Panchromatic SEM-CL imaging of microdiamonds and other minerals in uncoated thin sections

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High-pressure granulites of the Bohemian Massif, which belongs to European Variscan belt, provide a unique material – recently described micro-diamonds (Kotková et al., 2011). In-situ microdiamonds occur both in felsic quartzofeldspathic and intermediate garnet-pyroxene granulites. The microdiamond inclusions in kyanite and garnet grains reach 5-30 μm in size. Because of the small size of the examined micro-diamond inclusions, their study requires micro- and nano-techniques with carefully though-about sample preparation. For this reason, normal-thickness (30 μm) as well as thick (100 and 300 μm) thin sections were tested. Importantly, the sections were not polished using diamond abrasives in order to avoid the risk of contamination.

The samples were first studied under transmitted and reflected light using Olympic BX 41 optical microscope to identify micro-diamond inclusions in major silicates. Second, cathodoluminescence (CL) analysis, as quite common and very useful method for revealing internal structure of various minerals including diamonds, was chosen to determine the way how these inclusions and surrounding material were formed.

For the CL application, the thin sections were not coated with carbon, to enable a follow-up study using other micro-analytical methods such as micro-Raman spectroscopy. For instance, the carbon from coating could be mistaken for a retrograde carbon rimming the diamond. Furthermore, graphite inclusions in the diamond could be overlooked because of the contamination. Evidently, the specific conditions of the CL analysis had to be adjusted to gain the highest possible quality results.

In general, there are two CL methods depending on used equipment – OM-CL (hot or cold cathode) and SEM-CL. Finally, SEM-CL was chosen for its better resolution and the possibility to use high beam currents under low-vacuum conditions on uncoated samples. The imaging has been done using the TESCAN scanning electron microscope (SEM) VEGA3 (LaB6 emitter), equipped with TESCAN panchromatic cathodoluminescence (CL) detector and ultra-fast YAG scintillator back-scattered electron detector (BSE). The samples were observed in Low Vacuum mode (10 to 100 Pa) and in a specific atmosphere of N_2. Accelerating voltages for the SEM were 25 to 30 kV.

The diamond inclusion in the garnet has a high CL in contrast to the garnet, thus the study of the diamond internal structure is possible. On the other hand, the diamond enclosed in kyanite appears as a dark grain without zoning due to the high CL characteristic of kyanite. In conclusion, even though the adjustment of the investigation is sufficient for the diamond identification, certain restriction for the detailed detection of the diamond structure has been revealed.

It is also possible to observe spectacular kyanite zoning under CL. Although other trace elements can play a role, Fe^{2+} is known as a CL quencher. The location of the micro-diamonds in relation to the zoning can therefore reflect the diamond-forming processes (reduction – oxidation). In addition, zoning of feldspars was noted. The CL zoning of the grains, which reveal the retrograde evolution, has not been determined yet.

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Reference:

Kotková J, O´Brien PJ, Ziemann M. (2011) Diamond and coesite discovered in Saxony-type granulite: Solution to the Variscan garnet peridotite enigma. Geology 39:667–670