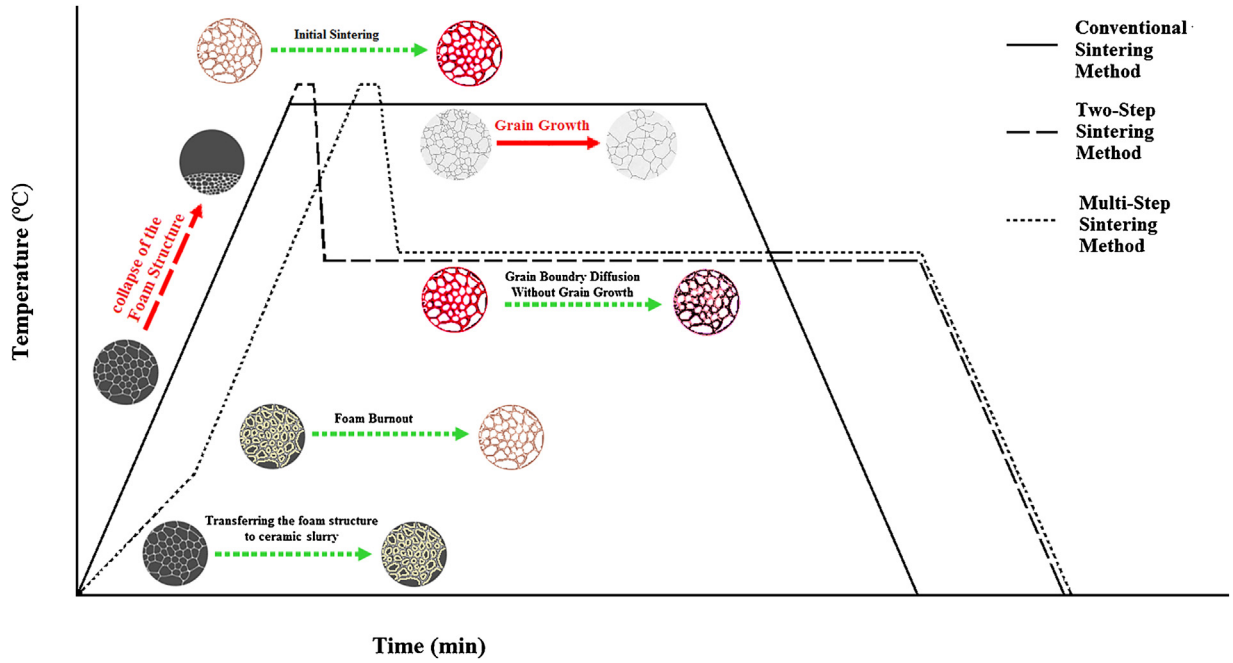

Fig. 2. A schematic diagram of sintering process in conventional, two-step and multi-step sintering methods.

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