Temperature Calibration for High-Temperature X-Ray Diffraction

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The accuracy of the high temperature diffraction technique depends on thorough calibration of the instrument, both in terms of the diffraction pattern and the temperature response. The lack of standard reference materials specific to high-temperature X-ray diffraction makes calibration of the temperature response of the heating system a complex procedure.

Two complementary methods can be used for temperature calibration. One is through the use of materials with well defined thermal expansion, such as Al₂O₃, CaO and MgO. The second is through the use of materials with a well-defined phase transformation or melting temperature. Such a method is used in the calibration of thermal analysis instrumentation, and certified NIST standard reference materials are available for this purpose.

The performance of approximately 20 of thermal expansion and phase transformation calibration materials was investigated. The study included NIST-certified and non-certified materials, and the advantages and disadvantages of each method are presented. Experimental considerations such as atmosphere and material reversibility are described in detail and recommendations for a comprehensive calibration method will be provided.