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Freeze-casting to create micro-channels in La$_{0.66}$Ca$_{0.33-x}$Sr$_x$Mn$_{1.05}$O$_3$

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Introduction

The templating technique of freeze casting is utilized as a way of creating directional porosity in the form of micro-channels in La$_{0.66}$Ca$_{0.33-x}$Sr$_x$Mn$_{1.05}$O$_3$ (LCSM). LCSM is a magnetocaloric material in which the Curie temperature can be controlled by varying strontium doping (x), making it ideal for application as a regenerator material in magnetic refrigeration [1]. One way to increase the cooling performance of a magnetic regenerator is by optimizing its geometry: while maintaining a large surface area to increase heat exchange with the regenerator fluid, it must not provide too much resistance to flow. It has been proposed that a matrix of micro-channels with a width of 100 µm gives optimum performance [2].

Freeze-casting results in channels of widths of 10 to 100 µm, where the porosity depends on the solid load while the size and homogeneity of the channels depends on freezing conditions [3][4].

The figure on the right shows solid magnetic regenerators made of magnetocaloric materials of packed irregular particles and stacked plate geometries. A geometry in-between – such as a micro-channel matrix – would be optimum.

Objectives

- Fabrication of LCSM ceramics with micro-channels by freeze casting
- Increase homogeneity of channels along the height of the sample by implementing dynamic freezing profiles instead of static freezing profiles
- Alter morphology of channels by gelation freeze casting, i.e. using gelatin to create a stable gel suspension before the freezing step

Materials and methods

The standard freeze casting route is altered in two steps:

- Dynamic and static freezing: Samples were frozen either statically at -90°C or dynamically at -10 °C/min.

Gelation freeze casting: Gelatin was added at 0.3 wt% (oil solids) and left to harden before freezing

Powders were used as received from Cer-Po-Tech and characterized as follows:

<table>
<thead>
<tr>
<th>s/cm</th>
<th>Density [g/cm³]</th>
<th>Surface area [m²/g]</th>
<th>pH</th>
<th>Zeta potential [mV]</th>
</tr>
</thead>
<tbody>
<tr>
<td>LCSM006</td>
<td>0.96</td>
<td>0.0911</td>
<td>10.12</td>
<td>0.008</td>
</tr>
<tr>
<td>LCSM007</td>
<td>0.87</td>
<td>0.8991</td>
<td>17.02</td>
<td>0.024</td>
</tr>
<tr>
<td>LCSM0075</td>
<td>0.075</td>
<td>4.9603</td>
<td>0.014</td>
<td>6.052</td>
</tr>
</tbody>
</table>

Results: Varying particle size

SEM images showing the cross section perpendicular and parallel to the freezing direction of 14 vol% LCSM006 frozen with no control of temperature.

Results: Dynamic and static (gelation) freezing

Slimes of 20 vol% LCSM were freeze casted without control of temperature besides that of liquid N$_2$.

Results: Copper rod immersed directly in liquid N$_2$

Slimes of 14 vol% LCSM were freeze casted without control of temperature besides that of liquid N$_2$.

Conclusions and outlook

Anisotropic porosity in the form of lamellar channels where achieved in LCSM ceramics by freeze casting, with increased homogeneity and lower aspect ratios achieved by implementing dynamic freezing profiles and an additional gelation step, respectively. Thus, future work includes:

- X-ray tomography to establish 3D structure (i.e. pore connectivity, quantification of gelation)
- Increased control of freezing for homogenized macrostructure
- Detailed quantification of microstructure to establish correlation between processing (specifically freezing and sintering) and structure
- Structure of ceramics vs. performance as regenerator material in magnetocaloric refrigeration systems

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References