Experimental report

07/09/2022

Proposal:	**CRG-2474**	**Council:**	4/2017
Title:	Magnetic determination of the hexagonal Fe3Sn phase		
Research area:			

This proposal is a new proposal

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Samples: Fe3Sn

Instrument	Requested days	Allocated days	From	To
D1B	2	1	22/09/2018	23/09/2018

Abstract:

Experimental Report– Proposal CRG-2474 (D1B) – 22/09/18

“Magnetic structure determination of the hexagonal Fe_3Sn phase”

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The global market of permanent magnets is continuously growing. The state-of-the-art compounds that are being nowadays employed in industry are those based on rare earth (RE) elements. These elements were identified as critical raw materials (CRM) by EU [1] and USA and there exists a need of looking for new compounds so that the market would not be restricted and dominated by China [2]. Moreover, these new materials would be cheaper and, hopefully, as powerfull as currently available Nd-Fe-B magnets.

In contrast to RE, the system we are studying here, Fe_3Sn, has absolutely no content of RE elements and its constituents are inexpensive and abundant (no CRM). There are only few reports on Fe_3Sn connected to permanent magnets applications, due to the fact that the stable compound on the Fe-Sn phase diagram [3] at RT is cubic (Fe_3Sn_2) and, thus, not suitable for this purpose. However, there exist a hexagonal phase, stable at 750-880ºC [3], that is of interest. This phase cannot be achieved by standard methods, i.e. arc melting, so other techniques have to be employed, such as solid state reaction (SSR) [4, 5]. Regarding permanent magnets applications, Fe_3Sn itself is not suitable as it has a planar anisotropy and uniaxial is mandatory. This could be achieved by substitution of Fe- and Sn-sites with other elements, such as Mn and/or Co (in the Fe-site) or Sb, Ga, Ge… (in the Sn-site) [4] or nanostructuring. Despite other groups have succeeded in producing this alloy by similar techniques [4, 5], no magnetic structure determination has been published so far. Thus, this was the objective of our experiment.

For this, we synthesized a large sample (2 grams) of Fe_3Sn by SSR, by the following steps. First, we hand-milled stoichiometric amounts of the starting powders, which were compacted using a cold press (with pressures up to 4.5 tons), at RT. This pellet was then inserted in a quartz ampoule and heated to 800ºC for 48 hours, in vacuum. As observed in other Fe_3Sn samples [6], two subsequent SSR steps were mandatory for the obtaining of a 100% of hexagonal Fe_3Sn phase. Thus, the process described above was repeated twice.

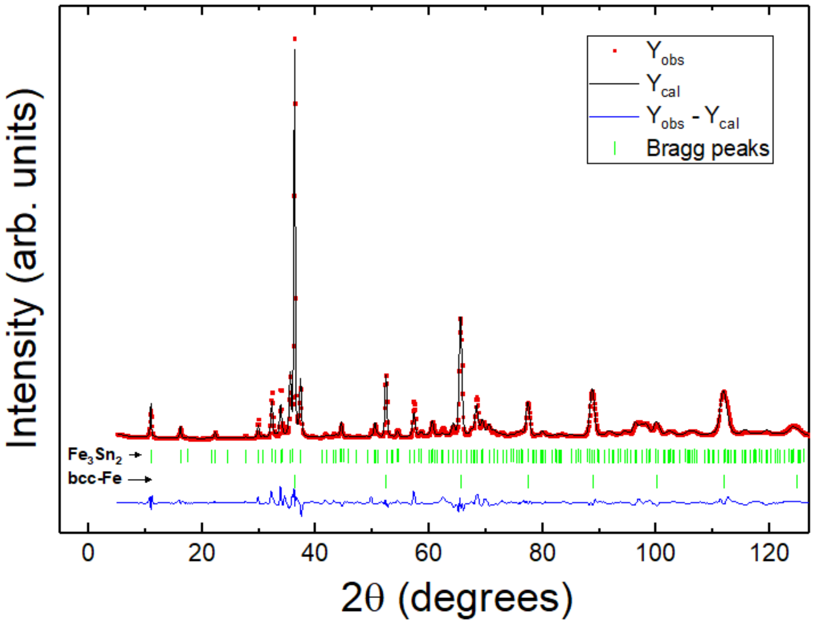

Figure 1. Rietveld refinement of the XRD data of the Fe_3Sn sample that users brought to ILL.

