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Report:

The experiment was a success and the results have been already published on ***Journal of Applied Crystallography:***

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In situ synchrotron powder diffraction study of active belite clinkers

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The clinkerization processes to form belite clinkers, with theoretical compositions close to 60 wt% of Ca_2SiO_4, have been studied *in situ* by high-resolution high-energy (λ = 0.30 Å) synchrotron X-ray powder diffraction. In order to obtain active belite cements, different amounts of K_2O, Na_2O and SO_3 have been added. The existence range of the high-temperature phases has been established and, furthermore, Rietveld quantitative phase analyses at high temperature have been performed for all patterns. The following high-temperature reactions have been investigated: (i) polymorphic transformations of dicalcium silicate, α'_L-$Ca_2SiO_4 \leftrightarrow \alpha'_H$-$Ca_2SiO_4$ from 1170 to 1230 K, and α'_H-$Ca_2SiO_4 \leftrightarrow \alpha$-$Ca_2SiO_4$ from 1500 to 1600 K; (ii) melting of the aluminates phases, $Ca_3Al_2O_6$ and $Ca_4(Al_2Fe_2)O_{10}$, above ~1570 K; and (iii) reaction of Ca_2SiO_4 with CaO to yield Ca_3SiO_5 above ~1550 K. Moreover, in all the studied compositions the temperature of the polymorphic transformation α'_H-$Ca_2SiO_4 \leftrightarrow \alpha$-$Ca_2SiO_4$ has decreased with the addition of activators. Finally, active belite clinkers were produced as the final samples contained α-belite phases.

Six previously decarbonated mixtures of belite clinkers have been studied between 1150 and 1650 K and quenched to get the final phase assemblage. Figure 1 shows a selected range of the SXRPD raw data for one composition at the collected temperatures as a representative example. Figure 2 shows quantitative phase analyses results for one of the compositions as a function of temperature.

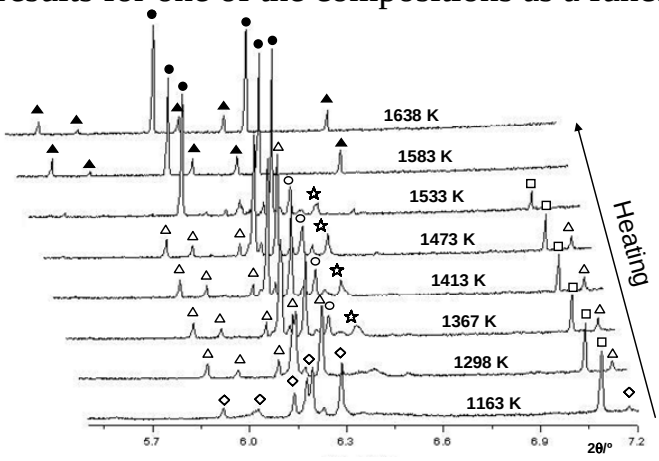

Figure 1. Selected range of the raw patterns for one of the samples collected on heating from 1163 K to 1638 K. Main peaks due to a given phase have been labelled: CaO (open square), α'_L-C_2S (rombus), α'_H-C_2S (open triangle), C_4AF (star);C_3A (open circle), α-C_2S (solid circle) and C_3S (solid triangle).

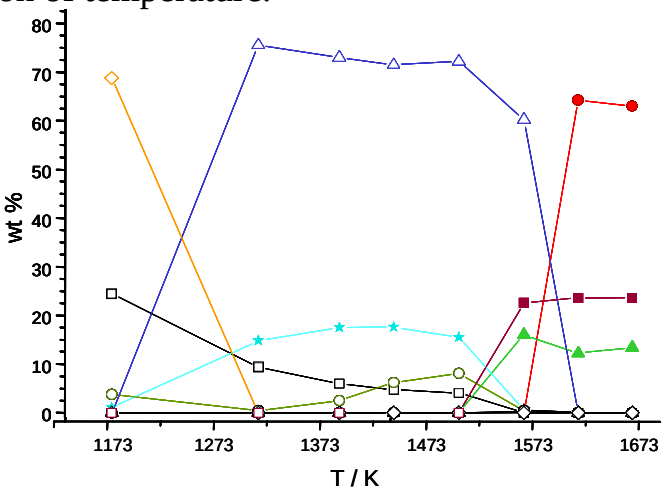

Figure 2. Rietveld quantitative phase analysis results versus temperature on heating for one of the compositions. Remaining symbols as in Figure 1 (solid square stands for liquid phase).

In order to perform Rietveld quantitative phase analyses at each temperature, Pt peaks were removed, i.e. we used all available raw data but excluding the angles were Pt diffracts. Figure 3 shows a full SXRPD powder pattern (left). The green square (right) highlights an angular range where clinker main peaks are placed.

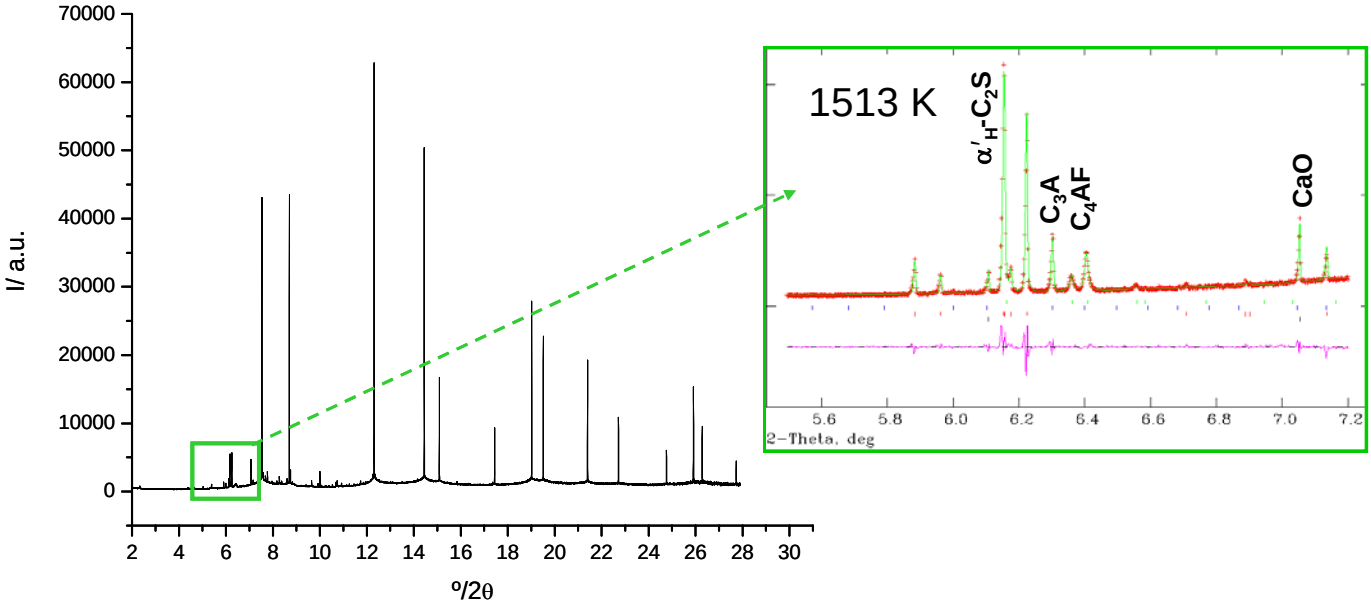

Figure 3. Full SXRPD powder pattern of a selected composition collected on heating at 1513 K. Rietveld plot of a selected range, where clinkers main peaks are placed, is shown for the sake of clarity.