

Synthesis and Characterization of Dolomite Reference Materials for In Situ Carbon and Magnesium Isotopic Microanalysis

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In situ microanalysis of carbon and magnesium isotopes in dolomite offers critical insights into various fields of earth sciences. However, matrix effects arising from compositional heterogeneity in natural dolomites pose significant analytical challenges, particularly for high-precision techniques such as laser ablation multi-collector inductively coupled plasma mass spectrometry (LA-MC-ICP-MS). This study presents a novel pressureless sintering protocol to synthesize isotopically homogeneous dolomite reference materials. The crux of this method involves precise optimization of precursor particle size and sintering temperature to achieve micrometer-scale homogeneity in sintered dolomite powder pellets (SDPPs).

Experimental results demonstrate that reducing the d_{90} particle size markedly enhances isotopic uniformity. SDPPs with $d_{90} = 3.17 \mu\text{m}$ exhibit superior laser ablation repeatability for $\delta^{26}\text{Mg}$ (0.11‰, 2SD) and $\delta^{25}\text{Mg}$ (0.09‰, 2SD), representing a 2–4-fold improvement over SDPPs with $d_{90} = 10.37 \mu\text{m}$. Furthermore, sintering temperature critically influences pellet performance: thermal treatment at 400°C (below dolomite decomposition threshold) elevates Vickers hardness to 41.58 kgf mm⁻² by enhancing interparticle cohesion. These optimized SDPPs achieve exceptional in $\delta^{13}\text{C}$, $\delta^{26}\text{Mg}$, and $\delta^{25}\text{Mg}$ homogeneity, yielding 2SD values of 0.37‰, 0.11‰, and 0.06‰ (n=15), respectively.

To verify the reliability of these SDPPs, using natural dolomite as standard, LA-MC-ICP-MS was used to analyze the C and Mg isotopes of SDPPs. The results showed that the isotopic fractionation between SDPPs and natural dolomites was almost negligible. This finding further confirms the feasibility and reliability of the proposed protocol for preparing dolomite isotopic reference materials via pressureless sintering. In earth science research, these high-quality SDPPs will serve as a powerful tool for isotopic microanalysis.