Data Repository

Experimental evidence on the origin of Ca-rich carbonated melts formed by interaction between sedimentary limestones and mantlederived ultrabasic magmas

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Experimental and Analytical Procedures

The starting material used for this study was prepared starting from a Paleocene monticellite-bearing melilitite (vesecite, Vesec, Czech Republic; BM2), a Paleocene melilititeolivine-nephelinite (polzenite, Osečná Complex, Czech Republic; BM1), and a basanite (Jauernick, Germany; BM3). The three samples were melted at 1400 °C for two hours using a vertical furnace RHTV 120-300/18 (Nabertherm) and then quenched in water. The glass was then powdered and melted again at 1450 °C for four hours in the same furnace and then reduced to powder again. The composition of the volatile-bearing natural samples and that of the volatile-free glasses are reported in Table 1. The powders were eventually mixed with different amounts (10, 30 and 50 wt%) of $CaCO_3$ (99.9% purity; AlfaAesar) to obtain three mixtures for each samples (Table 1).

In each experiment the 3 mixtures starting materials were loaded in Pt capsules with a length of ~6-9 mm and ~3 mm to ensure the same P-T conditions. Subsequently, the capsules were welded at the bottom using Welding Microscopes - Lampert PUK U3 under Ar atmosphere. The experiments were performed in a non-end loaded piston cylinder ("QUICKpress" design by Depths of the Earth Co.; Masotta et al., 2012) available at the HP-HT laboratory of the INGV in Rome (Fig. 1). This device generates high pressures (up to 10 kbar) by a hydraulic oil system that pushes a piston into a cylinder consisting of concentric layers of hardened-steel around a WC (tungsten carbide) core. The pressure is estimated from the ratio of hydraulic oil force that is necessary to drive the main ram and the area ratio between the ram and the piston. The uniaxial pressure is then distributed hydrostatically over the sample through deformation of the cell assembly materials. The high temperature is generated by applying a regulated voltage through a cylindrical graphite furnace surrounding the sample. The temperature is monitored with a C-type (W95Re5-W74Re26) thermocouple placed to one end of the capsule.

The 19-25 mm assembly (numbers referred to outer diameter of assembly; Figure 2) has been used for the experiments. It consists of a 11 mm outer diameter graphite furnace, a borosilicate glass insulator (diameter 19 mm), all hosted by an outer NaCl cylinder that fits inside the WC bomb. The sample container used was a modified drilled MgO rod with three holes with diameter of 2 mm. In this way three Pt capsules could be loaded for each experimental run.

In each run, the assembly was first pressurized to a nominal pressure 10% higher than that desired for the experiment and held for few minutes. Pressure was then decreased to 2 kbar (target pressure), and maintained manually constant for the duration of the experiment.

All the experiments were performed at a pressure of 2 kbar at different temperatures, 1100 °C (QP_123, RM_44), 1200 °C (QP_125, RM_41, RM_43), and 1300 °C (QP_124, RM_37, RM_42) from 1 to 12 hours. The experiments were finally quenched manually by shutting down the power. The recovered samples were embedded in epoxy resin and polished without water to avoid dissolution of the carbonate fraction of the melt using the LaboPol-5 Struers using SiC abrasive papers and diamond paste with 0.25 μ m for final polishing.

Qualitative and semi-quantitative analyses of the samples were collected using the scanning electron microscopy (SEM) available at the Dipartimento di Scienze della Terra Sapienza University of Rome and at INGV in Rome. The instrument exploits a bundle of focused primary electrons that hit the sample. The quantitative chemical composition of the glass and mineral phases were analysed by electron probe microanalysis (EPMA) at the CNR-IGAG laboratories (Dipartimento di Scienze della Terra, Sapienza University of Rome) using a Cameca SX50 probe and at INGV in Rome, using a Jeol-JXA8200EDS-WDS.

Cameca SX50 Electron Microprobe at the CNR-IGAG lboratory operating conditions were 15 kV accelerating voltage, 15 nA beam current and 1 μ m beam diameter for olivine, pyroxene and opaque minerals, 10 μ m for glass. Counting times for all elements were 20 s for the peak and 10 s for the background on each side of the peak. Used standards: Na₂O = jadeite; MgO = periclase; TiO₂ = rutile; FeO = magnetite; SiO₂ and CaO = wollastonite; Al₂O₃ = corundum; MnO = rhodonite; K₂O = orthoclase; BaO = barite; SrO = celestine; NiO = metallic Ni.

