Synchrotron diffraction characterization of alternative powder diffraction standards

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Abstract

A set of standard reference materials (SRM) for powder diffraction were developed at the Laboratory of Applied Crystallography of the Nuclear and Energy Research Institute - IPEN/CNEN, aiming to offer an alternative to the expensive standards produced and sold by the US National Institute of Standards and Technology - NIST. The materials studied as standards were high purity samples of CeO₂, Y₂O₃, Si and Al₂O₃ submitted to thermal treatments and careful selection by sieving. These standard materials were characterized by diffraction at two synchrotron sources and were compared to the NIST ones. The results show that the produced SRM's present quality similar or, in some cases, superior to the NIST ones.

Keywords: standard reference materials, powder diffraction, synchrotron

1. Introduction

X-ray powder diffraction is one of the most useful tools in the study and development of materials. To achieve precision and repeatability the calibration of the XRD instruments is extremely important. The calibration of diffraction equipment require the use of standard samples for the alignment of the geometry, determination of radiation wavelengths and intrinsic instrumental resolution for conventional, synchrotron and neutron powder diffractometers. Furthermore, for some experimental procedures it may be necessary the use of internal standards mixed to samples to calibrate the position of reflections or their intensities [1]. Additionally, for size-strain profile analysis a standard material is generally necessary to determine and correct the instrumental broadening. The requirements for these materials, known as standard reference materials (SRM), may vary for each application but, in general, must: be stable with well-defined cell parameters, present intense reflections without overlaps, present large crystallite sizes and negligible microstrains [2]. The SRM's widely used for these applications are the Standard Reference Materials for Powder Diffraction produced and sold by the US National Institute of Standards and Technology - NIST. However, due to their high prices, some laboratories do not use these standards or produce their own "home-made" standards, usually not certified [2]. In order to provide an alternative to NIST standards we developed a set of SRM samples especially treated in order to present the required properties for a SRM [2]. In this work are presented the results for these samples which are compared to the results of NIST SRM's.

2. Materials and methods

The starting materials were high purity CeO₂, Si, Y₂O₃ and Al₂O₃ which were heat treated and selected by sieving in order to present high crystallite sizes, negligible microstrains, and particle sizes in the range of 37 to 44 μm, as described in previous works [2-5]. These SRM samples, named "IPEN-SRM" were analyzed by high-resolution synchrotron diffraction at the Brazilian Synchrotron Laboratory (LNLS - Campinas - Brazil) and at Alba Synchrotron (Barcelona - Spain) and compared to the NIST SRM samples. In Tab.1 are shown the conditions of heat treatment for each material.

Table 1: Conditions used for the heat treatments of the materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>CeO₂</th>
<th>Si</th>
<th>Y₂O₃</th>
<th>Al₂O₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1350</td>
<td>900</td>
<td>1350</td>
<td>1300</td>
</tr>
<tr>
<td>Time (h)</td>
<td>72</td>
<td>24</td>
<td>72</td>
<td>72</td>
</tr>
<tr>
<td>Ambient</td>
<td>Air</td>
<td>Vacuum</td>
<td>Air</td>
<td>Air</td>
</tr>
</tbody>
</table>

3. Results

In Fig. 1 are shown the diffraction profiles for (a) CeO₂, (b) Y₂O₃, (c) Al₂O₃ and (d) Si, measured at the Brazilian National Synchrotron Laboratory - LNLS (Campinas - Brazil) in high-resolution configuration. In Fig. 2 are shown the diffraction profiles for (a) Y₂O₃, (b) Si and (c) CeO₂, measured at the ALBA Synchrotron Facility (Barcelona - Spain).
From Figs. 1 and 2 it can be seen that all the SRM's present narrow and high intensity diffraction profiles, as expected for ideal diffraction standards.

In Fig. 3 are shown details of normalized XRD profiles for IPEN's and NIST's Al₂O₃ samples in which it can be seen that the IPEN's sample profile presents more symmetrical and narrow reflections.
From Tab. 2 it can be observed that in the cases in which there are similar IPEN and NIST SRM's (Al$_2$O$_3$ and Si) the IPEN's ones present higher crystallite sizes, which is evidenced by the narrower reflections of Al$_2$O$_3$-IPEN sample compared to Al$_2$O$_3$-NIST sample as can be seen in Fig. 3. The CeO$_2$-IPEN SRM presents the highest mean crystallite size, denoting that it is a good standard for instrumental breadth determination. The Y$_2$O$_3$-IPEN SRM also presents high crystallite size and, additionally, a great number of reflections, which make it a good standard for Rietveld refinement method for which this characteristic allows good determinations of profile function and instrumental parameters. The Al$_2$O$_3$-IPEN SRM presents more symmetric and narrow profiles than NIST's sample, evidencing its higher crystallinity.

4. Conclusion

It can be concluded that the IPEN's powder diffraction SRM's are fully similar or, for some of them, better than NIST SRM's. The IPEN powder diffraction SRM's are already being used in more than 25 research laboratories in Brazil and abroad, including LNLS and ALBA synchrotrons Laboratories and IPEN's neutron powder diffractometer.

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6. References