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Pressure solution indentation experiments on K-alum single crystals

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Pressure solution indentation experiments were carried out on single crystals of an elastic-brittle salt (potassium- or K-alum) having a high solubility and fast dissolution/growth rate. The experiments were carried out to study why pressure solution indentation in this brittle material is several orders of magnitude faster than in rock salt, whereas solubility and dissolution/growth rate are comparable. The crystals were solution-grown and cut with a wire saw in rectangular pieces of 5 x 5 x 3 mm. They were mounted in an Ertalon™ vessel containing saturated K-alum solution that could not escape by evaporation. The crystals were loaded perpendicular to a polished (111) face, parallel to the short direction of the sample, using stainless steel pistons of 0.50, 0.75, 1.00 and 1.20 mm diameter. In the first series of experiments, pistons were loaded with a dead weight corresponding to a stress of 15 MPa, i.e. well below the brittle strength of alum. Experiments were carried out at room temperature (20 to 21°C) and atmospheric pressure. Displacement of the piston was monitored as a function of time with a dial gauge. Experiments typically lasted for 1 to 2.5 days. No measurable indentation occured in experiments with no solution present even after weeks under stress. In experiments *with* solution, indentation started-off at relatively low rate (5 to 10 µm/day) for about the first 3 to 6 hours, then increased up to 100 to 800 µm/day, depending on piston diameter and other experimental conditions. Indentation rate was very constant during this stage of the experiment and approximately inversed proportional to the square of the piston diameter. Indentation pits were 1 to 2 mm deep, with a diameter of about the piston diameter. The pits had partly negative crystal structure, mostly along their rim. Near the centre of the pits a small platform was commonly present that must have supported the piston. It was generally about 100 to 500 µm in diameter and 20 to 70 µm high and full of tiny microcracks. Experiments were repeated under the microscope, in-situ, between glass slides. The most remarkable information gained was the development of tiny gas bubbles at the contact zone between the piston and the crystal. It was inferred that this was hydrogen gas, produced during oxidation of the Fe-bearing "stainless steel" pistons. Oxidation could take place because the K-alum solution is weakly acid due to partial hydrolysis of Al^{3+} ions to $Al(OH)_n^{m+}$ (n=1-3, m=2-0). Continuous consumption of H^+ used up in the oxidation reaction caused a continuous hydrolysis of Al^{3+} and consequently, a continuous free face dissolution of K-alum around the piston (irrespective of stress). Indentation of the piston took place by undercutting (due to undersaturation caused by the redox-reaction) and subsequent brittle collapse of the remaining platform. Experiments were repeated with pistons of magnesia and of Cr-Steel that could not oxidise. In both these cases indentation rate remained very low (10-20 µm/day for weeks).