Chemical analyses collected at INGV were performed using 15 kV accelerating voltage and 7.5 nA beam current and a defocused electron beam of 2.5 μ m. The following standards were used for analyses: MgO = forsterite; K₂O = orthoclase; FeO = augite; Na₂O, SiO₂ and Al₂O₃ = albite; TiO₂ = rutile; CaO and P₂O₅ = fluoroapatite; MnO = rodonite, Cr = metallic Cr; Ni = metallic Ni.



Figure 1. Quick Press apparatus. Picture from http://www.depthsoftheearth.com/instrumentation.htm.

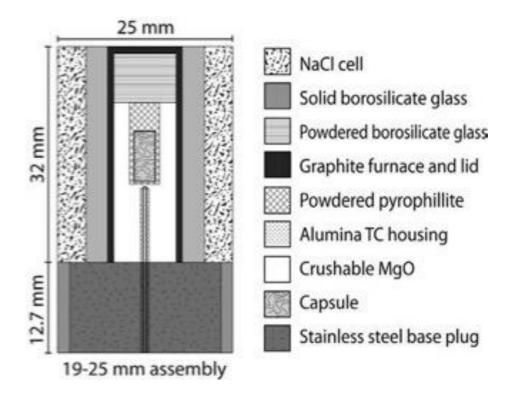


Figure 2. 19-25mm assembly for quick press used to perform the experiments (Masotta et al., 2012).

