Masterful Sample Grilling – Next Starts

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A comprehensive understanding of the relationship between structure and properties is crucial for designing materials with specific characteristics for targeted applications. Single crystal X-ray diffraction stands out as one of the most effective analytical techniques to gain this insight. Modern scientific advancements have enabled experiments under non-ambient conditions, typically involving increased pressure and decreased temperature.

However, conducting measurements at very high temperatures is less common due to the scarcity of suitable heating devices that are both easy to install and safe to operate. Recently, the integration of a Hot Air Gas Blower heater into the D8 VENTURE has facilitated convenient and precise measurement of single crystal X-ray diffraction data at temperatures up to 1000 °C, significantly expanding the experimental capabilities available to researchers.

Vanadinite (Pb5(VO4)3Cl), an apatite mineral, is the primary industrial source of vanadium and, to a lesser extent, lead. It has been documented that vanadinite undergoes a phase transition at 23.1 GPa and 600 K, whereas its synthetic analog Pb5(VO4)3I, where chlorine is replaced by iodine, decomposes at 540 K. Studies on various phosphate and vanadate analogs have revealed phase transitions to monoclinic, pseudohexagonal phases.

We present the high-temperature measurement of a vanadinite (Pb5(VO4)3Cl) crystal mounted on the tip of a quartz capillary and demonstrate how the integration of advanced heating devices like the Hot Air Gas Blower heater into modern X-ray diffraction equipment significantly enhances the ability to study materials under extreme conditions. This advancement not only broadens the scope of experimental research but also provides deeper insights into the behavior of materials, paving the way for the development of new materials with tailored properties for various industrial applications.

[1] Y. Liu et al., Minerals 2021, 11, 121.

[2] Gatta et al., Physics and Chemistry of Minerals 2009, 36, 311–317.

[3] S. Redfern et al., Mineralogical Magazine (2012) 76 (4): 997–1003.