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# Effect of sintering conditions on the microstructural and mechanical characteristics of porous magnesium materials prepared by powder metallurgy

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ABSTRACT

There has recently been an increased demand for porous magnesium materials in many applications, especially in the medical field. Powder metallurgy appears to be a promising approach for the preparation of such materials. Many works have dealt with the preparation of porous magnesium; however, the effect of sintering conditions on material properties has rarely been investigated. In this work, we investigated porous magnesium samples that were prepared by powder metallurgy using ammonium bicarbonate spacer particles. The effects of the purity of the argon atmosphere and sintering time on the microstructure (SEM, EDX and XRD) and mechanical behaviour (universal loading machine and Vickers hardness tester) of porous magnesium were studied. The porosities of the prepared samples ranged from 24 to 29 vol.% depending on the sintering conditions. The purity of atmosphere played a significant role when the sintering time exceeded 6 h. Under a gettered argon atmosphere, a prolonged sintering time enhanced diffusion connections between magnesium particles and improved the mechanical properties of the samples, whereas under a technical argon atmosphere, oxidation at the particle surfaces caused deterioration in the mechanical properties of the samples. These results suggest that a refined atmosphere is required to improve the mechanical properties of porous magnesium.



## 1. Introduction

Magnesium and magnesium alloys have recently been studied for use in many applications, such as in the automotive and aerospace industries because of their low densities and good mechanical properties [1]. Many successful studies have been performed on biocompatible and biodegradable magnesium-based materials, which are considered suitable materials for orthopaedic applications, such as for nails, screws, splints, etc. [2–6]. For some applications, porous implants, called scaffolds, are required because they possess mechanical properties, such as the modulus of elasticity, that are relatively similar to those found in natural bone tissue [7–14]. The mechanical biocompatibility is important to aid in the remodelling of new tissue [7–14]. Moreover, an interconnected porous structure allows the transport of body fluids to damaged or wounded tissue and supports the incorporation of new tissue in the implant [7,15,16]. Therefore, porous magnesium materials and their preparation methods have been extensively studied in recent years [7–14].

Many methods have been developed for fabricating porous metallic materials [17]. However, because biomaterials should contain interconnected pores [12] and should not be contaminated with harmful impurities [4], only a few of these methods are used for the production of biomaterials. In the available literature, there are five "non-machining" approaches that have been reported for the fabrication of porous magnesium materials: (1) injection of an inert gas into a melt [4], (2) directional solidification of the metal–gas eutectic (the GASAR process) [9], (3) plaster casting [4], (4) negative salt pattern moulding [12] and (5) powder metallurgical techniques [11,13,14]. However, the first two methods mentioned above do not necessarily produce open-cell structures, and the following two methods may contaminate or corrode the final product during pattern removal [18]. Suitable modifications to powder metallurgy (PM), for example, using space-holder particles, allow the fabrication of materials with interconnected pores. This modification consists of preparing a green compact that contains a powdered mixture of magnesium and a space-holder material, which is later removed by leaching or thermal decomposition. Subsequently, the porous green body is sintered at high temperatures [8,10,13,14]. In principle, any solid matter may be used as the space-holder material; however, in practice, this selection is limited because the spacer material has to be removed without contaminating the product. In the literature, urea and ammonium bicarbonate have been successfully used as spacer materials in the preparation of PM porous magnesium [8,11,13,14,19]. Hao et al. [8] removed the urea by leaching the material in a NaOH-water solution, whereas other authors removed the space-holder particles by thermal decomposition [10,11,13,14]. The majority of these authors used urea as the spacer material, even though its complete decomposition occurs at temperatures above the melting point of magnesium. Because urea only partially decomposes at lower

