Experimental report

03/09/2019

Proposal: 1-04-156 **Council:** 10/2018

Title: Oxygen vacancies and persistent luminescence in Sr2MgSi2O7 glass-ceramics

Research area: Materials

This proposal is a new proposal

Main proposer: Maria Jesus PASCUAL

Experimental team: Maria Jesus PASCUAL
Laura FERNANDEZ RODRIGUEZ
Maria de las Mercedes SEDANO RODRIGUEZ

Local contacts: Stanislav SAVVIN
Gabriel Julio CUELLO

Samples: Sr2MgSi2O7 glass-ceramics 2
Sr2MgSi2O7 glass-ceramics 3
Sr2MgSi2O7 glass-ceramics 1

Instrument	Requested days	Allocated days	From	To
D20	4	4	05/09/2019	09/09/2019
D2B	3	3	28/06/2019	01/07/2019

Abstract:

The aim of this proposal is to study oxygen vacancy location and environment in the crystalline phase Sr2MgSi2O7 obtained from the glass-ceramics route. On doping with Eu/Dy, the glass-ceramics are very efficient photoluminescence (PL) materials. The luminescence properties are also dependent on the temperature, which strongly indicates that oxygen vacancies play an important role in the still poorly understood luminescence mechanism. More information concerning the vacancies is, thus, essential for optimizing the materials composition and their optical behavior.

Oxygen vacancies and persistent luminescence in $Sr_2MgSi_2O_7$ glass-ceramics

Report

From 28.06.2019 to 01.07.2019 the neutron diffraction experiment number **1-04-156** took place at ILL, Grenoble, France.

The used instrument was D2B, using its λ = 1.594 Å line for an ex situ measurement. A complete diffraction pattern is obtained after about 25 steps of 0.05° in 2θ, the detector was set to span a 2Θ range of 0 to 160°. Cryofurnace was used to measure temperatures from -1.4K to 423K.

Three glass compositions prepared in different conditions (Table 1) were thermally treated at 1100 °C for different 1 minute to achieve the crystallization of $Sr_2MgSi_2O_7$ in the glass. The neutron diffraction patterns of the corresponding glass-ceramics were measured in 9 mm diameter vanadium sample holders. The diffraction patterns of NAC and Si standards were also recorded, to subtract the background and determine instrument parameters such as zero-point displacement.

The accumulation time of the measurements was between 3 and 6 hours depending on the sample. Figure 1 shows a spectra of three samples measured at room temperature obtained using the Mantid program and Figure 2 shows one of the samples measured at different temperatures with cryofurnace. The purpose of the measurement is to make a combined refinement of Rietveld with X-rays and neutron diffraction data from the same samples for different temperatures of the glass-ceramic in question. During the temperature change the emission of the glass-ceramic also changes from green to red in a reversible way. With the combined refinement of Rietveld with X-ray and neutron diffraction data, information will be obtained on the possible oxygen and strontium vacancies presented by the three samples in order to elucidate its role in the luminescence process. Figure 3 shows the neutron diffraction pattern with the corresponding Rietveld refinement of sample F6 at room temperature ($Sr_2MgSi_2O_7$:Eu^{2+}, Dy^{3+}). In order to get conclusive results, all the difractograms need to be recorded during long times of the order of 6 hours, for this reason a new application will be done to apply for additional measuring time.

Table 1. Composition of the glasses

Composition (%mol)	E0 Mg1.5-55(Sr) Electrical furnace	E4 Mg1.5-55(Sr)+1Eu-0.5Dy Electrical furnace	F6 Mg1.5-55(Sr)+1Eu-0.5Dy Gas Furnace
MgO	18	18	18
SiO_2	55	55	55
SrO	27	27	27
Eu_2O_3	0	1	1
Dy_2O_3	0	0.5	0.5

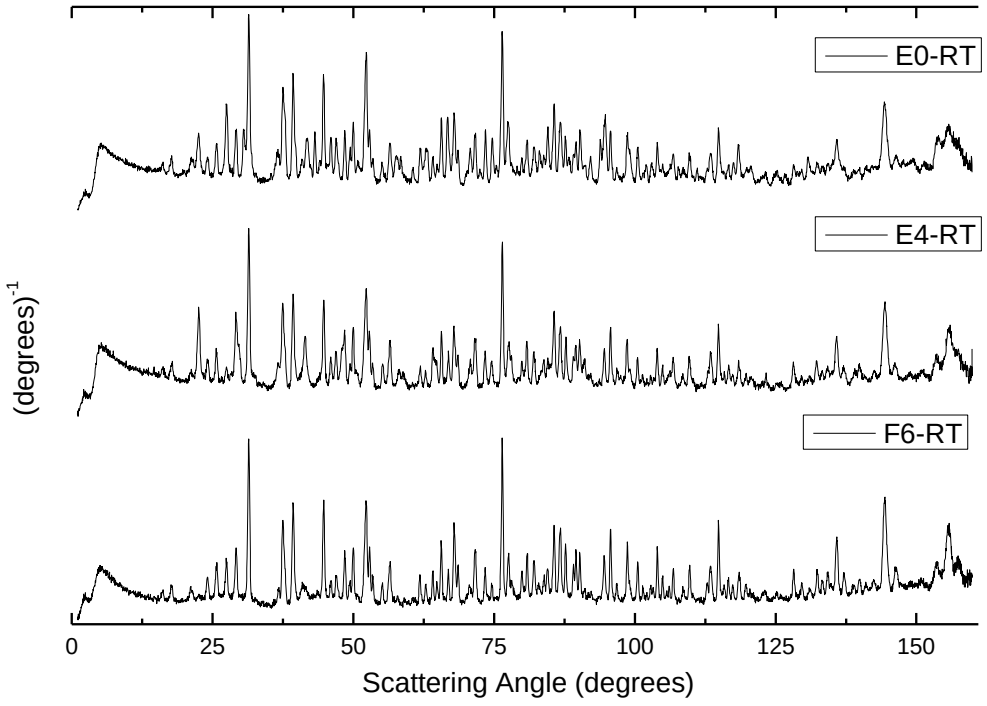

Figure 1. Neutron diffraction patterns of three samples at room temperature.

