



Ceramics / Glass Lab



Glass Oxide Synthesis

- ▶ Glasses are composed of one, two (binary), three (ternary) or multiple oxides containing at least one network former (such as SiO_2 , B_2O_3 , GeO_2 , etc.).
- ▶ The raw oxides must be dried at specific temperatures and kept in desiccators.
- ▶ The desired amounts in mol% are weighed, mixed and brought to Pt-crucibles.
- ▶ Non-purified oxides, such as carbonates (Li_2CO_3), must go through a decarbonization heat-treatment prior to the final melting temperature.

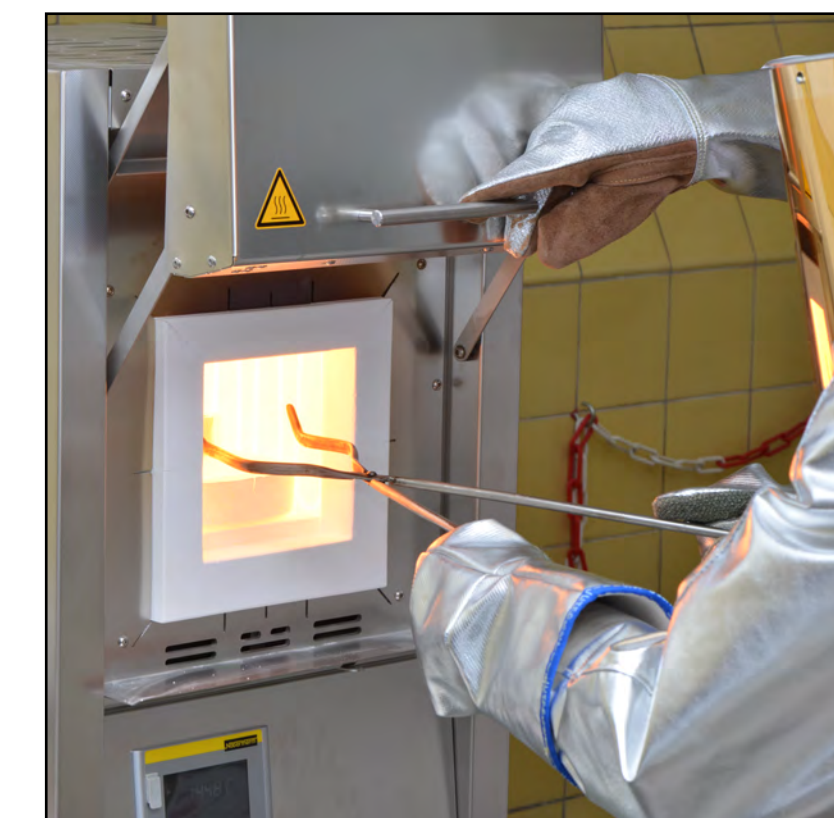


- ▶ (Left): Raw oxides used for the synthesis of glass, weighed in a high-precision balance.
- ▶ (Right): Desiccator containing pre-dried oxide powders to avoid humidity.



Glass Melting

- ▶ After decarbonization in the oven at about 900 °C, the temperature is increased to 1400 °C - 1600 °C for the oxides to enter the liquid state, to about 0.5 - 2 h.
- ▶ Depending on the melting temperature and oxide reactivity, the glass can be melted in Pt-crucibles (up to 1500 °C) or need Pt-Rh crucibles (up to 1650 °C).
- ▶ Some glasses may contain volatile elements that may be lost, so that a lid may need to be used covering the crucible to prevent gas escaping.



- ▶ (Left): Safety clothing for high-temperature exposures used during glass melting.
- ▶ (Right): Platinum crucible being removed from the oven at the melting temperature at 1500 °C for casting.

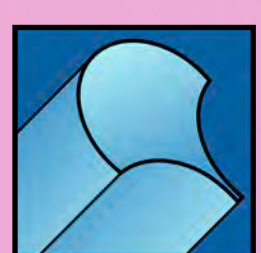


Quenching / Splat-Cooling

- ▶ Because a glass is a mixture of different oxides, it must be well homogenised so to obtain a uniform glass structure after the final casting.
- ▶ One way to assure the homogeneity of the glass is to perform multiple (2-3x) quenching of the melt, whether in water or on a cold steel surface.
- ▶ In that process, the glass solidifies very fast and fractures due to thermal stresses. The pieces can be milled to a powder or not, and go through several further melts.



- ▶ (Left): Quenching of glass by fast cooling of crucible in a cold water bath.
- ▶ (Right): Splat-cooling of glass melt by pressing between two cold metal plates for homogenisation.

