Experimental report

13/12/2024

Proposal: **CRG-3117** **Council:** 4/2024

Title: Recycled spinel ferrite powders for 3D printing of composites for electromagnetic absorption

Research area:

This proposal is a new proposal

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Experimental team:

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Samples: Cu0,2Ni0,3Zn0,5Fe2O4

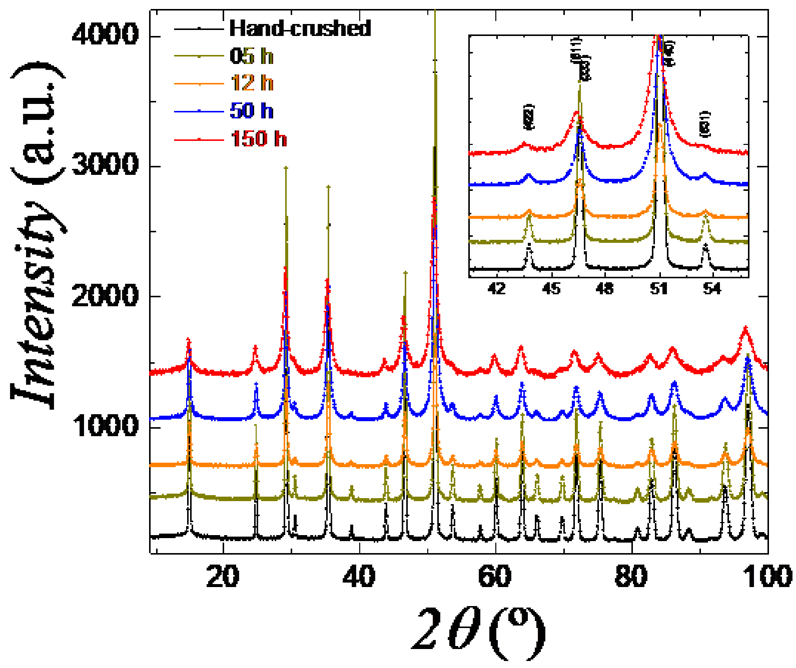
Instrument	Requested days	Allocated days	From	To
D1B	1	1	04/04/2024	05/04/2024

Abstract:

__Recycled spinel ferrite powders for 3D printing of composites for electromagnetic absorption__

Spinel ferrites are one of the most used magnetic material in electromagnetic shielding as absorbing materials due to their relatively high dielectric/magnetic loss, excellent chemical stability, and low costs. Our purpose is to recycle ferrite cable cores to produce 3D printable polymer matrix composites. Ball Milling has been used to produce microparticles (ϕ<63 μm) and they were introduced in polymers using a solution casting method. The aim of this work is analyze the effect of the milling process on the powdered material. The influence of the grinding process on the magnetic and structural properties of recycled $Cu_{0,15}Ni_{0,3}Zn_{0,55}Fe_2O_4$ spinel ferrites have been analyzed by Neutron diffraction measurements at D1B after different milling times (As crushed, 0.5 h, 12 h, 50 h and 150 h). Besides, the recovery (high temperature treatments) of the microstructure generated during deformation has also been studied by In situ" thermo-diffraction measurements from RT up to 800ºC

Neutron diffraction measurements performed on the entire set of ball-milled samples show the presence of the spinel structure. (figure 1).



º: Room temperature Neutron diffraction patterns for As crushed, 0.5, 12, 50 and 150 hours milled samples.

The similar scattering factors associated with the atoms involved in the material (Zn, Ni, Fe and Cu) make X-ray diffraction analysis difficult, so the objective was to confirm or discard the possible change in the distribution of the cations. The results seems to indicate no changes in the cation distribution after deformation (similar relative peak intensities). Apart from the reduction in particle size, no other effects on the crystal structure (space group Fd3m) or on the cation distribution (between tetrahedral and octahedral sites) have been observed. A Rietveld analysis is being performed to confirm this preliminary hypothesis based on the null variation in the relative intensity of the peaks.

The recovery studied by "In situ" thermo-diffraction up to 800ºC was performed on the more deformed sample (150 H milled sample, see figure 2). The results indicate an increase of the crystallite size and a decrease of the internal stresses with temperature according to a recovery process (Inset in figure 2). Although a change from a Maghemite to a Magnetite type structure is expected, at first glance, no such change is observed. The similar Crystallographic structure of both phases requires a more detailed and careful Rietveld Analysis (not yet performed).

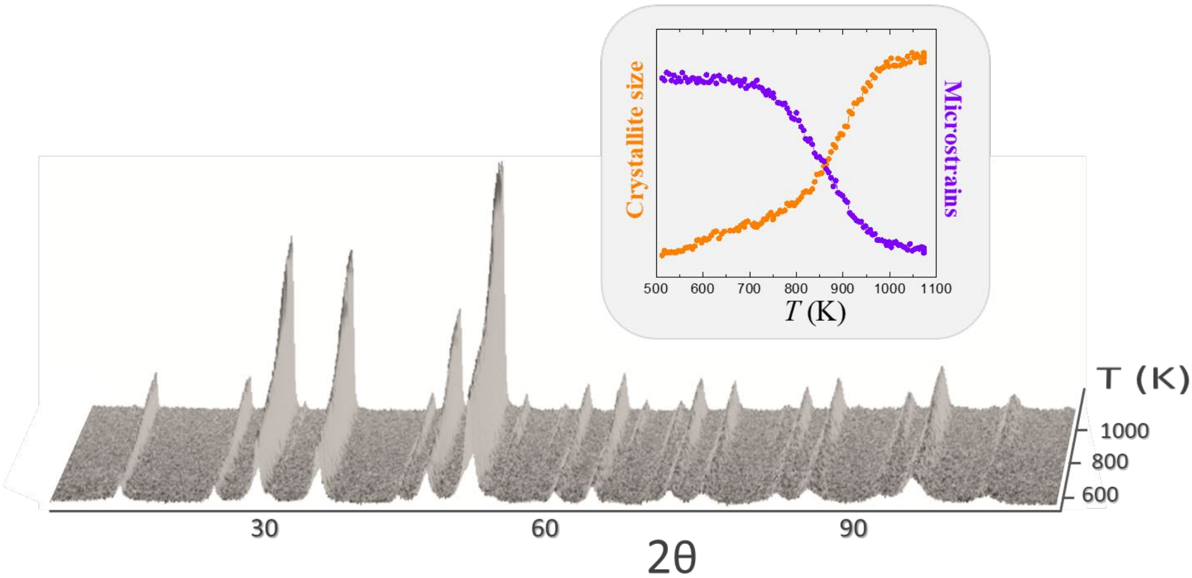


Figure 2: “In situ” thermo-diffraction from RT up to 800ºC performed on the 150 H milled sample.