| Run# | T (°C) | P (kbar) | Cc added (wt%) | SiO ₂ | TiO ₂ | Al ₂ O ₃ | FeO | MnO | MgO | CaO | Na₂O | K₂O | P ₂ O ₅ | Total | CO ₂ (100-tot) | CaO/Al ₂ O ₃ | Mg# |
|--|---------|----------|----------------|------------------|------------------|--------------------------------|-------|-------|-------|-------|------|------|-------------------------------|-------|---------------------------|------------------------------------|------|
| BM1 (natural sample; polzenite) | | | | 38.52 | 2.86 | 9.85 | 11.20 | 0.18 | 15.94 | 13.58 | 2.75 | 1.82 | 1.15 | 97.84 | 2.2 | 1.4 | 0.73 |
| BM1 (quenched glass) 0 | | | 40.11 | 3.00 | 9.66 | 9.98 | 0.17 | 15.94 | 13.40 | 2.88 | 1.73 | 1.09 | 98.09 | 1.9 | 1.4 | 0.75 | |
| QP1_123_10 | 1100 | 2 | 10 | 38.87 | 2.54 | 10.94 | 7.57 | 0.18 | 5.88 | 22.93 | 3.90 | 2.66 | 1.25 | 96.73 | 3.3 | 2.10 | 0.59 |
| QP1_123_30 | 1100 | 2 | 30 | 31.25 | 1.24 | 6.73 | 4.29 | 0.12 | 2.92 | 29.54 | 3.60 | 3.51 | 1.93 | 85.13 | 14.9 | 4.39 | 0.56 |
| QP1_123_50 | 1100 | 2 | 50 | 29.49 | 1.23 | 3.98 | 2.05 | 0.14 | 4.57 | 40.40 | 3.36 | 2.08 | 0.73 | 88.03 | 12.0 | 10.15 | 0.81 |
| QP1_125_10 | 1200 | 2 | 10 | 37.19 | 2.77 | 9.69 | 8.80 | 0.16 | 10.88 | 20.20 | 2.91 | 1.82 | 1.13 | 95.56 | 4.4 | 2.08 | 0.70 |
| QP1_125_30 | 1200 | 2 | 30 | 33.21 | 2.43 | 7.89 | 6.91 | 0.14 | 11.33 | 26.56 | 2.42 | 1.55 | 0.98 | 93.42 | 6.6 | 3.37 | 0.76 |
| QP1_125_50 | 1200 | 2 | 50 | 29.34 | 2.15 | 5.91 | 5.48 | 0.13 | 10.46 | 31.33 | 2.19 | 1.36 | 0.89 | 89.23 | 10.8 | 5.30 | 0.78 |
| QP1_124_10 | 1300 | 2 | 10 | 37.40 | 2.55 | 8.95 | 9.30 | 0.15 | 14.26 | 18.61 | 2.62 | 1.65 | 1.03 | 96.52 | 3.5 | 2.08 | 0.74 |
| QP1_124_30 | 1300 | 2 | 30 | 33.26 | 2.28 | 7.71 | 7.60 | 0.14 | 12.67 | 25.88 | 2.41 | 1.47 | 1.00 | 94.41 | 5.6 | 3.36 | 0.76 |
| QP1_124_50 | 1300 | 2 | 50 | 29.78 | 2.00 | 6.88 | 6.42 | 0.14 | 11.54 | 30.52 | 2.11 | 1.30 | 0.89 | 91.57 | 8.4 | 4.43 | 0.77 |
| BM2 (natural | sample: | vesecit | e) | 30.29 | 2.27 | 7.39 | 11.25 | 0.20 | 17.10 | 21.96 | 0.93 | 0.74 | 1.46 | 93.59 | 6.4 | 3.0 | 0.74 |
| BM2 (quenched glass) 0 | | | | 32.84 | | 7.68 | 11.12 | | 18.23 | | | | | 98.80 | 1.2 | 2.97 | 0.76 |
| RM41 10 | 1200 | 2 | 10 | 30.70 | 2.50 | 6.56 | 6.44 | 0.19 | 10.80 | 30.16 | 1.23 | 0.78 | | 91.35 | 8.6 | 4.60 | 0.76 |
| RM41 30 | 1200 | 2 | 30 | 21.69 | 1.75 | 2.83 | 3.15 | 0.18 | 8.21 | 38.80 | 1.64 | 1.15 | 2.25 | 81.64 | 18.4 | 13.73 | 0.83 |
| RM41 50 | 1200 | 2 | 50 | 15.62 | 1.45 | 1.62 | 2.40 | 0.12 | 6.92 | 43.58 | 1.05 | 0.81 | 1.34 | 74.90 | 25.1 | 26.89 | 0.84 |
| RM37 10 | 1300 | 2 | 10 | 30.13 | 2.17 | 6.10 | 7.71 | 0.19 | 14.39 | 29.19 | 1.01 | 0.69 | 1.57 | 93.14 | 6.9 | 4.79 | 0.78 |
| RM37_30 | 1300 | 2 | 30 | 24.79 | 1.67 | 3.98 | 5.11 | 0.15 | 12.08 | 36.25 | 0.79 | 0.51 | 1.28 | 86.59 | 13.4 | 9.12 | 0.82 |
| RM37_50 | 1300 | 2 | 50 | 17.83 | 1.23 | 1.99 | 2.97 | 0.12 | 8.42 | 43.37 | 0.53 | 0.37 | 0.96 | 77.81 | 22.2 | 21.76 | 0.84 |
| BM3 (natural | sample | hasanit | o) | 12 92 | 1 9/ | 10.91 | 11.61 | 0 17 | 16.93 | 10.49 | 2.35 | 1.00 | 0.55 | 99.87 | 0.1 | 1.0 | 0.73 |
| BM3 (natural sample; basanite) BM3 (quenched glass) 0 | | | | | | 10.81 | | | 16.86 | | | | 0.68 | | 0.2 | 0.94 | 0.75 |
| RM44 10 | 1100 | 2 | 10 | | | 10.05 | 7.82 | 0.09 | 11.91 | | 1.24 | 0.32 | | 99.51 | 0.5 | 2.07 | 0.74 |
| RM44_30 | 1100 | 2 | 30 | 42.38 | | | 2.03 | 0.05 | 12.60 | | 0.51 | | | 98.16 | 1.8 | 5.49 | 0.92 |
| RM44 50 | 1100 | 2 | 50 | 16.51 | | 2.19 | 2.24 | 0.12 | 6.01 | 43.47 | | 0.94 | | 75.57 | 24.4 | 19.85 | 0.84 |
| RM43 10 | 1200 | 2 | 10 | | | 10.92 | 8.69 | 0.18 | 11.41 | | 2.44 | | 0.60 | 97.78 | 2.2 | 1.64 | 0.71 |
| RM43 30 | 1200 | 2 | 30 | 35.76 | | | 6.48 | 0.14 | 11.45 | 28.20 | | 0.78 | | 95.04 | 5.0 | 3.39 | 0.77 |
| RM43 50 | 1200 | 2 | 50 | 26.10 | | 5.13 | 3.16 | 0.07 | 8.94 | 38.69 | 1.33 | | 0.36 | 85.40 | 14.6 | 7.54 | 0.84 |
| RM42 10 | 1300 | 2 | 10 | 41.74 | 1.89 | 10.65 | 9.39 | 0.15 | 12.44 | 16.94 | 2.31 | 0.96 | 0.54 | 97.01 | 3.0 | 1.59 | 0.71 |
| RM42 30 | 1300 | 2 | 30 | 35.85 | | 8.19 | 7.08 | 0.12 | 12.93 | 27.27 | 1.85 | 0.73 | | 95.94 | 4.1 | 3.33 | 0.78 |
| RM42 50 | 1300 | 2 | 50 | 26.85 | 1.08 | 5.74 | 4.67 | 0.09 | 9.41 | 37.90 | 1.33 | 0.50 | 0.35 | 87.92 | 12.1 | 6.60 | 0.79 |

