

THE ROLE OF MELT/OLIVINE RATIO IN DISSOLUTION AND REACTIVE CRYSTALLIZATION: AN EXPERIMENTAL AND MICROSTRUCTURAL STUDY (ELECTRON BACK-SCATTERED DIFFRACTION – EBSD) AT 0.5 GPa

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INTRODUCTION

Microstructural and geochemical studies of olivine-rich troctolites have suggested that melt/rock ratios play a key role in the interaction between an olivine matrix (dunite) and an impregnating MORB-type melt, affecting the textural features and the geochemical composition of these rocks (*e.g.*, Suhr *et al.*, 2008; Drouin *et al.*, 2009, 2010; Renna & Tribuzio, 2011; Sanfilippo & Tribuzio, 2011, 2013; Sanfilippo *et al.*, 2014, 2015; Rampone *et al.*, 2016) and the evolution of MORBs in the lower oceanic crust (*e.g.*, Collier & Kelemen, 2010; Paquet *et al.*, 2016). In literature, two dominant petrogenetic hypotheses have been formulated on the origin of the olivine-rich troctolite. According to the first theory, the olivine-rich troctolites are olivine and plagioclase cumulates derived by a magmatic segregation as a response to cooling of primitive basalts emplaced within the lower crust and/or mantle-crust transition (Renna *et al.*, 2016). According to the second theory, the olivine-rich troctolites originated by the reaction between an olivine-rich matrix (dunite) and migrating MORB-type melts saturated in clinopyroxene and plagioclase. In the last decades, this interpretation has been invoked for several troctolites from oceanic (*e.g.*, Suhr *et al.*, 2008; Drouin *et al.*, 2009, 2010; Sanfilippo *et al.*, 2016) and ophiolitic environments (*e.g.*, Renna & Tribuzio, 2011; Sanfilippo & Tribuzio, 2011, 2013; Sanfilippo *et al.*, 2014, 2015; Rampone *et al.*, 2016). Recently, it has been demonstrated the hybrid and reactive nature of some olivine-rich troctolites from Mt. Maggiore peridotitic Massif (Alpine Corsica, France) and Erro-Tobbio ultramafic body (Western Alps, Italy) combining the results of microstructural and geochemical studies (Basch *et al.*, 2018, 2019). Two main generations of olivine were observed: i) large subhedral crystals with both straight and lobate rims and ii) smaller rounded undeformed grains. Clinopyroxene and plagioclase, occurring as interstitial phases between boundaries of adjacent olivine grains, do not show evidence of deformation (Basch *et al.*, 2019). As suggested by several authors, this textural evidence suggests a disequilibrium: the originally subhedral olivines have been partially corroded and the clinopyroxene and plagioclase crystallized simultaneously from the reacted melt (*e.g.*, Drouin *et al.*, 2009; Sanfilippo & Tribuzio, 2013; Sanfilippo *et al.*, 2016; Basch *et al.*, 2019).

From a chemical point of view, high X_{Mg} value (X_{Mg} = molar $Mg/(Mg + Fe^{tot})$) and NiO content in olivine have been theorized to support the hypothesis of a mantle origin of some troctolites (*e.g.*, Drouin *et al.*, 2009; Sanfilippo *et al.*, 2014; Rampone *et al.*, 2016). The interstitial phases like clinopyroxene and plagioclase have been generally used to define the chemistry of the reacted melt and the possible influence of pressure in the crystallization process. In detail, a high-Mg# clinopyroxene (*i.e.*, 89-90) is inconsistent with an origin by a low-pressure crystallization of MORB-type melt. The more likely hypothesis is that high-Mg# clinopyroxene forms when a relatively evolved, clinopyroxene saturated melt reacts with primitive cumulates (*e.g.*, Lissenberg & Dick, 2008; Lissenberg & MacLeod, 2016; Sanfilippo *et al.*, 2015; Basch *et al.*, 2018). Overall, it has been observed that the reaction processes lead to a more enriched chemical composition of the impregnating melt. It has been proposed that the reactive crystallization Liquid Line of Descent (LLDs) present a nearly constant MgO content coupled with a strong decrease in Ca# and higher SiO₂ and Na₂O contents than ones predicted in fractional crystallization (Collier & Kelemen, 2010).