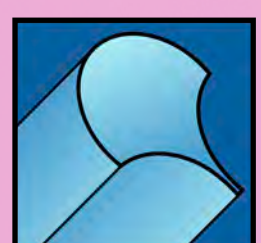


Glass Casting

- ▶ The casting temperature is determined mainly by the viscosity of the melt, which has to be low enough to flow from the crucible before it cools down.
- ▶ The crucible is removed from the oven with a pair of pliers and quickly poured onto a mould made out of brass or graphite having the desired form.
- ▶ The glass must cool down rapidly in the mould to avoid spontaneous crystallization, but be removed right before the glass transition temperature into an annealing oven.

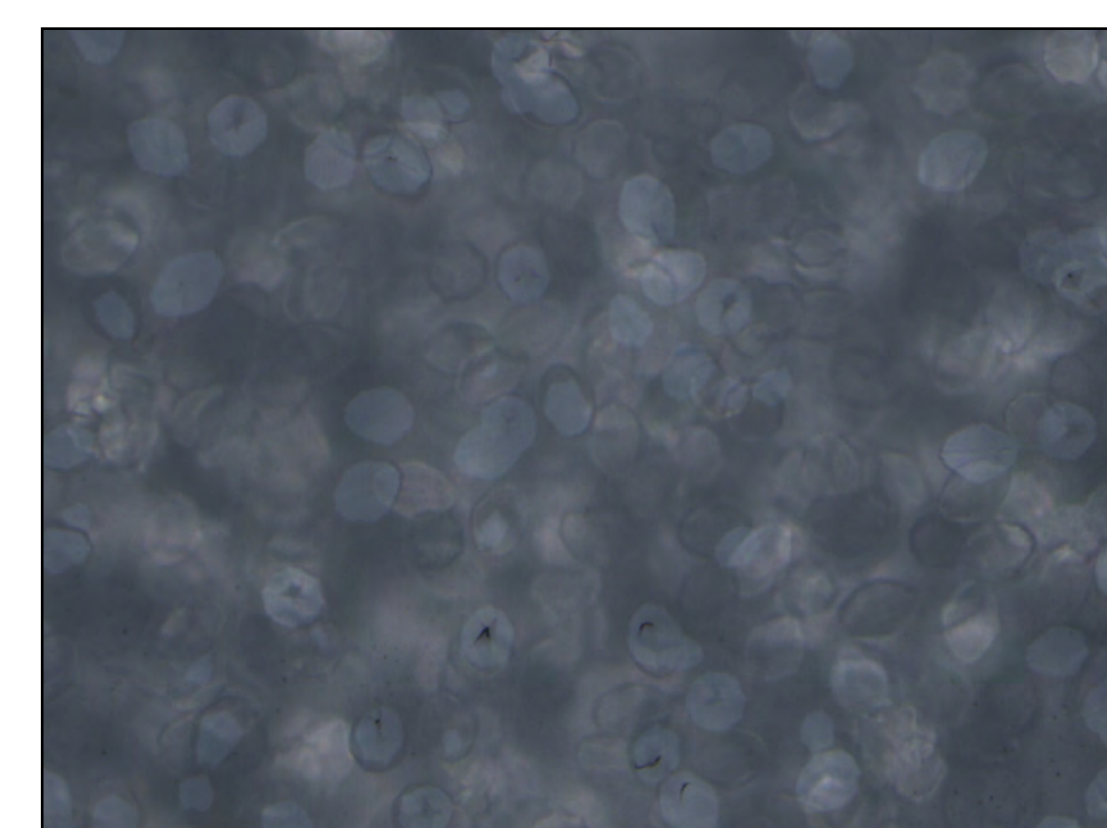
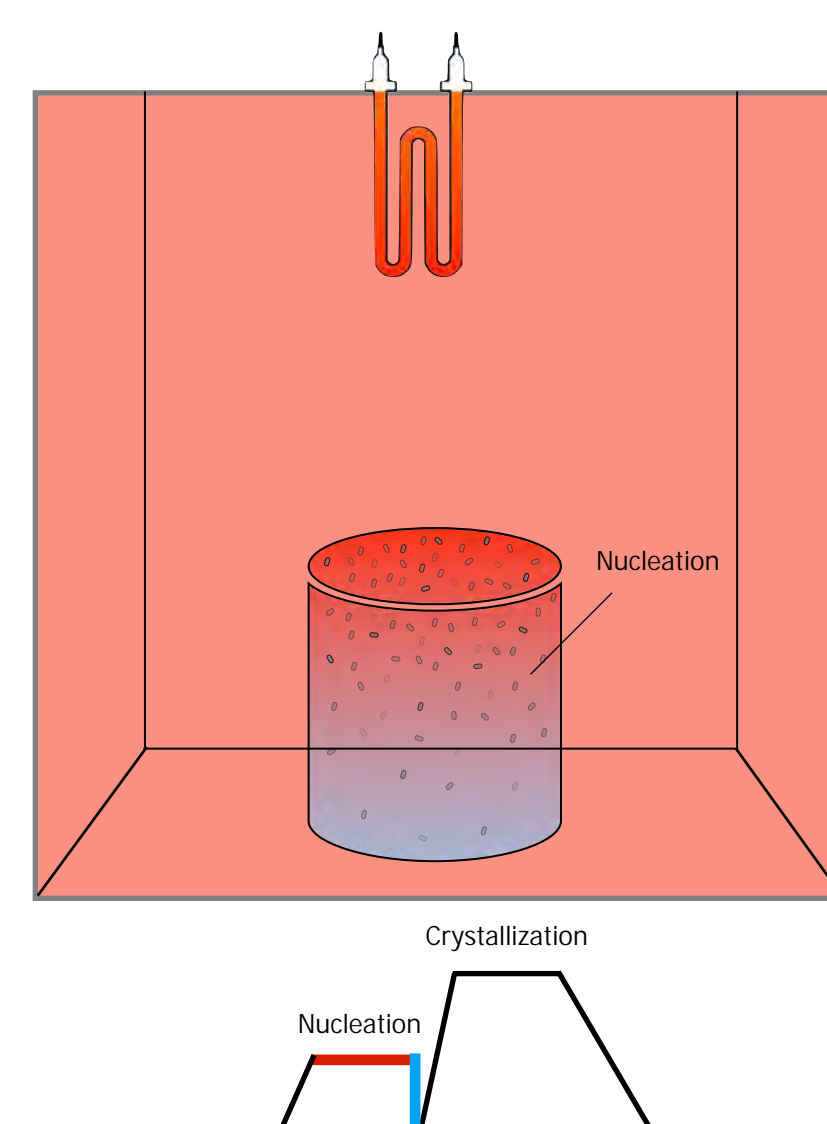


