SiEN$_2$*A*H: Custom *in situ* sample environment for fast and high-temperature sintering in mixed nitrogen/oxygen atmospheres using 2D PXRD

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SiEN$_2$*A*H is a custom sample environment that enables the study of materials using *in situ* 2D powder X-ray diffraction (PXRD) at heating rates up to 100 °C/s and temperatures up to 1400 °C in mixed nitrogen/oxygen atmospheres, see Figure 1 a). The high temperatures in air and mixed nitrogen/oxygen atmospheres are enabled by the silicon nitride heating elements, which have been configured to create a 15 × 15 × 6 mm^3 concentrated heating zone, see Figure 1 b). The oven was primarily designed to calcine and sinter powder compacted into pellets with a diameter of up to 10 mm. The acrylic environment chamber with polyethyleneimine (PEI) windows allows for changing the sintering atmosphere by flushing the chamber with the desired gas and collecting 2D PXRD images as a function of temperature. An electrochemical oxygen sensor is used to measure the oxygen content. The custom electronics allow for monitoring the temperature at three different positions in the heater: the hottest point of the heater used for PID temperature control, the side of the sample, and in the sample holder using Type-S thermocouples. The electronics also perform the PID temperature control, vertical motion control of the heater for quenching of the sample and sample rotation.

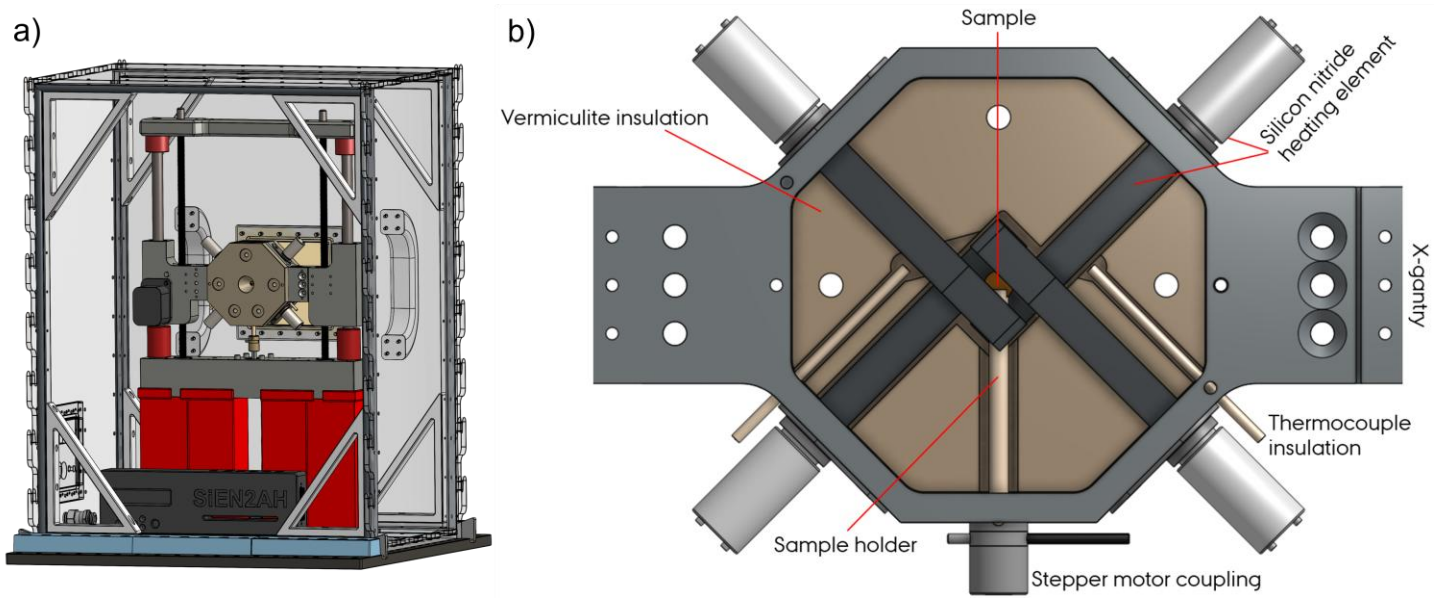


Figure 1. *a) CAD drawing of SiEN$_2$AH along with its environment chamber. b) Close-up of heater design.*

The capabilities of SiEN$_2$*A*H will be demonstrated through temperature calibrations of both sintered alumina and pressed powder of alumina (α-Al_2O_3). The temperature calibrations showed good agreement between the measured temperatures and the sample temperature. Fast phase formation of various hexaferrite types (M, W and X) [1] will also be demonstrated with formation times on the order of tens of seconds. The sintering atmosphere's effect on the formation of the hexaferrites will also be demonstrated, especially for the X-type hexaferrite for which the formation temperature could be lowered up to 80 °C using an oxygen-lean atmosphere. This is an important result, opening the possibilities of using X-type hexaferrites as a permanent magnetic material and improving the performance of the state-of-the-art permanent magnetic material, M-type hexaferrite.

[1] R. C. Pullar, Prog. Mater. Sci. (2012). 7, 1191–1334.