# A shallow origin for diamonds in ophiolitic chromitites

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## Data Repository 1: Analytical methods

### 1.1. Sample preparation

Chromitite samples were collected from Tehuitzingo serpentinites. Thin and thick polished sections were prepared at the *Serveis de lámina prima* of the University of Barcelona. The thin sections were polished using diamond abrasive paste of 1 μm particle size. Due to the fact that most of the identified diamonds are bigger than 1 μm, we can exclude the possibility of them being the result of contamination by polishing material. A second set of duplicate thin sections from samples containing diamond was prepared using $Al_2O_3$ as a carbon-free polishing medium to exclude contamination. An ultrasonic bath was used to remove any remaining polishing material from the sample.

### 1.2. Field emission scanning electron microscopy (FE-SEM)

The samples were examined under a Field Emission Scanning Electron Microscope (FE-SEM) Jeol JSM-7100 at 20 kV, at the *Centres Científics i Tecnològics de la Universitat de Barcelona* (CCiTUB), Spain. The FE-SEM is equipped with SE, BSE and EDS detectors.

### 1.3. Micro-Raman spectroscopy

Micro-Raman spectra of the inclusions and chromite identified in the thin section and the polished monolayers were obtained with a HORIBA JobinYvon LabRam HR 800 dispersive spectrometer equipped with an Olympus BXFM optical microscope in the CCiT-UB. Non polarized Raman spectra were obtained in confocal geometry by applying a 532 nm laser, using a 100x objective (beam size around 2 μm), with 5 measurement repetitions for 10 seconds each. The instrument was calibrated by checking the position of the metallic Si band at ~520 $cm^{-1}$. The micro-Raman spectra were processed using the LabSpec® software (JobinYvon).

### 1.4. Electron probe microanalyses (EPMA)

The analyses of chromite and the inclusions were done with four-channel CAMECA S x 50 electron microprobe at the CCiTUB. The analytical conditions were 20 kV accelerating voltage, 20 nA beam current, 2 μm beam diameter, and counting

time of 10 s per element, using WDS detectors. Calibrations were performed using natural and synthetic standards: chromite (Cr, Al, Fe), periclase (Mg), rhodonite (Mn), rutile (Ti), NiO (Ni), sphalerite (Zn), and metallic V. The chemical data for Cr-spinels were stoichiometrically recalculated in order to distinguish FeO from $Fe_2O_3$ according to the procedure described by Carmichael (1967). On the other hand, the analytical conditions used for the inclusions were 12 kV accelerating voltage, 1 nA and 60 s (live time) of counting time per element, using EDS detectors. These conditions were used in order to improve spatial resolution and minimize the electronic impact and the error derived from the imperfect polished of the sample. Calibrations were performed using natural and synthetic standards: albite (Na), periclase (Mg), corundum (Al), wollastonite (Si and Ca), orthoclase (K), rutile (Ti), rhodonite (Mn), $Cr_2O_3$ and $Fe_2O_3$

### 1.5. FIB-TEM and EELS

One of the diamonds, the d6 diamond from sample CR-05, was selected to be studied by transmission electron microscopy (TEM). In order to study the inclusion, a lamella was extracted from the chromite grain at the Barcelona Research Center in Multiscale Science and Engineering, from the *Universitat Politècnica de Catalunya* (UPC). The TEM lamella preparation was done using a double-beam workstation (Neon40, CarlZeiss) equipped with a Schotky FE-SEM and FIB $Ga^+$ columns. The place where the inclusion was found was first covered by a thin layer (~100 nm) of Pt by means of e-beam assisted gas deposition. The Pt layer acts as a protection layer when milling and polishing the lamella. The bulk material on both sides of the lamella was removed first by coarse milling with 30kV:10nA ion current and subsequent finer milling steps with 30kV:2nA and 30kV:500pA currents until a lamella thickness of about 1 μm was achieved. It contained the entire embedded inclusion. The lamella was then lift-out and transferred to a TEM grid by using Kleindiek micromanipulator with a tungsten tip. Ion-beam assisted Pt deposition was used to fix the lamella to the micromanipulator tip and after to the TEM grid. The final step was polishing the lamella only in the place where the inclusion is located until an electron transparency was achieved. This was done using milling steps with 30kV:50pA and 30kV:10pA ion currents. The electron transparency was monitored by the conventional Everhart-Thornley SE detector and 5kV e-beam energy. After achieving electron transparency, the lamella was quickly polished using low energy 5kV:10pA ion current in order to

reduce the amorphization damage on the surface. The resulting lamella thickness of the inclusion was ~95 nm.

The TEM study of the FIB section was performed at the CCiTUB by a JEOL JEM-2100 LaB6 transmission electron microscope with energy dispersed analysis of X-rays (EDX), operating at 200kV in STEM mode. The beam size used in this mode is ~15 nm. The spectrometer is an Oxford Instruments INCA x-sight, with Si (Li) detector. Images were acquired using a Gatan CCD Camera Orius SC1000 and treated with the software Digital Micrograph Version 1.71.38

The electron energy loss spectroscopy (EELS) was performed at the *Centro de Instrumentación Científica de la Universidad de Granada* (CIC-UGR) by a Zeiss Libra 120 plus LaB6 transmission electron microscope with Omega energy filtering, operating at 120kV in STEM mode. The EELS analyses were performed at 80000x magnification, with a current emission of 1-2 μA on an area of 300 nm of diameter.

**References:**

Carmichael, I.S.E., 1967, the iron-titanium oxides of salic volcanic rocks and their associated ferro-magnesian silicates. Contributions to Mineralogy and Petrology, v. 14, p. 36–64