# Nucleation and crystallization kinetics of a complex lithium disilicate glass: *in situ* and *time-resolved* synchrotron powder diffraction study

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**Introduction:** Lithium disilicate glass-ceramic has been documented since 1950s [1] and now it has applications such as dental restorations and magnetic disks. $P_2O_5$ is one of the most widely used nucleating agents.[2] Even though many techniques have been employed, such as laboratory XRD, electron microscopy, thermal analysis and NMR, the fundamental understanding of the nucleation and crystallization process is incomplete, and the role of $P_2O_5$ as a nucleating agent is still unclear.[3]

**Objectives:** The aims of this study were to investigate the effects of temperature and holding time on phase transformation and crystallite size; to estimate its nucleation kinetics by using a KM-JMA equation;[4] and to investigate the crystallization kinetics and mechanism of this glass. Moreover, we revisited the role of $P_2O_5$ on the nucleation of this glass.
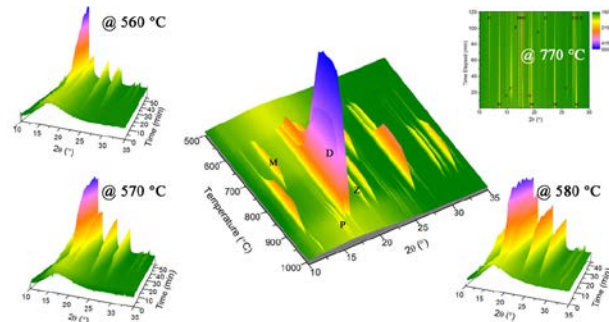


**Fig. 1.** XRD patterns of a lithium disilicate glass at isothermal and non-isothermal annealing processes.

**Methods:** To investigate nucleation and crystallization kinetics, we conducted isothermal annealing at 560, 570, 580 and 770 °C and non-isothermal annealing from 500 to 1010 °C on a multi-component lithium disilicate glass in the $SiO_2$–$Li_2O$–$P_2O_5$–$Al_2O_3$–$ZrO_2$ glass system, and the high-temperature XRD patterns of this glass (Fig. 1) were monitored *in situ* and *real time* with synchrotron radiation during annealing processes. The high-resolution synchrotron XRD data were used for the quantitative phase analysis via Rietveld method. [3]

**Results:** The nucleation kinetics is temperature dependent, and the induction period of nucleation is longer at a lower temperature. The volume fraction data of the isothermal nucleation experiments were modelled using a modified KM-JMA equation to incorporate the initial induction period. The Avrami exponent ($n$) for $Li_2Si_2O_5$ ($LS_2$) phase (1.74–1.84) is higher than that of $Li_2SiO_3$ (LS) phase (1.24–1.49). The activation energy $E_a$ of $LS_2$ and LS are calculated to be 275 and 213 kJ/mol, respectively. The $LS_2$ crystals grow at the expense of the LS, cristobalite and quartz phases in the glass during the isothermal crystallization process at 770°C. Besides, it is found that the nucleation of LS and $LS_2$ in this complex glass is triggered by the steep compositional gradients associated with the disordered lithium phosphate precursors in the glass matrix. [3]

[1] S. D. Stookey, *Ind. Eng. Chem.* **51**, 805-808 (1959).
[2] E. Apel, C. van't Hoen, V. Rheinberger, W. Höland, *J. Eur. Ceram. Soc.* **27**, 1571-1577 (2007).
[3] S. F. Huang, P. Cao, Y. Li, Z. H. Huang and W. Gao, *Cryst. Growth Des.* (2013) Revision under review.
[4] M. Hillert, *Metall. Mater. Trans. A* **42**, 3241-3241 (2011).