Geophysical Research Abstracts
Vol. 19, EGU2017-1944, 2017
EGU General Assembly 2017

Laser ablation ICP-MS analysis on nano-powder pellets and applications to granite bulk rock analysis

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Granites are a ubiquitous component of the continental crust and knowing their precise trace element signatures is essential in understanding the origins and evolution of the continental crust. ICP-MS bulk analysis of granite is generally conducted on solution after acid-digestion. However this technique has several deficiencies related to the difficulty of completely dissolving accessary minerals such as zircon and the instability/adsorption of high valence trace elements (Nb, Ta et al.) in acid solutions. The development of a nano-powder pellet technique by using wet milling procedure, and its combination with laser ablation ICP-MS has been proposed to overcome these problems. In this study, we produced nano-powders from a series of granite rock standards by wet milling in agate using a high power planetary ball mill instrument. The procedure was tested and optimized by modifying parameters (ball to powder ratio, water to powder ratio, milling power etc.). Characterization of nano-powders was conducted by various techniques including electron microprobe (EMP), secondary electron imaging, polarizing microscope, and laser particle size analyzer (LPSA) and laser scanning confocal microscope (LSCM). Particle sizes range from a few nm to $\sim$5 μm with a small secondary mode at around 10 to 20 μm that probably represent particle aggregates rather than remaining crystal grains after milling. Pellets of 5 mm in diameter were pressed into molds of cellulose at 1.75 *10̂3 N/cm2. Surface roughness of the pellets was measured by LSCM and gave a Ra of 0.494 μm, which is an order higher than the surface of polished ATGH-G reference glass surface (Ra: 0.048 μm), but sufficient for laser ablation. Sources of contamination either from abrading agate balls or from ultrapure water were evaluated and quantified. The homogeneity of powder pellets down to less than 5 μm size was documented based on EMPA element mapping and statistical analyses of LA-ICP-MS in discrete spot and line scanning analytical mode. We report data from major to trace element (to < 0.1 ppm) of currently available international granite reference materials (JG-2, JG-3, GWB07103, GEB07111, GSP-2 and G-3) to evaluate analytical precision and accuracy of LA-ICP-MS measurements. Our results illustrate the potential of this method for high precision analysis of trace elements and e.g. Zr/Hf and Nb/Ta ratios in granites.