From the experimental point of view, numerous studies have been focused on the role of peridotite-basalt interaction during melt migration in the upper mantle-crust transition zone through dissolution (*e.g.*, Daines & Kohlstedt, 1994; Lambart *et al.*, 2008; Van den Bleeken *et al.*, 2010, 2011) and crystallization experiments (*e.g.*, Tursack & Liang, 2012; Saper & Liang, 2014). Recently, Borghini *et al.* (2018) carried out reactive dissolution

and crystallization experiments between basalt and dunite (0.5 - 0.7 GPa) in order to constrain the origin of olivine-rich troctolite. Their results suggested that the initial melt/rock ratio strongly influences the extent of interaction and, thus, the final mineral abundances and chemistry. However, the reaction couple strategy used in their experiments did not allow a quantification of the role of melt/rock ratio. Yang *et al.*, (2019) performed experiments between a molten MORB and troctolite at 0.5 GPa in order to investigate the role of melt-rock reaction in affecting the mechanisms of dissolution-reprecipitation and chemical consequences of mineral phases and reacted melts. Hence, the role of different melt/rock ratio in the reactive dissolution crystallization processes has not been defined yet.

At this purpose, we performed isothermal experiments at 0.5 GPa in order to study how the reaction processes between an olivine matrix and a reacting MORB-type melt can influence the texture and chemistry as well as the amount of dissolved olivine and how the reacted melt evolved through the reaction. On the other hand, the step-cooled experiments allowed to study the crystallization process of the reacted melt by analyzing the resulted lithology from the crystallization of the reacted melt.

In this study, for the first time, the experimental procedure coupled with the chemical analysis allowed to quantify how different melt/olivine ratios affect the olivine dissolution process as well as the total amount of dissolved olivine and the residual reacted melt. The experiments aim to provide new detailed insights on melt-rock reaction processes by relating the chemical data obtained from Electron Microprobe Analysis (EMPA) with a detailed textural study on microstructures and intra-crystalline deformation of olivine grains using Electron Back-Scattered Diffraction (EBSD) analysis.

EXPERIMENTAL PROCEDURE

Ten experiments were carried out at 0.5 GPa in End-Loaded piston cylinder apparatus at the Laboratory of Experimental Petrology, Department of Earth Science “Ardito Desio” (University of Milano). The dunitic matrix has been simulated using San Carlos olivine ($Fe_{0.0}$) ranging from 36-63 μm , whereas the starting reacting melt is a MORB-type glass (AH6, Husen *et al.*, 2016) with an $X_{Mg} = 0.62$ ($X_{Mg} = Mg/(Mg + Fe^{tot})$) and an $X_{Ca} = 0.77$ ($X_{Ca} = Ca/(Ca + Na)$), mixed in different proportion (*i.e.*, the Initial Melt Amount - IMA, was equal to 10, 25 and 50 wt.%) (Fig. 1a). Out of ten experiments, seven were isothermal performed at 1200, 1250 and 1300°C for 24 hours, and three were step-cooled runs performed at a rate of 1°C/min from 1300°C for 24 hours down to 1100°C for 48 hours (Fig. 1b).

