Space charge characterisation by EDS microanalysis in spinel MgAl2O4

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Abstract

It is well known that spinel shows a grain boundary sliding deformation accommodated by diffusion during creep at high temperature. The observed interface reaction controlling diffusion at low stress (under 60 MPa) can be explained by a space charge layer at grain boundary in ionic ceramics. The non stoechiometric area due to ionic defect should create an electrostatic potential between the surface and the bulk of the grain. This surface dipole region suppose a free energy excess used to form cation and anion vacancies[1, 2]. The purpose of this presentation is to study the grain boundary region stoechiometry in order to check the validity of this theory. Fine grained spinel (average grain size under micron) showing an interface reaction at low stress is investigated by TEM microanalysis. Stoechiometry variations are observed from the grain boundary to the bulk. For example the Al/Mg ratio increases from 2.1 to 2.35. X-rays quantification allows us to reveal and to characterise the space charge layer around the interfaces. The boundaries are negatively charged due to the Mg vacancies. However the microanalysis spot size limits the spatial resolution and consequently avoids us to know exactly the stoechiometry in the boundaries. Then, it is necessary to take into account the positive charge near the boundary to describe the stoechiometry change.

[1] K.L. Kliewer et J.S. Koehler, Physical Review Vol 140 n4A (1965) 1226 [2] J. Jmnik et R. Raj, J. Am. Ceram. Soc 79 [5](1996) 193