Although this process has been successfully produced by our group several times, the sample that arrived to ILL did not have the proper structure. As it can be observed in Fig.1, the existing phases within the sample are Fe_3Sn_2 (61 wt.%) and bcc-Fe (39 wt.%), as resulted from the Rietveld refinement of the neutron diffraction pattern at RT. As these phases are more stable than the one we were trying to synthesize, we are not sure whether the sample was « destroyed » in the way to ILL.

We decided to heat up the sample up to 600ºC to see if we could either (i) induce the phase transformation of the whole sample to Fe3Sn or (ii) see a change in the pattern (in case there was a small percentage of the Fe_3Sn phase within the sample) from ferromagnetic to the paramagnetic region. Unfortunately there was no change at all and we cannot see any differences between patterns at RT and high temperature, only the expected displacement of lattice parameters (see Fig.2).

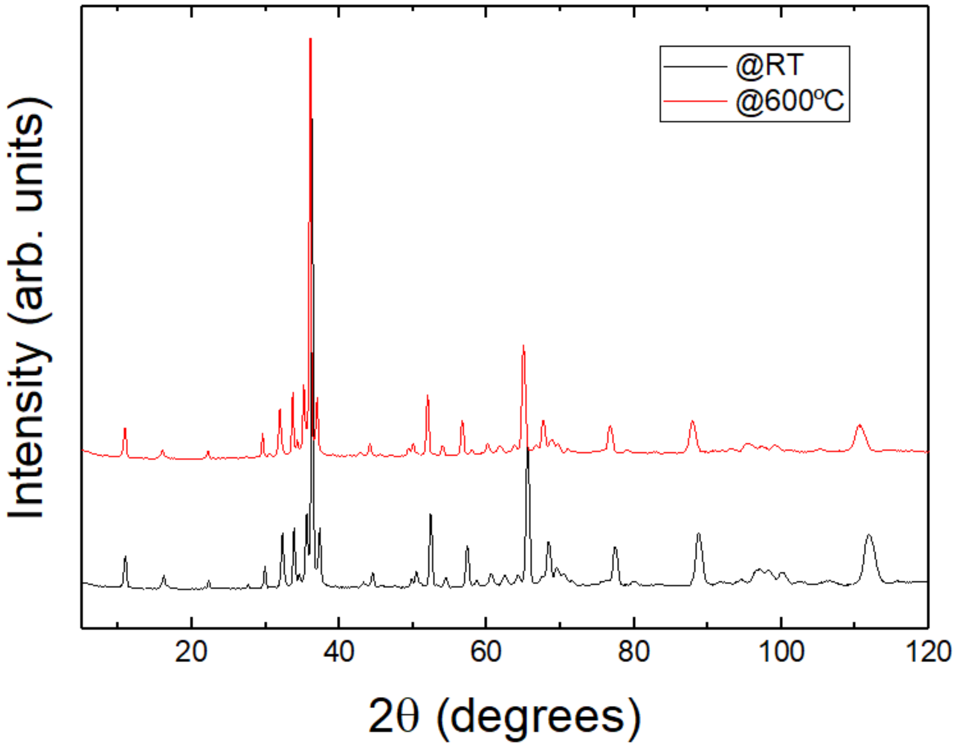

Figure 2. XRD patterns of the sample at room temperature (black) and at T=600ºC (red). No main differences can be observed between the two patterns.

Thus, we decided not to measure this sample anymore and to use the spare time to start our next experiment (CRG-2553).

References

[1] EU 2014 Report on Critical raw materials.
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[4] B. C. Sales et al., Sci. Rep. **4**, 7024 (2014).
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[6] C. Echevarria-Bonet et al., JALCOM **769**, 843 (2018).