Table 1. Natural samples and quenched glasses used for experiments. $Mg\# = 100 \times Mg/(Mg+Fe^{2+})$ assuming $Fe^{3+}/2Fe = 0.15$.

T= 1100 °C BM1 QP_123

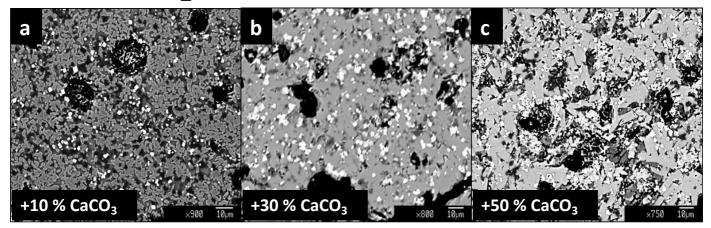
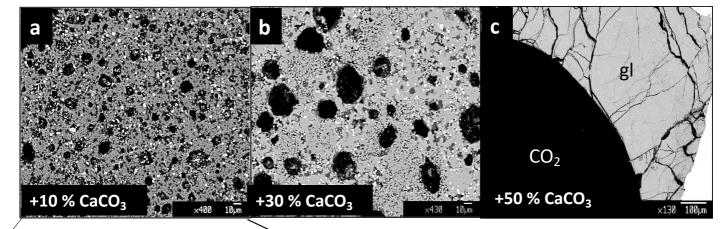


Figure 3. BSE images relative to QP123 run with BM1 starting material at 2 kbar and 1100 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments.

T= 1100 °C BM3 RM_44



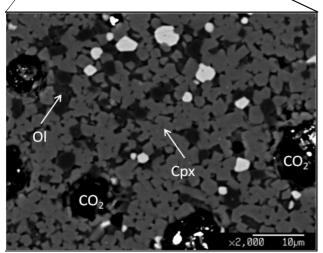


Figure 4. BSE images relative to RM44 run with BM31 starting material at 2 kbar and 1100 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = olivine. Cpx = clinopyroxene.

T= 1200 °C BM1 QP_125

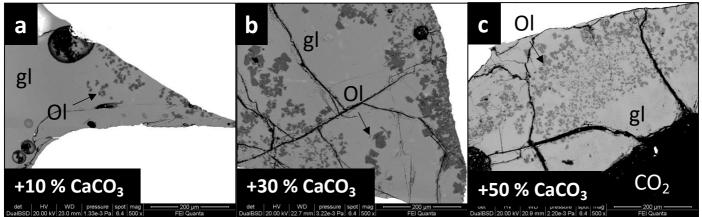


Figure 5. BSE images relative to QP1_125 run with BM1 starting material at 2 kbar and 1200 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = Olivine. GI = Glass.

T= 1200 °C BM2 RM_41

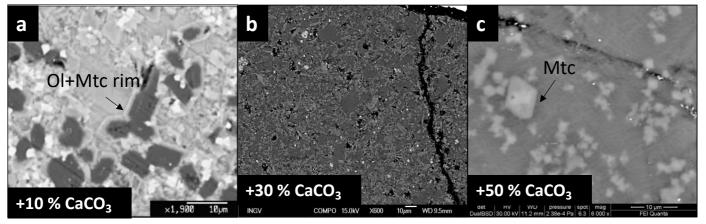


Figure 6. BSE images relative to RM_41 run with BM1 starting material at 2 kbar and 1200 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = olivine; Mtc = monticellite.

