

Update of the HTSI method: application to mechanical characterization of CaF₂ up to 800°C

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The recent development of the High-Temperature Scanning Indentation (HTSI) method [1] allows for the characterization of material mechanical properties quasi-continuously over a large temperature range in 1-day experiments. However, such a technique employs a nanoindentation cycle with a constant maximum load applied regardless of the temperature. Thus, for materials exhibiting an Indentation Size Effect (ISE), the variations in hardness with temperature can stem from both temperature and ISE. It becomes more challenging to differentiate between these two effects and analyze their impact on the mechanical properties.

To address this issue, a new 1-second indentation cycle has been implemented. A 0.5-second half-sine function is utilized during loading, followed by a 0.1 to 1-second creep segment and the 3-step unloading function used previously. To control the maximum achieved depth across temperatures, the maximum applied load is adjusted experimentally between each indentation, using previous indentation tests and results. This approach allows for the determination of hardness, Young's modulus, and creep properties of a material at a given maximum depth over a wide temperature range.

This methodology has been applied to CaF₂ single-crystal from room temperature (RT) to 800°C. The implemented cycle enables the characterization of this material at 1000nm depth over the entire temperature range. Comparison with tests performed using the previous indentation cycle highlighted the impact of the ISE in temperature. The obtained results were compared to conventional indentation results.

- [1] G. Tiphéne *et al.*, « High-Temperature Scanning Indentation: A new method to investigate in situ metallurgical evolution along temperature ramps », *J. Mater. Res.*, vol. 36, n° 12, p. 2383-2396, juin 2021, doi: 10.1557/s43578-021-00107-7.