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The effects of stress concentrations on reaction progress: an example from experimental growth of magnesio-aluminate spinel at corundum – periclase interfaces under uniaxial load

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The study aims to understand the reaction progress and chemical, microstructural and textural evolution of magnesio-aluminate spinel reaction rims formed at varying experimental settings (load, temperature and experiment duration). The spinel rims were grown at the contacts between periclase and corundum at temperatures of 1250°C to 1350°C and dry atmosphere, maintained by a constant argon gas flow, under uniaxial load of 0.026 and 0.26 kN per 9 mm2 of initial contact area. Single crystals of periclase with [100] and of corundum with [0001] perpendicular to the polished reaction interface as well as polycrystalline corundum were used as starting materials. Two loading procedures, immediate application of the load before heating and loading after the desired temperature had been reached, were used.

An important byproduct of our experiments stemmed from the immediate application of the load, which led to deformation twinning and fracturing of corundum. This internal deformation of corundum disturbed the reaction interface and introduced loci of concentrated stress due to opening of void spaces in between the reactant crystals. Whenever cracks formed in the initial stages of an experiment, the void space opened immediately and no spinel formed along these interface segments. In the case of deformation twinning, the decreased rim thickness indicates later opening of void spaces. This is because next to twins, the reaction interface is characterized by tight physical contact on the one side and less tight contact on the other side of the twin individual. The tight contacts are characterized by enhanced reaction progress which together with the overall positive volume change of the reaction and limits on plasticity of the studied phases led to the opening of void spaces at places characterized by less tight contacts. The thickness variations are less pronounced in our high load (0.26 kN) experiments where periclase behaves plastically and to some extent reduces the opening void space. In contrast, our low load (0.026 kN) experiments with limited plasticity of periclase show large lateral variations in the rim thickness.

The spinel rims increase their thickness with increasing temperature and time and they show chemical zoning in the direction perpendicular to the rim manifested by deviation from stochiometry and aluminium excess towards the contact with corundum. Spinel shows strong topotactic relations to the reactant phases including full topotaxy between spinel and periclase, partial topotaxy with (111)spi‖(0001)cor and {101}spi‖{10-10}cor, and axiotaxy with (111)spi‖(0001)cor between spinel and corundum. The microstructure in the rim is dominated by a layer grown at the expense of periclase and showing topotaxy to periclase and a layer grown at the expense of corundum and showing spinel twins related by (111) twin law with topotaxy to corundum. In the case of disturbed reaction interface the latter layer is dominated by axiotaxy of spinel to corundum. Oriented nucleation and selective growth were identified as the main mechanism of texture formation. With respect to stress variations, it is shown that the stress concentrations and tight physical contacts across the reaction interface enhanced the topotactic relations of spinel to periclase and corundum.