Microinclusions in polycrystalline diamonds: insights into processes of diamond formation

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Polycrystalline diamond aggregates (framesites) contain silicates of eclogitic and peridotitic affinity (e.g. Kurat and Dobosi, 2000). The minerals occur mostly in interstices and are intimately intergrown with the diamonds, indicating contemporaneous crystallization within the diamond stability field in the Earth’s mantle. In addition to silicates, rarer phases such as Fe-carbide can sometimes be found in framesites that record unusually low local oxygen fugacity at the time of their formation (Jacob et al., 2004). Furthermore, while most gem-sized diamonds have old, often Archaean formation ages, some polycrystalline diamond aggregates have been shown to form directly preceding the kimberlite eruption (Jacob et al., 2000). Thus, these samples may provide a unique source of information on the nature and timing of small scale processes that lead to diamond formation and complement evidence from gem-sized diamonds.

Here, we present a study of micro- and nano-inclusions in diamonds from a polycrystalline diamond aggregate (framesite) from the Orapa Mine (Botswana) and combine results from TEM/FIB analyses with high-resolution computerized micro-tomography (HR-μ-CT) and electron microprobe analyses to further constrain the formation of diamond in the Earth’s mantle.

Results
In total, 14 microinclusions from fifteen FIB foils were investigated. Micro- and nano-inclusions identified by TEM were smaller than 1μm down to ca. 50nm in size, and are both monomineralic and multi-phase. The cavities are often lath-shaped and oriented parallel to each other; many show lattice dislocations in the surrounding diamond. In addition, inclusions are found along open cracks within the diamond single crystals. Mineral phases in the microinclusions comprise rutile, omphacite and a FeS phase (pyrrhotite). The multiphase inclusions most often consist of cavities that are only partly occupied (less than 50% of the total space), suggesting that the empty space was originally filled by a fluid. One multiphase inclusion was found to be still fluid-bearing, showing characteristic continuous changes in diffraction contrast due to density fluctuations caused by the electron beam. No other elements than carbon were detected during AEM of this area which suggests that the fluid consists of relatively pure C-H-O species. In addition to the fluid, this inclusion contained fine-grained FeS, a silicate phase rich in Fe, P, Mg, Al, Ca and K and a quench phase, rich in Fe, P and Si.

Macroinclusions (>5μm) are magnetite, often surrounded by hematite, FeS, low-Cr garnet (Py50Alm39Grs11) and omphacite (Jd23). Garnet and cpx were found as non-touching inclusions and yield 1256°C at 5 GPa. Most of the magnetite inclusions are single crystals and some are strongly deformed with signs of recrystallization. Hematite occurs as porous aggregates of nano-granules of ca. 5-7 nm sizes.

High Resolution μ-Computer Tomography (HR-μCT) shows pores in the sample and the included mineral phases as areas of differing grey-values. These are a direct function of the specific x-ray density of the specific phase and can be used to differentiate oxides and silicates. Based on the 3D tomogram, the amount of pores per total volume of the diamond plus inclusion matrix is calculated to be 0.65 vol%, while magnetite inclusions amount to 3.16 vol%. The average equivalent radius of the magnetite grains (radius of a sphere with the same volume as the grain) is 17.8 μm, while that of the pores is 12.6μm.

Discussion
The occurrence of omphacite, rutile and FeS as microinclusions within the diamond crystals clearly shows that
these phases are cogenetic to the diamonds. However, magnetite and hematite were only encountered as large inclusions in cavities that appear to be interstitial porosity. Moreover, analysis of the equivalent radius distribution of the pores and the magnetite inclusions derived from HR-µCT shows a complete overlap of the mode, indicating that magnetite preferentially fills the porosity in the sample. Furthermore, hematite occurs exclusively along the outer rim of the magnetite crystals and textural features suggest that this phase is a late replacement product of magnetite. This shows that the magnetite-bearing cavities were not shielded from the outside by the host diamond and may indicate that magnetite itself was introduced after diamond formation or during a secondary event that may still, however, have taken place at PT conditions of the diamond stability field. The microinclusion suite described here is distinct from that found in fibrous (e.g. Klein-BenDavid et al., 2007) and in microdiamonds (Kvasnytsya et al., 2006). Carbonates, halides and phosphates, that are typical for fibrous and microdiamonds were not encountered in our study. Instead, the microinclusion suite found in the framesite consists of the typical eclogitic minerals (rutile, garnet, omphacite, sulphide) plus a C-H-O fluid.