Quantitative chemical analysis of carbon and oxygen in molten Fe-rich alloy by analytical transmission electron microscopy.

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Carbon dissolved in metal is likely to strongly affect metal-silicate partitioning of siderophile elements, such as Co, Ni, V, Cr, Si, and O, between molten Fe-rich alloy and silicate melt in a laser-heated diamond anvil cell (LHDAC). The source of such carbon is chemical reaction between the sample and the diamond anvils. In order to determine reliable metal-silicate partition coefficients, corrections must be made based on the concentration of carbon in the metal. Unfortunately carbon is extremely difficult to analyse quantitatively using transmission electron microscopy (TEM), due to various sources of hydrocarbon contamination during sample preparation and in the TEM. The analysis of oxygen contents is also important for evaluating the metal-silicate partitioning of this element.

Here we report a procedure to evaluate carbon and oxygen contents in focused ion beam (FIB) prepared TEM lamellae from LHDAC samples. The FIB lamellae were cleaned by a plasma cleaning system prior to TEM analysis. We calibrated ratios of partial cross-sections of carbon and oxygen against iron in electron energy-loss spectroscopy (EELS) analysis in a TEM operating at 200 kV under an oil-free vacuum system. The advantage is to enable the quantitative chemical analysis of carbon and oxygen dissolved in the molten Fe-rich alloy, combined with energy dispersive X-ray spectroscopy (EDXS) of the other heavier elements.

Preliminary results indicate 3-6 at% carbon and 16-31 at% oxygen in Fe-rich alloys recovered from 43-100 GPa and 3700-5500 K as evaluated by EELS, together with metal-silicate partitioning by EDXS. We are planning to cross-check the obtained carbon contents with the other established analytical techniques (e.g., Nano-SIMS).