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A New Form of $MgTa_2O_6$ obtained by the Molten Salt Method

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$MgTa_2O_6$ is an important dielectric material and has uses in the microwave frequency range. $MgTa_2O_6$ is reported to crystallize in the trirutile structure (tetragonal, space group $P4_2/mmm$) with lattice parameters of a = 4.695 and c = 9.147 Å. Here the structure is made up of strings of edge-shared octahedra extending along the c - direction and these strings are linked together by sharing corners . The edge-sharing occurs at opposite edges in each octahedron leading to linear octahedral strings in the trirutile structure. The above structure may be obtained by solid state reactions of $MgCO_3$ and Ta_2O_5 at high temperatures of 1200 ^oC to 1400 ^oC. We have been interested to prepare oxides by alternate low temperature routes such as by the molten salt or flux method [1,2]. In this report we discuss our studies on the synthesis of a new form of magnesium tantalate by using a NaCl - KCl flux.

Our starting reagents are $Mg(NO_3)_2.6H_2O$ and Ta_2O_5 to which we add a 1 : 1 mixture of NaCl - KCl flux. After proper mixing and grinding the mixture is heated in a furnace at 900C for 6h. the product is washed with distilled water several times and checked for absence of Cl^- ions in the filtrate. This is to make sure that all the flux is washed off. The powder is then dried at 120C.
Powder x-ray diffraction studies suggest an altogether different phase as compared to the reported $MgTa_2O_6$, and peak overlap is also possible. To find out the probable lattice parameters we have carried out electron microscopic investigations using a combination of HRTEM and electron diffraction procedures to determine the lattice parameters. These complex multi-crystalline powder compounds were found to be very beam sensitive and low electron dose conditions were therefore employed [3,4]. Using electron accelerating voltages of 200kV in a FEI CM30 HRTEM, we have succeeded in high resolution imaging and electron diffraction measurements of the compound. Crystals were tilted to different zone axes, including [12-1] projections. High precision composition analysis was carried out simultaneously using energy dispersive X-ray spectroscopy (EDX) in the HRTEM. Initial electron diffraction data have shown a dominant orthorhombic phase with approximate lattice parameters of a~ 15.4 Å, b~ 13.4 Å, and c ~ 12.2 Å,. Electron diffraction pattern, for example, in [12-1] and the corresponding lattice image are shown in FIG.1 and FIG. 2, respectively. EDX composition analysis of the compound is shown in FIG.3. Some minor phases are also present [FIG.4]. We have satisfactorily indexed the powder x-ray diffraction pattern on the basis of the above orthorhombic cell and all the lines could be indexed. The refined lattice parameters of the new structure obtained are, a = 15.35(1) Å, b = 13.33(1) Å, and c = 12.057(9) Å.

References:
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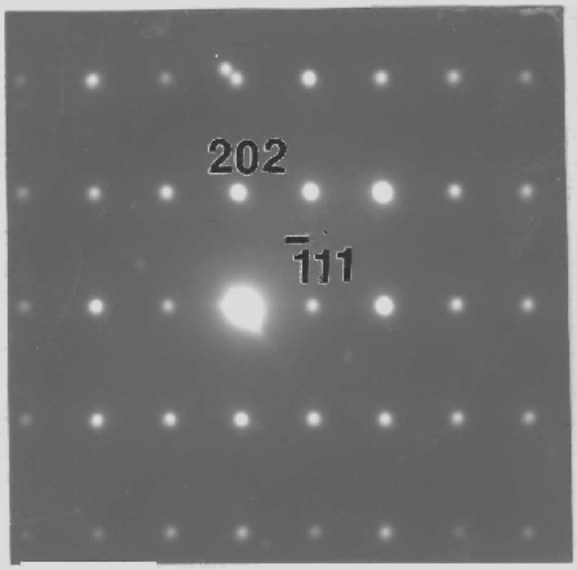