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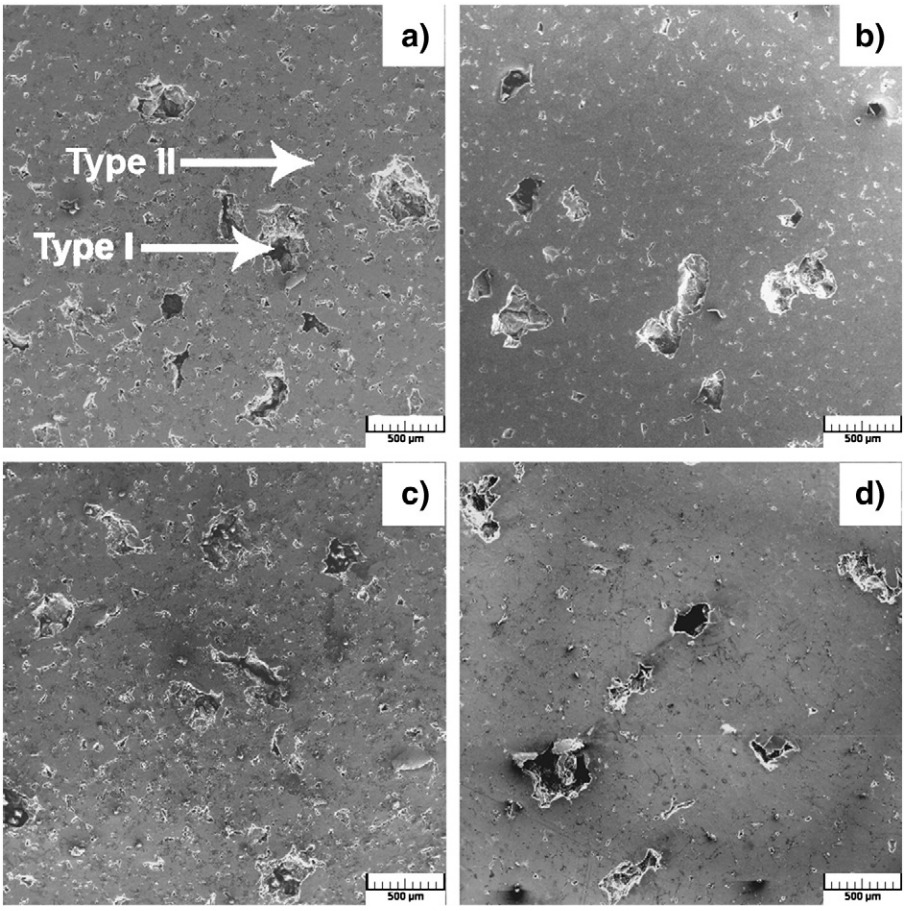


**Fig. 1.** SEM micrographs (SE detector) of sample cross sections prepared under the following sintering conditions: (a) technical argon for 6 h, (b) technical argon for 24 h, (c) gettered argon for 6 h and (d) gettered argon for 24 h.

temperatures, contamination may occur [20]. For example, Zhuang et al. [13] found traces of carbon and oxygen on pore walls, which they attributed to contamination by urea residues. Ammonium bicarbonate, which decomposes at significantly lower temperatures (36 °C–60 °C), appears to be a more suitable spacer material for the preparation of PM porous magnesium. In addition, leaching the spacer material has the disadvantage of possibly hermetically enclosing some of the spacer particles in the magnesium matrix, which prevents their dissolution. Moreover, water-based dissolving agents are corrosive to magnesium [21].

The mechanical properties of PM porous magnesium are influenced not only by the total porosity but also by the pore size, distribution and shape and the connection between magnesium particles [8,10,13]. These structural characteristics can be adjusted by selecting the optimal sintering time and temperature, compacting pressure and size, shape and volume ratio of the starting material powders [8,11,13,19,22]. Grain coarsening within each magnesium particle, which is expected during the sintering process, may also affect the mechanical properties of the final material [23,24].

In our previous work [19], we showed that porous magnesium can, in principle, be successfully prepared by PM using ammonium bicarbonate as the space-holder material. Although several works on the preparation of porous magnesium have been reported, to the best of our knowledge, no systematic study on the influence of the processing parameters, namely, the sintering time and atmosphere purity, on the microstructure and mechanical properties of porous magnesium is available. Therefore, our study focused on sintering kinetics and the effect of the sintering atmosphere on the important characteristics of PM porous magnesium.

**Table 1**
The influence of sintering conditions on sample porosity (in vol.%).

| Atmosphere/sintering time | 0 h | 3 h | 6 h | 12 h | 24 h |
|---|---|---|---|---|---|
| Technical argon | 29 ± 2 | 28 ± 1 | 28 ± 2 | 29 ± 3 | 31 ± 4 |
| Gettered argon | 29 ± 2 | 28 ± 1 | 27 ± 2 | 25 ± 3 | 24 ± 2 |

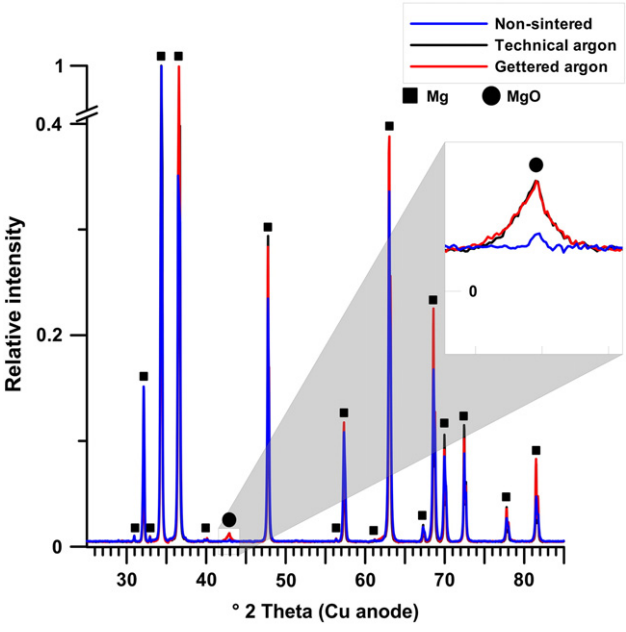


**Fig. 2.** XRD patterns of samples sintered for 6 h.