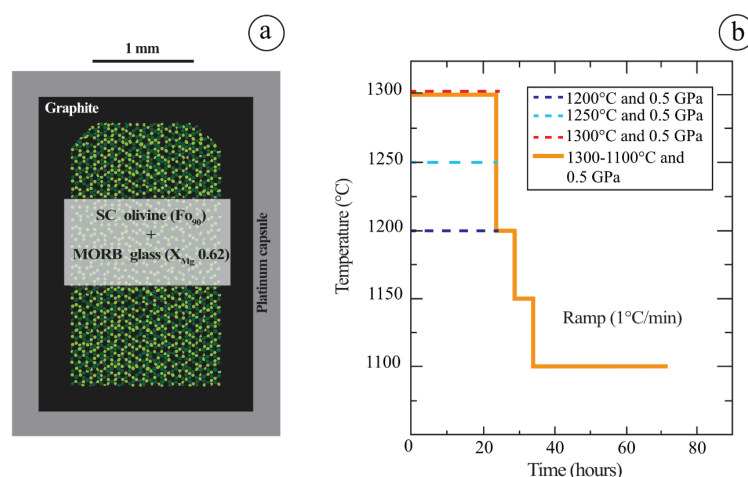


Fig. 1 - Schematic illustration of experimental setup used in this study: the melt/olivine mixture (black and green spheres respectively) is put in a double Platinum-Graphite capsule and (b) temperature vs. time path of the dissolution-crystallization experiments.

ANALYTICAL TECHNIQUE

Electron Microprobe analysis (EMPA) and Electron Back-Scattered Diffraction analysis (EBSD)

Run products have been texturally and chemically characterized by Electron Microprobe analysis – EMPA (BSE/SE images, X-ray chemical maps and WDS/EDS chemical analysis) using JEOL 8200 Super Probe at the Department of Earth Science “Ardito Desio”, University of Milano.

On the other hand, the microstructures of the experimental assemblages were investigated by Electron Back-Scattered Diffraction technique. The system used was a Bruker Quantax e-Flash HD system equipped with Argus diodes for FSE/BSE (forwardly and backscattered electron) imaging and attached to a Field Emission Gun (FEG) Scanning Electron Microscope (SEM) Zeiss Gemini 500, installed at the Platform of Microscopy of the University of Milano-Bicocca. EBSD raw data were first processed using the ESPRIT Software in order to remove wild-spikes or zero-solutions. EBSD post-processing texture analysis was done using MTEX (Mainprice *et al.*, 2011) an open-source MatLab toolbox. In order to understand how different melt/olivine ratio and T affect the olivine texture, EBSD maps allowed to investigate five geometrical parameters: 1) Equivalent diameter (μm) and 2) Grain Area (μm^2) define the olivine grain size, 3) the Shape Factor and 4) the PARIS Factor define the roundness and the tortuosity of olivine crystals, 5) the Aspect Ratio define the elongation of olivine grains. Furthermore, EBSD maps allowed to study the three intra-crystalline deformation parameters: 1) the Grain Orientation Spread (GOS) defines an average intra-granular deformation of a grain, 2) the Kernel Average Misorientation (KAM) allows to identify the sub-grains and 3) the Misorientation to Mean (Mis2Mean) allows to identify the kink bands.

RESULTS

Phase assemblages and texture

Run products of isothermal experiments at 1200°C consist of olivine, plagioclase, clinopyroxene and traces of glass, whereas at 1300°C consist only of olivine and glass. Texturally we distinguish two types of olivine: i) large subhedral crystals (up to 100 μm) with both straight and lobate rims and ii) smaller (5-20 μm) rounded grains. Olivine grain size increases with the IMA. Moreover, the number of olivine grains with regular morphology, as rounded and euhedral habit is higher than those larger with irregular habit.

Run products of step-cooled experiments at final T of 1100°C consist of olivine, plagioclase and clinopyroxene and traces of glass. In particular, the experiments with 25 wt.%IMA is characterized by the early clinopyroxene crystallization rather than plagioclase.

Minerals and glass chemistry

At 1100 and 1200°C, mineral chemistry of olivine suggests that X_{Mg} values are not correlated with different IMAs. At 1300°C, X_{Mg} in olivine increases from 0.89 ± 0.01 to 0.91 ± 0.01 with the increase of the IMAs. At constant high melt amount (50 wt.%), T strongly influences the NiO content in reacted olivine. In particular, it decreases with the increase of temperature from 1200 to 1300°C. At fixed T of 1300°C, the NiO content depends on the initial melt amount, decreasing with the IMAs. At 1100°C, the NiO content in olivine is not correlated with the IMAs.

