Activity report for the InterRidge Graduate Student Fellowship awarded in 2018 to Simone Pujatti.

The funds granted through the InterRidge Graduate Student Fellowship program allowed me to travel to Utrecht University (NL) in February 2019 to perform ultra-high resolution focused ion beam scansion electron microscopy (FIB-SEM) analysis on one thin section prepared from the Ocean Drilling Program core sample 209/1274A/3R/1/61-71, which was recovered from the Mid-Atlantic Ridge (MAR). The instrument used was operated by Dr. Oliver Plümper (Assistant Professor at Utrecht University).
Our aim was to collect imaging data with ultra-high resolution recording the nanoporosity content of serpentinized rocks. The underlying hypothesis of this research project is that the interaction of peridotites with aqueous fluids produces serpentine minerals containing a network of nanopores. The latter act as pathways for the supply of fluids to the reaction front, allowing the serpentinization reaction to proceed to completion and produce a completely serpentinized peridotite.

Methodology

A FIB-SEM instrument integrates a traditional scansion electron microscope with an ion gun, a gas injector to apply a carbon coating that prevents charging (alternatively gold or platinum are employed instead of carbon) and a sample stage that can be tilted to obtain an angle of 50° to 60° between the electron beam and a focused ion beam (typically Gallium ions are used). The ion beam mills a series of consecutive nanometric slices. After one slice is removed, the system collects a back-scattered electron (BSE) image. A consecutive iteration of this approach allows to obtain a stack of 2D images that can be reconstructed in 3D with commercially available image processing software. The original plan entailed analyzing two different samples to guarantee the representativity of the results. However, the high resolution required to achieve detection of the nanoporosity in the samples complicated the acquisition stage, since both electronic and stage drifting resulted in several unsuccessful runs and limited the extent of sample analysis we could achieve during the measurement timespan covered by the granted funds. In other instances, the redeposition of amorphous material after the milling operated by the ion gun covered the field of view detected by the electron beam, making the resulting dataset unusable.
Regardless of the complications encountered, two datasets were successfully acquired using the FEI Helios NanoLab™ 660 DualBeam SEM/FIB available at Utrecht University. In the first one, labelled “MAR01”, we achieved a resolution of 7.71 nm in the XY directions and 10 nm along Z. In the second one, labelled “MAR02” we successfully pushed the limits of the instrument and obtained a tomographic dataset with a resolution of approximately 1.69 nm in the XY directions and 10 nm along Z. These datasets were processed at the University of Calgary with the 3D visualization and data analysis packages Pergeos and ImageJ. After applying a series of filters to the 2D stacks of images to highlight the internal microstructures of the sample, subvolumes of ~ 32 μm^3 and 7 μm^3 were selected respectively from MAR01 and MAR02 to be rendered in 3D for microstructural quantification.

Results & discussion

Both datasets MAR01 and MAR02 showed the presence of a channeled wormhole-like porous network found at the serpentinization front. This porosity shows a preferential orientation parallel to the grain boundaries of the partly replaced olivine. This structure is interpreted as evidence of the incipient stage of replacement, entailing that serpentinization is initially dominated by the dissolution of olivine.
Additionally, a smaller sized nanoporosity was identified in the higher resolution dataset MAR02. This porous structure is found at a distance from the reaction front and is scattered throughout the fully replaced serpentine matrix. Importantly, it is subparallel to the coarser channeled porosity. It is interpreted as the product of an advanced stage of alteration, when the reaction approaches equilibrium and the precipitation of serpentine minerals dominates over dissolution, thus partially clogging the channeled porosity. The nanoporosity represents the remnants of what initially was channeled porosity.
These results are evidence of the dynamic evolution and transient nature of porosity in serpentinites. The subparallel orientation of the pores has likely been inherited from a primary crystallographic-preferred orientation (CPO), originally formed as a magmatic texture in the peridotite and subsequently transferred to the alteration products. This interpretation bears strong implications for the extent of serpentinization achievable in natural settings, since primary CPOs would control the direction of fluid transport and hence the direction of propagation of the serpentinization front. This work urges researchers who produce physical models of the extent of serpentinization in the oceanic lithosphere to consider the presence of an anisotropic porosity, which governs the directions along which serpentinization reactions can progress.

Conclusions

The InterRidge Graduate Student Fellowship allowed me to visit Utrecht University and acquire two ultra-high resolution tomographic datasets by means of FIB-SEM techniques. The high resolutions obtained allowed to achieve the intended goal, since nanoporous structures were traced in both datasets collected in the Netherlands. The data were processed at the University of Calgary and presented in poster format at the conference AGU Fall Meeting 2019 (an ePposter is available at https://docs.google.com/viewer?url=https://agu.confex.com/agu/fm19/mediafile/Handout/Paper593483/Transient%2520porosity%2520generated%2520at%2520the%2520reaction%2520front.pdf). The poster presentation received highly positive feedbacks and attracted the interest of many experts in the field, granting a great opportunity for networking as several researcher proposed potential collaboration for the future. Presently, the collaboration with Utrecht University is ongoing, since we are planning to perform electron back-scattered diffraction (EBSD) / transmission electron microscopy (TEM) analysis to verify the hypothesis that primary minerals in peridotites have a CPO that is inherited after hydration reactions by the nanopores in serpentinite rocks. If the results validate this hypothesis, confirming the importance of primary textures in mantle rocks, a manuscript will be prepared for publication in high-impact scientific journals during spring 2020.