Maximising precision and accuracy in laser quadrupole ICPMS U-Pb geochronology

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Laser mass spectrometry is widely used to determine the age of rocks throughout the earth science. However, analytical problems related to ablation cells, standards, mass spectrometers and calculations have meant that it is difficult to fully characterise precision and accuracy of the technique.

Experiments performed at the University of Tasmania over the last 10 years have shown that the following parameters affect the precision and accuracy of measurements: 1) homogeneity of the signal in the ablation cell, 2) type, number and position of standards analysed within the analytical sequence 3) accuracy dead time corrections 4) amount of instrumental drift, 5) contamination during cleaning and polishing 6) calculation method used 7) tuning parameters and signal intensity 8) harmonic interference between the laser and the mass spectrometer (spectral skew).

The contribution of each of these parameters to the uncertainty of the final measurements is complex and changes with age and uranium concentration of the crystals. For example young and low U zircons (those which have accumulated <0.2 ppm radiogenic Pb) must be carefully cleaned and calculated using the 'ratio of the mean' rather than the 'mean of the ratio' method but are relatively insensitive to the position and homogeneity of the standards, dead time corrections, harmonic interferences and instrumental drift compared to older high U zircons.

Mechanical instabilities induced by sulfate adsorption

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We present experimental data and analytical calculations which demonstrate the effect of negative surface charge caused by sulfate adsorption in high porosity chalks. Rock mechanical tests on three high-porosity outcrop chalks flooded with Na₂SO₄ at 130 ^oC show a significant decrease in the yield strength and in the bulk modulus compared to that of NaCl flooding. At 50 °C, however, no significant difference in the mechanical strength between cores exposed to Na2SO4 and NaCl is observed. Relative to that of the distilled water flooding (i.e. no sulfate present) a decreasing strength of 20-40% in the yield and 10-70% in the bulk modulus are correlated well with the increasing sulfate concentration in the pore water at higher temperature (130 °C). In addition standard creep tests at constant stress level of 10.7 and 18.9 MPa show similarly high creep rate along the creep stages. We measured sulfate adsorption of 0.2 $\mu mol/m^2$ and 0.5-1 µmol/m² at 50 °C and 130 °C, respectively, while dissolution was found to be low (below 0.4 mM Ca2+ produced after 14 PV's flooded). The sulfate adsorption process is interpreted by using surface complexation model together with Gouy-Chapman theory to describe the electrical double layer. The analytical calculations give fairly good agreement with the measured sulfate adsorption and comparable with ζ -potential measurement data respectively. The reduced mechanical strength is related to the sulfate adsorption which leads to a negative (pH dependent) surface charge. We suggest that the interaction between charged surfaces specifically in the weak overlaps of electrical double layer gives rise to the total disjoining pressure, which acts as normal forces in the grains vicinity, and it counteracts the shear forces during the failure mechanism. Increasing magnitude of the disjoining pressure when chalk cores are exposed to different Na₂SO₄ concentration at 130 °C correlate well with the reduced strength from the mechanical test results. The correlation remarkably reproduces the same trend as observed in the yield strength and bulk modulus as a function of Na2SO4 concentrations.

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