



U-Pb ages of rare rutile inclusions in diamond indicate entrapment coeval with kimberlite formation

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The timing of diamond crystallization is generally inferred from radiometric dating of individual or multiple mineral inclusions in diamond, where ages are derived from isochrons or isotopic evolution models. Resulting ages often significantly predate the emplacement ages of host kimberlites, but age information from both approaches can be ambiguous due to long-lived mantle heterogeneities where mixing can mimic isochrons or in-situ aging. Direct dating of accessory mineral inclusions in diamonds, by contrast, has been rarely attempted because of the scarcity of such inclusions, requiring careful high-grading of large amounts of diamond concentrates. Here, we report secondary ionization mass spectrometry (SIMS) U-Pb ages from a suite of rare rutile inclusions which were extracted from diamond with eclogitic paragenesis from the Mir kimberlite pipe, Siberia, and screened for the highest U abundances (1.6-21 $\mu\text{g/g}$ U). Rutile inclusions with diamond crystal-shapes and petrographically identified as completely enclosed in diamond prior to cracking and extraction yielded a U-Pb concordia age of 375.5 ± 7.0 Ma (error 95% confidence; mean square of weighted deviates MSWD = 0.74; number of spots $n = 19$). No intra- or inter-inclusion age heterogeneity was detected. This age is nearly coeval with, but significantly (at the 95% confidence level) older than combined U-Pb rutile ages for rutile intergrown with polycrystalline fibrous diamond (362.9 ± 9.5 Ma; MSWD = 0.96; $n = 7$) and rutile from the eclogitic xenolith matrix (369 ± 16 Ma; MSWD = 0.30; $n = 5$). Rutile ages are also consistent with published ages for kimberlite emplacement (ca. 360 Ma), but much younger than Re-Os sulfide isochron ages between ca. 913 Ma and 2.1 Ga for inclusions in diamond from the same location. Diffusive Pb-loss from rutile inclusions over ca. 2 Ga, however, would only be possible if Pb-diffusion in the diamond host was significantly faster than experimentally determined Pb-diffusion in zircon, and Pb partitioning from rutile into diamond was near or greater unity. Alternatively, if Pb is immobile in diamond, ancient rutile ages could have been reset due to processes other than volume diffusion through diamond (e.g., due to fluid migration along cracks). However, in this scenario it remains difficult to explain why all rutile inclusions dated here would have been located along cracks, whereas sulfide inclusions investigated in other studies were not. It therefore is reasonably assumed that rutile inclusions were entrapped at the time of diamond formation, and that this event is recorded by the U-Pb rutile ages. Trace element heterogeneity in rutile suggests that they originated in chemically different environments, and thus likely predate diamond formation, but Pb accumulation only started after entrapment in diamond (for inclusions) or eruptive quenching (for intergrown or matrix rutile). This requires that at least some diamond from the Mir pipe formed briefly (within the ca. 20 Ma age difference indicated by U-Pb rutile ages for inclusions and intergrown/matrix rutile) before kimberlite eruption, supporting models that link diamond formation with carbon-rich precursors of kimberlite magmas.