Plagioclase in the isothermal experiments run at 1200°C has $X_{\text{An}} = 0.68 \pm 0.03_{\text{IMA10wt.\%}}$, $0.61 \pm 0.06_{\text{IMA25wt.\%}}$ and $0.66 \pm 0.02_{\text{IMA50wt.\%}}$. In the step-cooled runs, plagioclase was detected only in the experiment with 50 wt.%IMA and has $X_{\text{An}} = 0.71 \pm 0.02$.

Clinopyroxene in the isothermal experiments at 1200°C presents $X_{\text{Mg}} = 0.89 \pm 0.00_{\text{IMA10wt.\%}}$, $0.89 \pm 0.02_{\text{IMA25wt.\%}}$ and $0.84 \pm 0.02_{\text{IMA50wt.\%}}$. Compared to isothermal runs, clinopyroxene resulted from the step-cooled experiments have higher X_{Mg} values ($0.91 \pm 0.01_{\text{IMA25wt.\%}}$ and $0.93 \pm 0.01_{\text{IMA50wt.\%}}$).

Reacted glasses at 1200°C have higher SiO_2 and lower X_{Mg} than glasses at 1300°C. At 1100°C no correlation is observed between the IMAs and final glasses compositions.

EBSD analysis: geometrical parameters

In order to understand how the reaction process might affect the olivine texture at given T and different IMAs, the EBSD analysis allowed to define quantitatively the following geometrical parameters: Equivalent diameter, Grain Area, Aspect Ratio, Shape Factor and PARIS Factor. The Aspect Ratio factor shows that the reaction at 1200°C results in more elongated crystals than those at 1300°C. On the other hand, at 1300°C the reaction does not affect the olivine texture in terms of elongation between smaller and larger grains as a function of different initial melt amounts. At 1100°C, no correlation it is observed between the olivine grain size and its elongation grade at different IMAs.

The Shape and PARIS Factors allowed to study the morphology of olivine grains. Both at 1200 and 1300°C, it has been observed that the larger grains have higher values of Shape and PARIS Factors. In particular, the reaction experiments at 1300°C result in more tortuous and less rounded grains than those at 1200°C. Moreover, the EBSD data allowed to quantify the amount of smaller rounded grains confirming that at 1300°C the increase of IMA from 10 to 50 wt.% leads to the decrease of the total amount of smaller rounded grains and total modal olivine (vol%). At 1100°C, negligible differences are observed, in terms of tortuosity grade, between smaller and larger olivine crystals.

EBSD analysis: intra-crystalline parameters

Before evaluating how different T and IMAs might influence the olivine intra-crystalline deformation during the interaction processes, it is worth to note that the San Carlos olivine, used as starting material, is not naturally deformed. In particular, during the first 10 minutes of the experimental procedure the T is at about 400°C, hence, the assembly is still relatively stiff, and the sample does not deform plastically, until it reaches the desired T of 1200-1300°C. The average intragranular deformation parameter defined by the Grain Orientation Spread (GOS), indicates that no differences occur between the two different olivine morphologies. The smaller and rounded olivine has $GOS = \sim 0.49$ and the larger one with straight and lobate rims has GOS value $= \sim 0.41$. Moreover, the Mis2Mean and KAM factors suggest that at high- T and 50 wt.% IMA, the kink bands and sub-grains in olivine are almost entirely recovered.

At 1100°C, the reactive dissolution and crystallization processes coupled to the kinetic result in low GOS value $= \sim 0.53$, independently of the olivine grain size and different IMAs.

