FIRST Newsletter

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XRD Characterization of an Equimolar Li-Sn Alloy: Comparative study of two solutions for sample degradation protection

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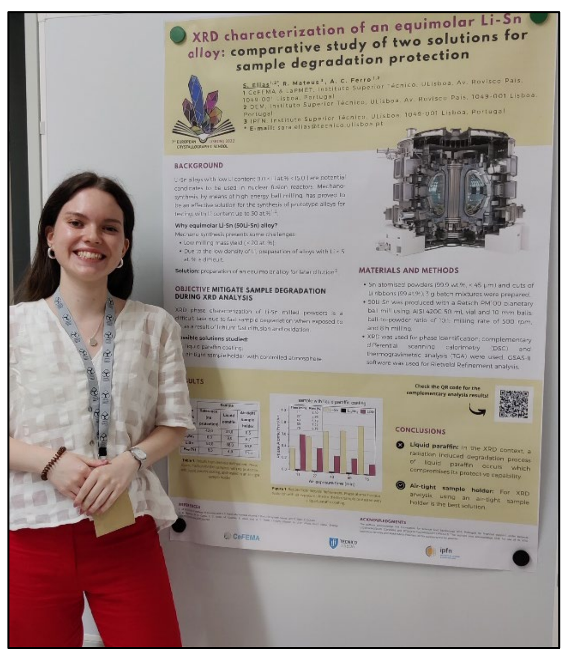
Overview

Sara Elias, a young researcher at Instituto Superior Técnico in Lisbon, recently worked on the X-Ray Diffraction (XRD) characterization of an equimolar Li-Sn alloy by performing a comparative study of two solutions for sample degradation protection.

Lithium-tin (Li-Sn) alloys are potential candidates for plasma-facing components of nuclear fusion reactors. Mechano-synthesis, by means of high-energy ball milling, has proven to be an effective solution for the synthesis of prototype alloys. However, the reaction path characterization faces some challenges, such as fast sample degradation when handled and exposed to air. This effect is due to lithium reaction with air (O_2, H_2O, and CO_2) and diffusion.

Two possible solutions to mitigate sample degradation during XRD analysis were investigated: a liquid paraffin coating and an airtight sample holder with a controlled atmosphere.

During XRD analysis, a radiation-induced degradation process of liquid paraffin occurs, compromising its protective capability. The study concluded that the use of an airtight sample holder is a mandatory requirement. Not only does this work apply to the study of Li-Sn alloys, but also to work being carried out on batteries where Li-based compounds have become the *de facto* standard.



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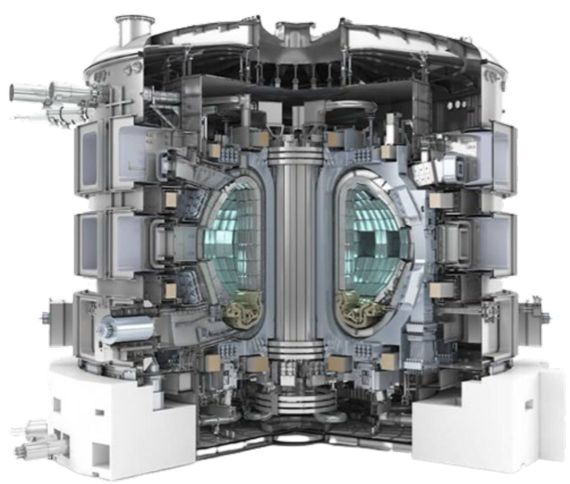
This research was conducted using a D8 ADVANCE diffractometer with an airtight specimen holder.

Background

Li-Sn alloys with low Li content (1.0 < Li at. % < 15.0) are potential candidates to be used in nuclear fusion reactors. Mechano-synthesis, by means of high-energy ball milling, has proved to be an effective solution for the synthesis of prototype alloys for testing, with Li content up to 50 at. %[1,2].



Why equimolar Li-Sn (50Li-Sn) alloy?