- ▶ (Left): Casting of glass in the liquidus state onto a cold or pre-heated metallic mould.
- ▶ (Right): Casted glass in prismatic form after cooling down and subsequently annealed in an oven at temperatures below T_g .



Nucleation / Crystal Growth

- ▶ Over a small temperature range above the glass transition temperature (T_g), the glass structure at the molecular level assumes a crystalline structure in form of small nuclei.
- ▶ The longer the glass stays within the nucleation range, the higher is the amount of nuclei that will later grow into crystals, forming a glass-ceramic.
- ▶ At a higher temperature range, the higher molecular mobility predisposes atomic migration from the glass towards the nuclei's interface, leading to crystal growth.

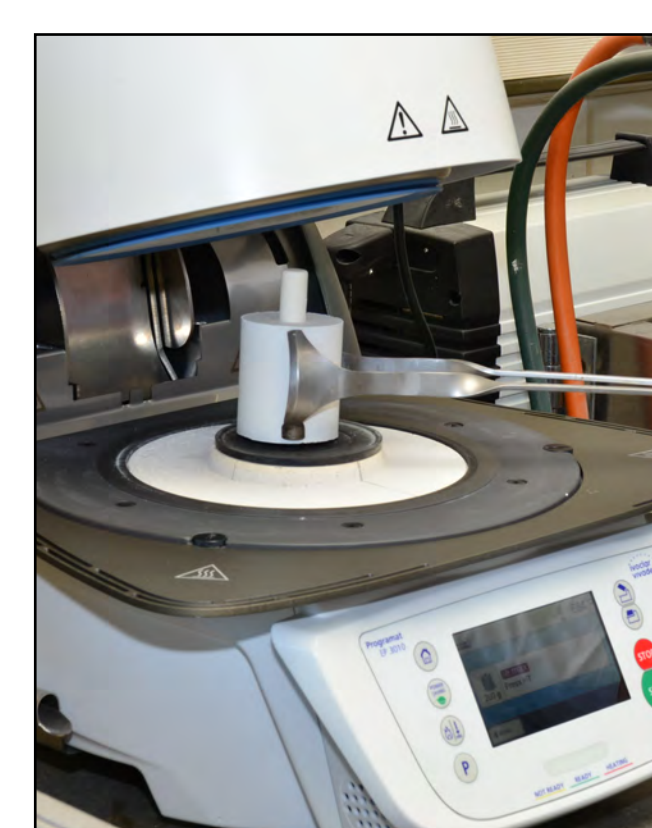


- ▶ (Left): Scheme showing the nucleating of a glass sample in an oven above T_g .
- ▶ (Right): Optical microscopy image of $\text{Li}_2\text{Si}_2\text{O}_5$ crystals grown after nucleation.



Injection-Moulding / Sintering

- ▶ Some glass-ceramics must undergo an injection-moulding process, where they are heated-up and the melt is pressed into a negative form in a refractory dye.
- ▶ Sintering is the process where a material powder fuses together without liquefaction, only by mass transport at the surface of the touching particles.
- ▶ The most typical material in dentistry that undergoes sintering is zirconia, which is sintered in two stages, into a machinable white-body and later to full density.



- ▶ (Left): Refractory dye inserted containing a glass-ceramic ingot into a pressing oven.
- ▶ (Right): Zirconia specimens before and after sintering showing the difference in size.