FIG.1

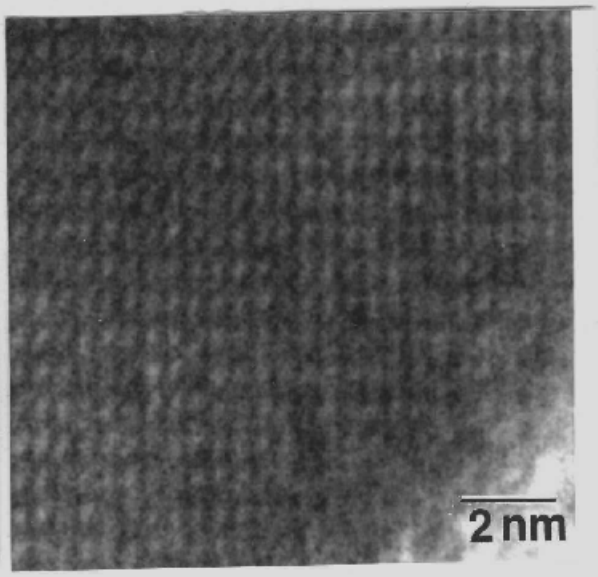

FIG.2

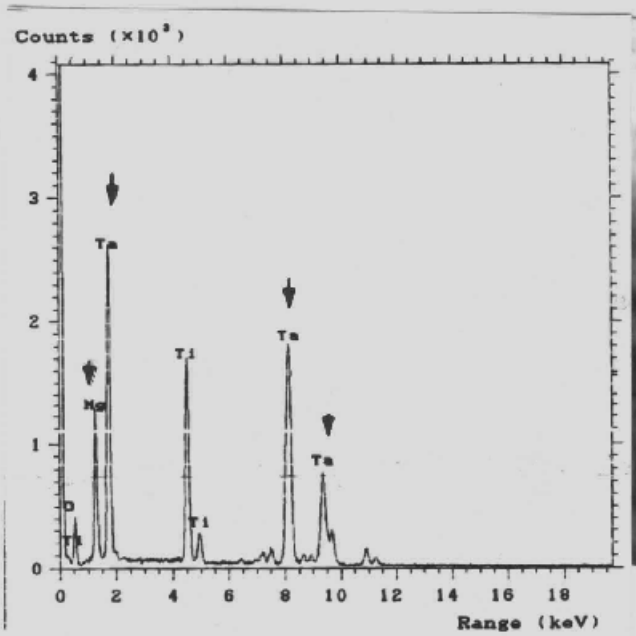

FIG.3

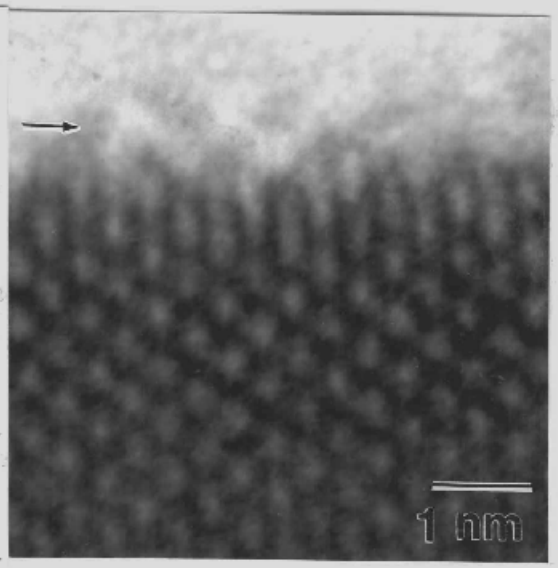

FIG.4

FIG. 1 Electron diffraction pattern of complex $MgTa_2O6$ along [12-1] zone axis
FIG. 2 HRTEM of the complex, beam sensitive oxide with well ordered crystals.
FIG.3 X-ray composition spectrum of the oxide supported on titanium grid.
FIG.4 Secondary phase observed in the Mg-Ta-O system, with surface layers.

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