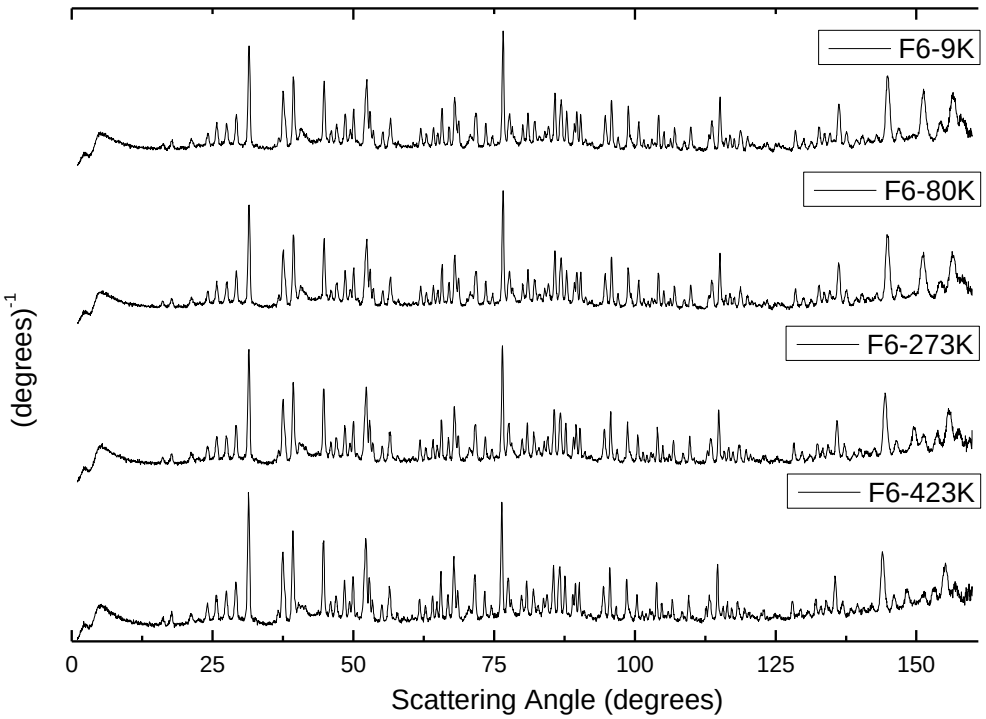

Figure 2. Neutron diffraction patterns of sample F6 at different temperatures using cryofurnace.

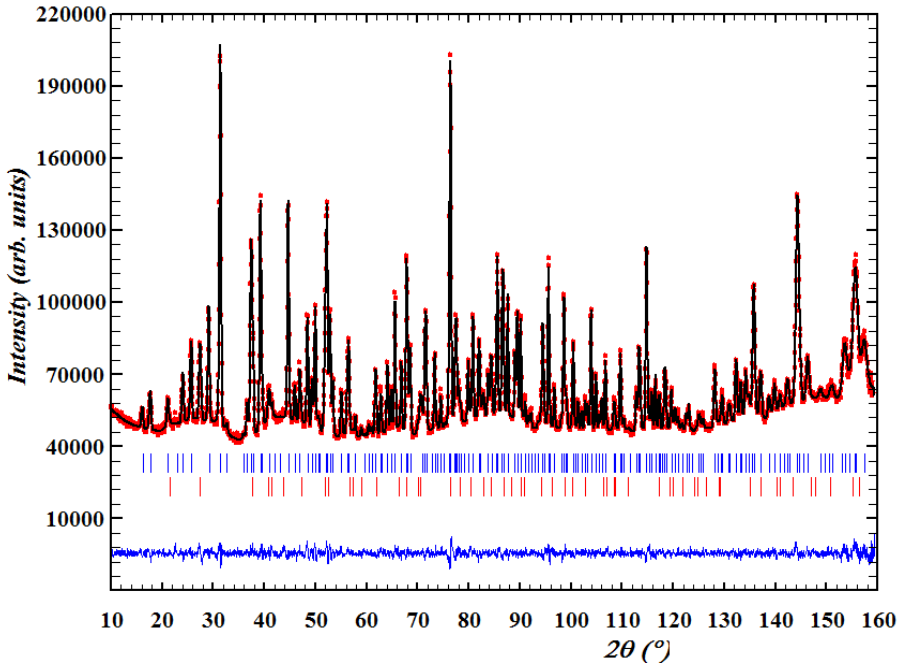

Figure 3. Neutron diffraction pattern measured at room temperature and corresponding Rietveld refinement of $Sr_2MgSi_2O_7:Eu^{2+}$, Dy^{3+} glass-ceramics (F6). Measuring time 6 hours.