Phase abundance

The phase abundance was obtained by mass-balance calculations based on the least squares (LS) method performed using Mathematica Software. In order to take into account, the error propagation of uncertainties in the analytical determination of the phases chemical composition, a Monte Carlo approach was considered. Due to the negligible chemical differences between smaller-rounded olivine grains and larger ones with irregular habit, the amount of neo-formed olivine cannot be defined by mass-balance calculations. Hence the experiments are described in terms of total amount of olivine and the proportions of interstitial phases, *i.e.*, plagioclase, clinopyroxene and residual glass.

At 1200°C, the higher IMA (50 wt.%) results in an increase of modal olivine (+ 8 wt.%) and in higher percentages of interstitial phases, *i.e.*, plagioclase, clinopyroxene (5.3 wt.%_{IMA10} to 24 wt.%_{IMA50} and from 3 wt.%_{IMA10} to 14 wt.%_{IMA50}, respectively). At 1250 and 1300°C in the runs with 50 wt.% IMA, olivine final proportion is ~ 46 wt.%, suggesting that high melt amount of 50 wt.% led to an increase of olivine dissolution at increasing of IMA.

At 1100°C, the crystallization of residual glass in the run with 50 wt.% IMA results in a slightly increase of the final amount of olivine (+ 5 wt.%) and in the gradual appearance of the interstitial phases.

DISCUSSION

Textural comparison between experiments and natural rocks

The lithologies and textures produced in the melt-olivine reaction experiments run at 1200-1300°C are comparable to those documented in natural rocks (*i.e.*, dunite, Ol-rich troctolite and Ol-gabbro) recovered from oceanic (*e.g.*, Suhr *et al.*, 2008; Drouin *et al.*, 2009, 2010; Sanfilippo & Tribuzio, 2013; Sanfilippo *et al.*, 2015, 2016) and ophiolitic environments (*e.g.*, Renna & Tribuzio, 2011; Sanfilippo & Tribuzio, 2011, 2013; Sanfilippo *et al.*, 2014, 2015; Rampone *et al.*, 2016; Renna *et al.*, 2016).

At 1200°C, different IMAs strongly affect the resulting lithology leading to the formation of dunite (10 wt.%_{IMA}), Ol-rich troctolite (25 wt.%_{IMA}) and Ol-gabbro (50 wt.%_{IMA}). At 1300°C, the reaction process allows to study how the dunite matrix can be impregnated by a reacting MORB-type melt varying the IMA. In particular, the interaction process with 50 wt.%_{IMA} strongly affects the olivine morphology, enhancing the reaction promoting the olivine crystal growth and dissolution, leading to high tortuosity of olivine grain boundaries. The microstructural data suggest that high-*T* and high melt amount can influence the impregnation process resulting in the formation of high-porosity dunite channels at mantle-crust transition.

At 1100°C, the resulted lithology is influenced by the different IMAs resulting in melt-bearing dunite (10-25 wt.%_{IMA}), and Ol-gabbro (50 wt.%_{IMA}). The EBSD data reveals that the olivine matrix in step-cooled experiments is texturally more homogeneous than one in isothermal runs. This difference could be related to the run duration: isothermal runs are carried out at 24h, whereas the step-cooled ones are performed for 72h.

In the step-cooled run with 25 wt.%_{IMA} the crystallization of the reacted melt leads to the early appearance of clinopyroxene rather than plagioclase. This is probably related to a combined effect of the MgO enrichment (Husen *et al.*, 2016) and small amounts of H₂O in reacted melts (Danyushevsky *et al.*, 2001).

