Influence of heating rate on two-step sintering behaviour of different hydroxyapatite nanopowders

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INTRODUCTION

Producing of dense nanostructured calcium phosphate-based bioceramics represents a challenging issue in biomaterial science. High volume fraction of energetically rich grain boundaries contributes to improved attachment of chemical species, which are important in the processes of bone tissue regeneration. Besides that, nanostructured ceramics exhibited better mechanical properties due to changed fracture path. The process of pressureless sintering is the most compatible route for industrial fabrication of dense bioceramic materials, but it is often connected with accelerated grain growth in final sintering stage. In the method of two-step sintering (TSS), the difference between kinetics of grain boundary diffusion and grain boundary migration is used to obtain almost full dense, nanostructured ceramics. However, designing of proper sintering parameters is very important in every sintering technique employed.

In this study, hydroxyapatite nanopowders were synthesized by different methods, precisely, hydrothermal processing of precipitate and chemical precipitation. The prepared powders were pressed in pellets and heated with different heating rates, with short isothermal dwell at certain temperature range. From that shrinkage curves the appropriate conditions were selected to design TSS experiments. The impact of heating rate on final density, phase composition, average grain size and microstructural uniformity is discussed.

RESULTS AND DISCUSSION

The obtained results are presented in the Table 1. SEM analysis of HAp 1 and HAp 2 nanopowders, together with TSS processed ceramics and its corresponding XRD patterns, are presented on Fig. 1 and Fig. 3, respectively. Fig. 2 represents the dependence of the average grain size for HAp 1 nanopowder on heating rate. It should be noticed that HAp 1 nanopowder exhibited higher crystallinity and lower agglomeration degree than HAp 2, which resulted in lower sintering temperatures for HAp 1.

Table 1. Characteristics of synthesized powders and conclusions from sintering experiments.

<table>
<thead>
<tr>
<th>Synthesized powder</th>
<th>HAp 1</th>
<th>HAp 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase composition</td>
<td>Hydroxyapatite</td>
<td>Hydroxyapatite</td>
</tr>
<tr>
<td>Morphology/Particle size (nm)</td>
<td>Elongated/length=100,width=50</td>
<td>Spherical/55</td>
</tr>
<tr>
<td>SSA (m²/g)</td>
<td>51</td>
<td>72</td>
</tr>
<tr>
<td>Does transformation of HAp to β-TCP occur during heating?</td>
<td>YES</td>
<td>NO</td>
</tr>
<tr>
<td>Does heating rate change influence microstructural characteristics?</td>
<td>YES</td>
<td>NO</td>
</tr>
</tbody>
</table>

Fig. 1. SEM micrographs of: a) HAp 1 nanopowder and sintered ceramics via TSS 2 cycle by following heating rates b) 2, c) 5 and d) 10 °C/min; e) XRD patterns of these systems.

CONCLUSION

1. Nanostructural HAp could be synthesized via both hydrothermal processing, HAp 1, and chemical precipitation, HAp 2.

2. The sintering behaviour is significantly influenced by powder characteristics and degree of agglomeration. HAp 1 could be sintered to full density at lower temperatures than HAp 2, indicating better sinterability of hydrothermally obtained material.

3. TSS method is useful approach in sintering nanopowders, leading to preserved fine-grained microstructure. However, starting powders characteristics are of the great importance.

4. In the systems occuring phase transformation from HAp to β-TCP, heating rate strongly affects microstructural uniformity and grain size, but the final density, too. For HAp 1 nanopowder, by increasing heating rate from 2 to 10 °C/min, the average grain size is almost 50 % decreased, following first order exponential decay. If there is no detectable phase transformation, no significant microstructure changes were observed, HAp 2.

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