

Experimental report

25/01/2022

Proposal: 1-04-206

Council: 4/2020

Title: Cationic and oxygen vacancies in Sr₂MgSi₂O₇ glass-ceramics

Research area: Materials

This proposal is a continuation of 1-04-156

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Samples: L1 Eu/Dy
L1 Eu
L5 Eu/Dy
L5 Eu

Instrument	Requested days	Allocated days	From	To
D2B	6	2	03/03/2021	05/03/2021

Abstract:

The aim of this proposal is continue to study oxygen and strontium vacancies location and the environment in the crystalline phase Sr₂MgSi₂O₇ obtained by two ways, from the glass-ceramic route and from aerodynamical levitation. 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. In order to optimize the composition of materials and their optical behavior, it is necessary to study further the different temperatures of the vacant environment of these glass ceramics.

Cationic and oxygen vacancies in $\text{Sr}_2\text{MgSi}_2\text{O}_7$ glass-ceramics

Report

From 03.03.2021 to 05.03.2021 the neutron diffraction experiment number **1-04-206** took place at ILL, Grenoble, France.

The used instrument was D2B, using its $\lambda = 1.594 \text{ \AA}$ 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° .

Samples of strontium silicate glass-ceramics (Table 1), which were sintered and crystallised at 1100°C for 1 minute, were measured. There are samples in both electric furnace and gas furnace to complete the results measured above. The samples were measured in 9 mm diameter vanadium holders. Diffraction patterns of the NAC and Si standards were also recorded to subtract the background and determine instrument parameters such as zero point shift.

The accumulation time of the measurements was between 10 and 12 hours per sample, depending on the sample. The purpose of the measurement is to make a combined Rietveld refinement with the X-ray and neutron diffraction data of the same samples for different temperatures of the glass-ceramic in question. With the combined Rietveld refinement with the X-ray and neutron diffraction data, information about possible oxygen and strontium vacancies in the three samples will be obtained. Figure 1 shows a spectrum of the electric furnace samples at room temperature obtained with the Mantid programme. Figure 2 shows Neutron diffraction combined with Rietveld refinement of sample E4 at room temperature ($\text{Sr}_2\text{MgSi}_2\text{O}_7$: Eu^{2+} , Dy^{3+}).

Table 1. Composition of the glass-ceramics (mol%)

Composition (%mol)	E0 Electrical furnace	E4 1Eu-0.5Dy Electrical furnace	F0 1Eu-0.5Dy Gas Furnace	E17 1Eu-1Dy Gas furnace
MgO	18	17.73	18	17.65
SiO ₂	55	54.19	55	53.92
SrO	27	26.60	27	26.47
Eu ₂ O ₃	0	0.99	0	0.98
Dy ₂ O ₃	0	0.49	0	0.98

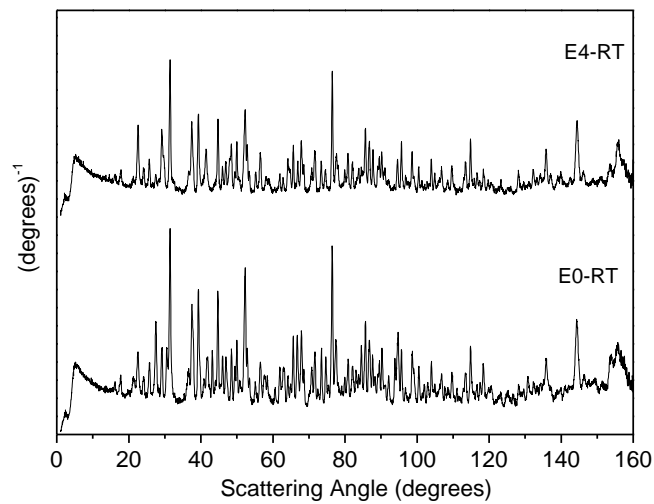


Figure 1. Neutron diffraction of the measurement of samples at room temperature.

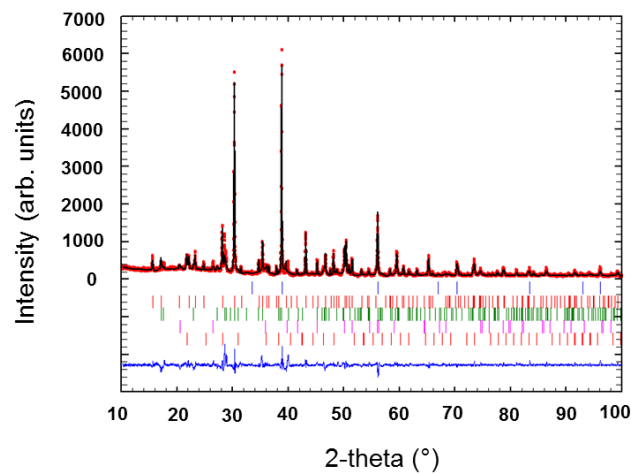


Figure 2. Neutron diffraction combined with Rietveld refinement of doped Sr₂MgSi₂O₇: Eu²⁺, Dy³⁺ glass-ceramics at room temperature (E4).