



Nano-correlative microscopy (TEM/APT) constrains the nature and timing of nanoclusters formation in monazite crystals

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Nano-structural and chemical characterization with Transmission Electron Microscope (TEM) of 1037 ± 19 Ma monazite crystals from an ultra-high-temperature granulite from Rogaland (Norway), revealed exsolutions of CaSO_4 nanoclusters in S-rich cores of these monazite crystals. These CaSO_4 nanoclusters, 5-10 nm in size, are homogeneously distributed within the monazite volume with a short modulation period of 15-25 nm. The cluster-matrix interfaces being almost coherent with slight disorientation of the lattice. To constrain the mechanisms and timing of nanoscale element mobility in monazite during homogenous exsolution and its impact on nano- and micro-geochronology, Atom Probe Tomography (APT) was combined to the TEM data. The APT $^{208}\text{Pb}/^{232}\text{Th}$ and $^{208}\text{Pb}/^{206}\text{Pb}$ ratios measured at nanoscale, revealed that radiogenic Pb was trapped during the nanoclusters formation and that the exsolution took place ~ 40 Myr after monazite crystallization. . Our study revealed that the U–Th–Pb systems were open at the nano-scale, leading to the separation of two different $^{208}\text{Pb}/^{232}\text{Th}$ reservoirs: a monazite matrix ($^{208}\text{Pb}/^{232}\text{Th}=0.0675$) and CaSO_4 -nano-clusters ($^{208}\text{Pb}/^{232}\text{Th}=0.2474$). However, the presence of nanoclusters induces only a minor modification of the bulk $^{208}\text{Pb}/^{232}\text{Th}$ ratio at the microscale ($\sim 1.5\%$ compared to the matrix). Since nanoclusters are homogeneously distributed in the sample and the Pb trapped inside nano-clusters is radiogenic, the U-Th-Pb geochronological systems remained closed at the micrometer-scale. Our results therefore validate the previous microscale dating and geochronological interpretations of Laurent et al. (2016), which argued for pulsed UHT metamorphism ($> 900^\circ\text{C}$) in contrast to protracted UHT metamorphism over more than 120 Myr in Rogaland as previously proposed and underline the usefulness of sulphur in monazite tracer despite phase exsolution. Finally, our study emphasizes the capabilities combining APT and TEM studies on the same sample to extract nano-isotopic and nano-structural data, respectively. Combining these techniques opens the era of nanogeochronology and allows to refine the geological history of the rock.

Laurent, A.T., Seydoux-Guillaume, A.-M., Duchene, S., Bingen, B., Bosse, V., and Datas, L. (2016) Sulphate incorporation in monazite lattice and dating the cycle of sulphur in metamorphic belts. *Contributions to Mineralogy and Petrology*, 171(11), 94.