T= 1200 °C BM3 RM_43

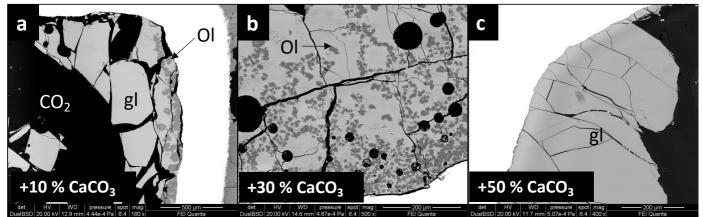


Figure 7. BSE images relative to RM_43 run with BM3 starting material at 2 kbar and 1200 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = Olivine; GI = Glass

T= 1300 °C BM1 QP_124

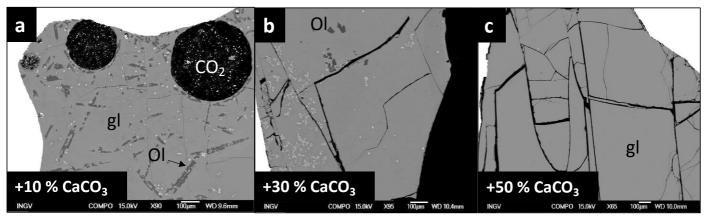


Figure 8. BSE images relative to QP_124 run with BM1 starting material at 2 kbar and 1300 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = olivine; GI = Glass.

T= 1300 °C BM2 RM_37

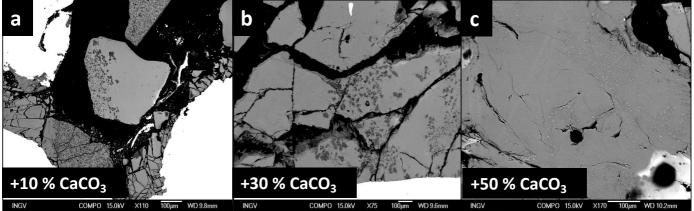


Figure 9. BSE images relative to RM_37 run with BM2 starting material at 2 kbar and 1300 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments.

T= 1300 °C BM3 RM_42

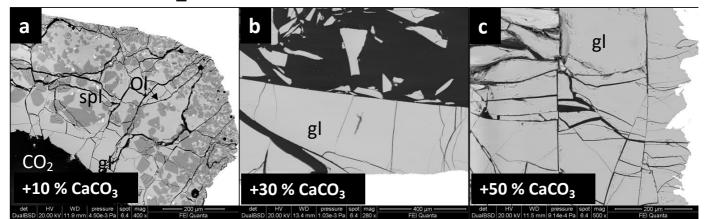


Figure 10. BSE images relative to RM_42 run with BM3 starting material at 2 kbar and 1300 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. Spl = Spinel. OI = olivine. GI = glass.

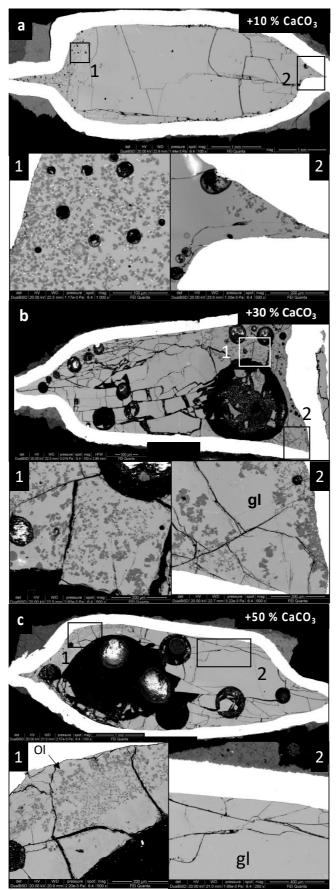


Figure 11. BSE images relative to the details of QP1_125 run with BM1 starting material at 2 kbar and 1200 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = OIivine. GI = Glass.

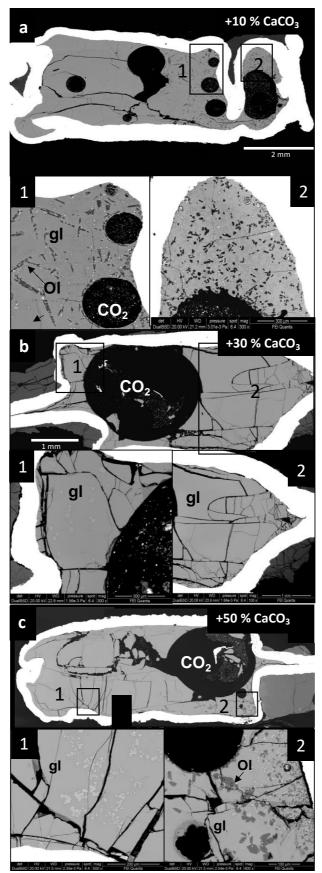


Figure 12. BSE images relative to the details of QP1_124 run with BM1 starting material at 2 kbar and 1300 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = OIivine. GI = Glass.

