	Experiment title:	Experiment number:
	Crystallisation kinetics in the systems $CaO-SiO_2$ and $MgO-CaO-SiO_2$	08-02-222
Beamline:	**Date of experiment**:	**Date of report**:
BM-08	from: 29/01/00 at 7:00 to: 31/01/00 at 7:00	22/10/01
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Report:

Introduction

The wollastonite ($CaSiO_3$) and the diopside ($MgCaSi_2O_6$) are two of the most interesting mineral-systems involved in the glass-ceramics industry

Wollastonite ($CaSiO_3$) is a single chain silicate, with a chain repeat unit of three tetrahedra (pyroxenoid). The chains are linked to Ca-octahedral columns. Our aims are the more accurate determination of the kinetic parameters, the identification of the phases and polytypes formed during the thermal treatment of the samples. Previous works [1,2] have shown that the polytypism is very complex and depends on the reactivity of the samples (i.e. depends on the particle size of the powder of the starting glass matrix). Such a characteristics requires the short collecting times allowed by X-ray diffraction by synchrotron radiation and the full angular information obtained by the use of translating Image Plate.

Diopside ($MgCaSi_2O_6$) is a single chain silicate with a chain repeat unit of two tetrahedra (pyroxene). The Si atoms occupy the tetrahedra cavity while the Ca and Mg atoms are in octahedral coordination. The octahedral chains link together the Si-chains. Our aim is the determination of the kinetic parameters about the crystallisation of diopside from glass-matrix such as in industrial processes.
In a previous experiments (08-02-189) the time allocated was insufficient for a complete determination of the kinetics parameters for all of the samples and/or the processes involved and experimantal problems had affect the data analyses. New beam-time was need for the study of the two processes, the crystallisation of the 2M-wollastonite from the glassy matrix and the polytypic conversion 1T- to 2M-wollastonite for the sample in the $CaO-SiO_2$ system, and one process, the direct crystallisation of the diopside from the glassy matrix in the $MgO-CaO-SiO_2$ system.

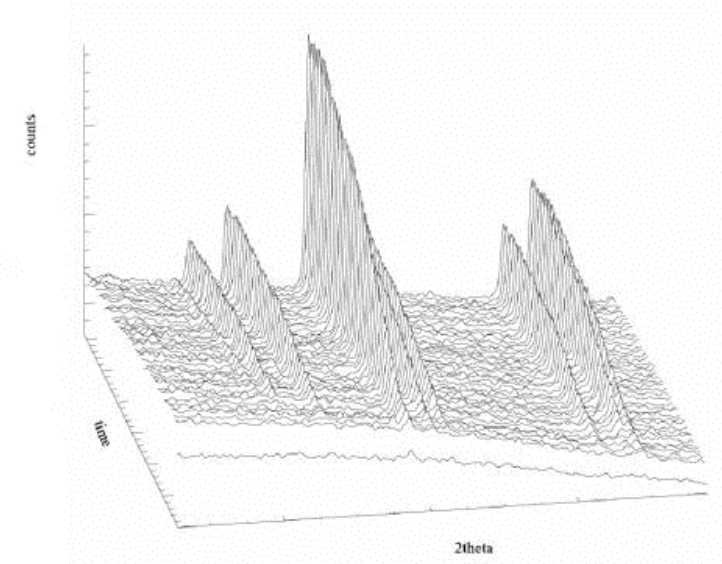
Experimental method

The experimantal setup [3,4] consists of a micro reaction chamber, a hot air heater, a Translating Imaging Plate (TIP) camera for collecting time resolved powder diffraction data and 0.5-1.0 mm quartz glass capillaries in air. This equipment has been developed by us in cooperation with personnel at the Italian

Beamline, GILDA. The experiments consist in four steps. The first step is the calibration of with experimantal set with one-shot X-ray powder diffraction of standard powder of LaB_6. The next step is the collection of one temperature ramp of standard powder of Si in the working temperature range (RT-1000 °C). The third step is the collection of the teperature ramps on the samples in the same range as the Si. The last step is the real kinetics experiment and consists in isothermal experiment at selected temperatures in the range 700-1010 °C (example in Fig.1 for the diopside system). The temperatures choice depends on the phases studied, here we are interested in the crystallisation of intermediate phases (disorder polytypes) of wollastonite, in the crystallisation of the final stable 2M-wollastonite and in the crystallisation of diopside.

Results

The results are affected by the same experimental problems that in the previuos experimental shifts (08-02-189 in date 9-11/10/1999), related to the characteristics of the sample and/or the experimental setup. One is the higher temperature limit, increased from previus experiments (from 990 to 1010 °C), that is still too low for achieved the crystallisation of 2M-wollastonite,again only in the temperature ramp experiment we can see the early stage of this process. The second problem is related to the experimental setup. The discovered of such a problem is possible only during the mathematical treatment of the data. In fact the lattice parameters obtained from the refinement of the Si standard spectra in the temperature ramp experiment are without sense. Moreover they depends on wich portion of the spectra is refined. Such a effect prevents a fully crystallographics study of the phases involved in the experiments and permits only a qualitative analyses. Moreover this problem prevents also a good calibration of the temperature for the kinetic study. This effect could be the coupling of different disalignement of the Image Plate.



References

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