STRUCTURAL CHARACTERIZATION OF Me-MgTiO₃ SOLID SOLUTION (Me = Zn, Mn) PRODUCED BY POWDERED MIXING

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ABSTRACT

Synthesis of Me-MgTiO₃ (Me = Zn or Mn, abbreviated as MeMT) solid solution has been done carried out with a solid state reaction method. The starting materials were MgO (Merck) and TiO₂ (Merck) powders. The additives were ZnO or MnO₂. Since MgO reacts easily with H₂O, it was calcinated at 600°C for half an hour before mixing it. Slight MgO-excessive equimolar (1.013:1) mixing of the powders was performed using Planetary Ball Mill Pulverisette 5 (Fritsch) instrument with variation of milling time of 10h, 14h, and 18h with 1h break after each 1h cycle of milling followed by calcination at 1100°C for 1 h. The samples were denoted as MT10, MT14 and MT18, respectively. X-ray diffraction data analyses showed that the samples contained only MgTiO₃ and MgTi₂O₅. Further analysis using Rietveld method showed that the MT10 sample contained the highest MgTiO₃ weight fraction, i.e. 84.2%, although it decreases with increasing milling time. Based on this result, the MeMT solid solution samples were prepared using 10h milling time. The designated samples were ZnₓMg₁₋ₓTiO₃ (ZMT) and MgMnₓTi₁₋ₓO₃ (MMT) with x = 0.1; 0.2; 0.3; 0.4 and 0.5. The calcinated samples were in powder form and characterised using XRD. Phase identification showed that there were two phases in ZMT sample, those are MgTiO₃ and MgTi₂O₅. Further analysis using Rietveld method were performed to reveal the phase composition and lattice parameter values. The formation of solid solution in the ZMT samples were evident from the absence of Zn, ZnO or Zn-O-Ti phases and from the systematic change in lattice parameter values of MgTiO₃ as well as support from SEM-EDX observation results. Interestingly, MMT samples contained MgTiO₃, MgO and Mg₂TiO₄. The formation of MMT solid solutions were also evident in a similar way to the ZMT samples not only occurs on the MgTiO₃ but also on the Mg₂TiO₄.

Key Word : ZnₓMg₁₋ₓTiO₃, MgMnₓTi₁₋ₓO₃, solid solution, structural characterization, solid-state reaction