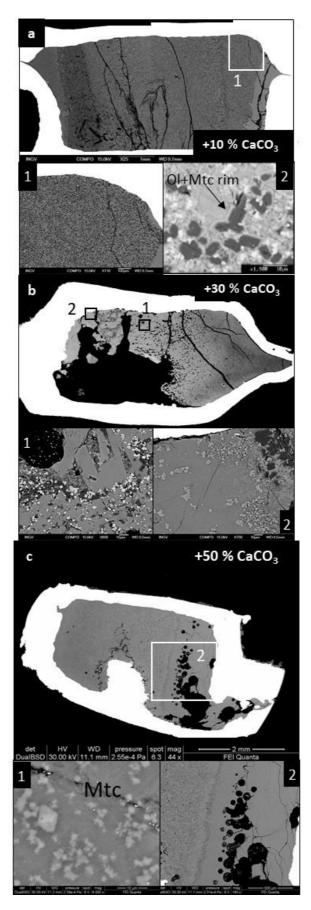


Figure 13. BSE images relative to the details of RM41 run with BM2 starting material at 2 kbar and 1200 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments. OI = Olivine. Mtc = monticellite.

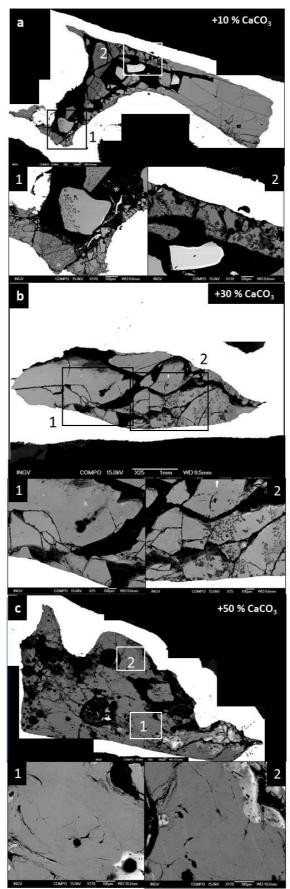


Figure 14. BSE images relative to the details of RM37 run with BM2 starting material at 2 kbar and 1300 °C as function of the CaCO₃ content mixed with the starting glass used for our experiments.

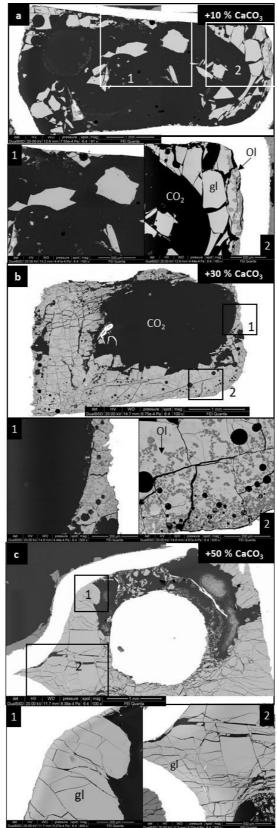


Figure 15. BSE images relative to the details of RM43 run with BM3 starting material at 2 kbar and 1200 °C as function of the $CaCO_3$ content mixed with the starting glass used for our experiments. OI = olivine; GI = glass.

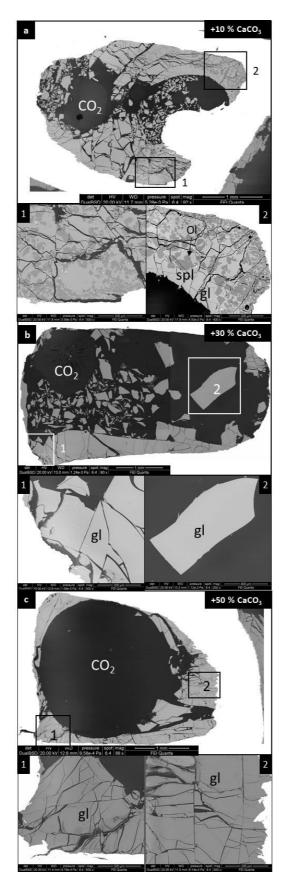


Figure 16. BSE images relative to the details of RM45 run with BM3 starting material at 2 kbar and 1300 °C as function of the $CaCO_3$ content mixed with the starting glass used for our experiments. OI = olivine; SpI = spinel; GI = glass.