Mechano-synthesis of low Li (< 25 at. % Li) Li-Sn alloys presents some challenges:

- low milling mass yield (< 20wt. %/wt.%)
- due to the low density of Li, preparation of prototype alloys with Li < 5 at. % is difficult

The solution is to prepare an equimolar alloy for later dilution.[2]

Objective: Mitigate sample degradation during XRD analysis

XRD phase characterization of Li-Sn milled powders is a difficult task due to fast sample degradation when exposed to air, due to fast lithium diffusion and oxidation. Possible solutions studied:

- liquid paraffin coating
- airtight sample holder with controlled atmosphere

Materials and Methods

- Sn atomized powders (99.9 wt. %, < 45 µm) and cuts of Li ribbon (99 at. %); 3 g batch mixtures were prepared.
- 50Li-Sn was produced with a Retsch PM100 planetary ball mill using a AISI 420C 50 mL vial and 10 mm balls with a ball-to-powder ratio of 10:1, milling rate of 500 rpm, and 8 h of milling.
- XRD was used for phase identification while complementary differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were also used. Rietveld Refinement analysis was performed in GSAS-II.

Results

Table 1. Results from Rietveld Refinement. Phase atomic fraction for film samples: with no protection, with liquid paraffin coating, and sealed in an airtight sample holder.

	Sample		
Phase Atomic Fraction (%)	**Reference (no protection)**	**Liquid paraffin**	**Airtight sample holder**
β-Sn	42.0	33.0	0.5
Li_2Sn_5	6.0	9.0	8.7
LiSn	52.0	58.0	90.8
R_{wp} (%)	8.3	4.9	11.5

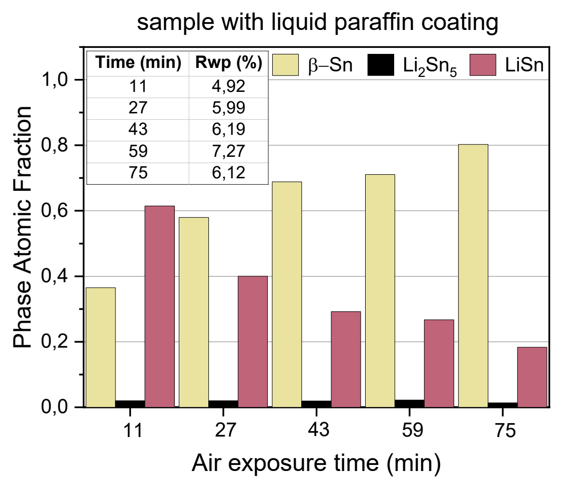


Figure 1. Results from Rietveld Refinement. Phase atomic fraction evolution with air exposure time for the film sample protected with a liquid paraffin coating.

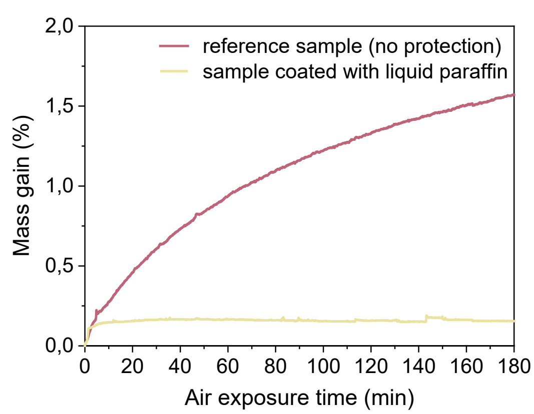


Figure 2. Complementary analysis results. TGA traces of reference sample (red) and sample coated with liquid paraffin (yellow). Liquid paraffin coating effectiveness, against oxidation in air atmosphere, is confirmed.

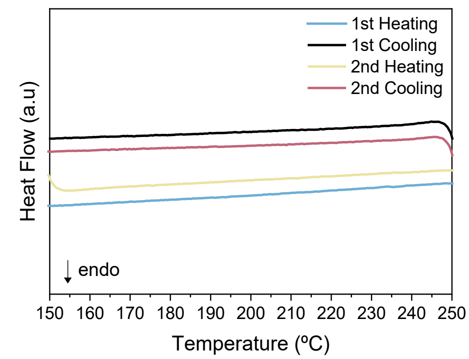


Figure 3. Complementary analysis results. DSC traces of produced 50Li-Sn powders. No peaks are detected, suggesting LiSn phase is the sole constituent.

Conclusions

- ✗ **Liquid paraffin:** In the XRD context, a radiation-induced degradation process of liquid paraffin occurs which compromises its protective capability.

- ✓ **Airtight sample holder:** For XRD analysis, an airtight sample holder is the best solution.

The research was carried out at the Structural Chemistry Center (CQE) X-ray Diffraction Facility of Instituto Superior Técnico (IST), in a joint research study between the Center of Physics and Engineering of Advanced Materials (CeFEMA) and the Institute for Plasmas and Nuclear Fusion (IPFN), under the supervision of Alberto Cabral Ferro and Rodrigo Mateus.

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References

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