Olivine chemistry: comparison between experiments and natural rocks

High X_{Mg} value and NiO content in olivine have been indicated as a marker of a mantle origin of olivine matrix in some troctolites (*e.g.*, Drouin *et al.*, 2009; Rampone *et al.*, 2016; Sanfilippo *et al.*, 2014). The use of Ni-free starting melt in our experiments leads to an olivine with lower NiO content than its natural counterpart. Reaction experiments performed at 1200°C show NiO contents comparable to those observed in natural rocks (*e.g.*, Renna & Tribuzio, 2011; Sanfilippo & Tribuzio, 2013; Basch *et al.*, 2019) in contrast to experiments performed at 1250 and 1300°C that, instead, show lower NiO contents (0.10 wt.% \pm 0.08 in run at 1250°C, 0.16 wt.%_{IMA10} \pm 0.08, 0.04 wt.%_{IMA25} \pm 0.08 and 0.02 wt.%_{IMA50} \pm 0.02 at 1300°C). On the contrary, the isothermal experiments run at 1300°C are characterized by lower Ni content than natural Ol-rich troctolites. At 1100°C, the NiO content is lower than data from natural rocks (*e.g.*, Renna & Tribuzio, 2011; Sanfilippo & Tribuzio, 2013; Basch *et al.*, 2019).

Plagioclase and clinopyroxene chemistry: comparison between experiments and natural rocks

Low X_{An} value in plagioclase and high X_{Mg} value in clinopyroxene from the reaction-crystallization lie within the range of anorthite content in Pineto gabbroic rocks (Sanfilippo & Tribuzio, 2013) and Erro-Tobbio troctolites (*e.g.*, Rampone *et al.*, 2016; Basch *et al.*, 2018). The experimental data of this study agree with chemical evidences observed in natural Ol-rich troctolites and attributing to the melt-rock reaction processes involving the olivine dissolution and subsequent crystallization of interstitial plagioclase and clinopyroxene (*e.g.*, Basch *et al.*, 2018, 2019). Further chemical evidence for a reactive origin of Ol-rich troctolite is that anorthite content from isothermal and step-cooled experiments are not comparable to ones from equilibrium crystallization runs performed by Husen *et al.* (2016). Indeed, X_{An} of plagioclase in equilibrium crystallization experiments is higher than ones in reaction-crystallization experiments of this study and increases with the increase of *T* from 1150 to 1200°C.

Glass chemistry: comparison between experiments and natural rocks

The chemical composition of the residual reacted melt in isothermal experiments were compared to the MORB field ($X_{Mg} > 0.67$; Gale *et al.*, 2013). The reacted melts at 1200°C are characterized by higher values of SiO₂, Al₂O₃, Na₂O but lower MgO contents compared to those documented in primitive MORB. The CaO contents are similar to those documented in primitive MORB.

On the contrary, the chemistry of the reacted melts resulted from the reaction at 1300°C, is consistent to the MORB range in terms SiO₂, Al₂O₃, and CaO. Reactive crystallization at 1200°C results in the decreasing of the MgO content in melt coupled to a nearly constant X_{Mg} , whereas the SiO₂ and Na₂O content evolve to higher values than ones at 1300°C. Hence, this chemical evidence suggests that the fractionation of melt through reactive crystallization at lower T of 1200°C results in more evolved melts than those after the reaction at 1300°C (*e.g.*, Collier & Kelemen, 2010). At 1100°C the reacted melt composition is not comparable to the MORB field.

NiO content in the experiments generally increases compared to the initial melt composition. Furthermore, in the reaction-crystallization experiments run at 1300-1100 and 1200°C, no correlation is observed between the NiO content and different IMAs. On the contrary, at 1300°C the NiO content increases proportionally to IMAs.

Olivine deformation recovery

The olivine dissolution and re-precipitation experiments revealed that the reaction process at 1300°C results more efficiently in the “recover” of the plastic deformation of the olivine grains. Fig. 2 shows that the employment of a high melt amount during the reaction with an olivine matrix, strongly enhances the annealing processes, independently of the grain size and shape. The microstructural features observed in olivines from reactive crystallization experiments (at final $T = 1100^\circ\text{C}$), suggested that the reactive dissolution and crystallization coupled to the kinetic also promote the “annealing” process of plastic deformation independently of the grain size and IMAs. Several authors observed two main generations of olivine: i) large subhedral and “deformed” crystals with both straight and lobate rims (*i.e.*, Olivine₁) and ii) smaller rounded “undeformed” grains (*i.e.*, Olivine₂) (*e.g.*, Drouin *et al.*, 2010; Rampone *et al.*, 2016; Basch *et al.*, 2018, 2019, 2021; Ferrando *et al.*, 2018). However, the distinction of the two olivine generations based on their deformation grade in reaction-crystallization experiments, is not totally correct. In fact, the olivine grains from the reaction experiments at 1200-1300°C with 50 wt.%IMA are undeformed (GOS = ~0), independently of the shape and the grain size. Hence, it is difficult to define the pre-existing olivine grain (Olivine₁) and a new crystallized on (Olivine₁). The study of olivine intra-crystalline deformation provides new insights on the role played by different melt/olivine ratio in the compaction and melt migration in gabbroic rocks of the lower oceanic crust.

