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Synchrotron Mössbauer Source technique for in situ measurement of iron-bearing inclusions in natural diamonds

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Natural diamonds containing silicate, oxide and sulfide inclusions are a popular focus of investigation as they uniquely provide a window into the conditions of the Earth's interior at extreme depths. Recent discoveries based on investigations of deep diamonds have considerably improved our knowledge of the Earth's deep carbon and water cycles and the oxygen fugacity of the Earth's interior. Super deep diamonds are those that are believed to have formed at depths of at least 300 km and some evidence suggests depths of at least 800 km. A common inclusion in these diamonds is ferropericlase, (Mg,Fe2+)O. Ferropericlase is the second most abundant mineral in the lower mantle, constituting up to about 20 mol% of its volume. The Fe3+/Fetot of ferropericlase is a strong function of oxygen fugacity, and provides a measure of the most recent redox conditions under which it equilibrated. Conventional Mössbauer spectroscopy using a 57Co point source has been used in the past decades to study the Fe3+/Fetot content in inclusions still trapped in their diamond's host, however its limitations are the low spatial resolution (not below $\sim$100 μm2) and the long acquisition time. The Flank method was also proposed, it is fast, it has high spatial resolution (down to $\sim$20 μm2) but it measures the bulk value of Fe3+/Fetot since it cannot distinguish between different phases. An ideal method to measure Fe3+/Fetot values of ferropericlase would combine (1) the advantage of Mössbauer spectroscopy to distinguish Fe3+ in different phases and measure inclusions while still in the diamond, with (2) the advantage of the Flank method to conduct rapid measurements with high spatial resolution. The only method that offers the possibility to satisfy all these requirements is the Synchrotron Mössbauer Source (SMS). We used the SMS for the first time, to study the iron content and iron distribution in ferropericlase inclusion still contained within its diamond host from Juina (Brazil). This definitive non-destructive technique with extremely high spatial resolution ($\sim$15 μm2) enabled measurements in multiple regions of the 150 $\times$ 150 μm2 inclusion to be sampled and showed that while Fe3+/Fetot values in ferropericlase were below the detection limit (0.02) overall, there was a magnetic component whose abundance varied systematically across the inclusion. Hyperfine parameters of the magnetic component are consistent with magnesioferrite, and the absence of superparamagnetism allows the minimum particle size to be estimated as $\sim$30 nm. Bulk Fe3+/Fetot values are similar to those reported for other ferropericlase inclusions from Juina. Their variation across the inclusion can provide constraints on its history, and ultimate on the deep carbon processes behind diamonds formation and their exhumation from the transition zone and shallow lower mantle regions.