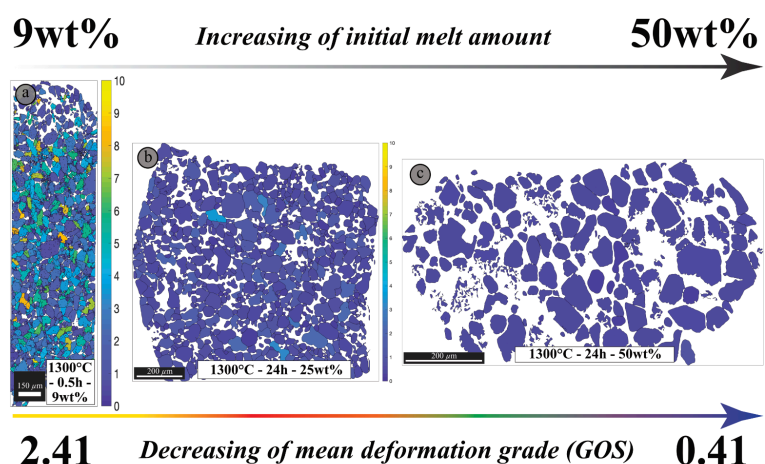


Fig. 2 - EBSD data show the decreasing of the olivine deformation grade with the increase of initial melt amount in reaction experiments performed at 1300°C and 0.5 GPa (the run with 9 wt.%IMA is from Francomme, PhD Thesis 2016-2017).

CONCLUDING REMARKS

Experimental results revealed that the reaction between an olivine matrix (Fo₉₀) and a relatively evolved MORB-type melt (X_{Mg} = 0.62) affects the olivine textures and the chemistry of mineral phases. The detailed microstructural study on olivine grain provided new insights on the origin of olivine-rich troctolite by a melt-dunite reaction process at the mantle-crust transition. The main results of isothermal experiments can be summarized as follows:

- At 1200°C, the reaction between olivine and different melt amount allows to study the resulted lithology.
- At 1300°C, a relatively high initial melt (50 wt.%) enhances the reaction promoting olivine dissolution and crystal growth which leads in an increase of olivine grain boundaries tortuosity.
- The NiO is influenced by *T* and different melt/olivine ratio.
- The interaction process strongly controls the chemical evolution of the reacted melt.
- The interaction between an olivine matrix and a high melt amount (50 wt.%) at 1300°C, strongly affects the pre-existing matrix promoting the “annealing” process. In this scenario, is difficult to define a pre-existing olivine (Olivine₁) and a new crystallized one (Olivine₂).

The main results provided by the step-cooled experiments can be summarized as follows:

- Reactive dissolution and crystallization experiments result in melt-bearing dunite (10-25 wt.%_{IMA}) and olivine-gabbro (50 wt.%_{IMA}).
- The olivine textures result in elongated crystals with higher tortuosity grade independently of the grain size and initial melt amount.
- Early clinopyroxene crystallization rather plagioclase at 0.5 GPa. This is probably related to a combined effect of the MgO enrichment and small amounts of H₂O in reacted melts.
- The reactive dissolution and crystallization processes coupled to the kinetic efficiently promote the “annealing” process of olivine crystals, resulting in a more homogenous texture.

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