Fabrication of Ca₁₂Al₁₄O₃₃ mayenite structure by spark plasma sintering

In this work, the Ca₁₂Al₁₄O₃₃ (C12A7) compounds were consolidated by spark plasma sintering (SPS) at 1100, 1200, 1300 and 1350 °C for 5 min, 50 MPa. The XRD result indicated the C12A7 phase in all specimens. The relative density increased from 87.1 to 99.4% with increasing sintering temperature. The electrical conductivity was also enhanced by increasing sintering temperature. The C12A7 specimens sintered at 1350 °C showed the highest electrical conductivity of 1.96 S m⁻¹ at 800 °C.

C12A7 compounds are a promising candidate for functional applications such as electrode [1], catalyst [2] and thermoelectric materials [3]. The unique structure consists of 12 nano-cages in a cubic unit cell while only 2 cages contain extra O^{2-} ion inside the cage. With the reduction process, the electron can be substituted for the O^{2-} ion, as a result, the carrier concentration for conduction could increase.

The aim of this study is to fabricate C12A7 by SPS under a reduction atmosphere. The SPS method possesses a fast heating rate with an applied pressure which could result in a high density product. The vacuum and the diffusion of carbon from graphite mold might induce the reduction atmosphere surrounding specimen. This could be the key factor to improve the electrical properties of C12A7.

Figure 1 shows XRD patterns of the sintered specimens and calcined powder. All sintered specimens were indicated as C12A7 (JCPDS no. 19-0629). The phase of C12A7 remained stable as increased temperature from 1100 to 1350 °C. However, after increase temperature to 1400 °C, the specimen was melted away.



Fig. 1. XRD patterns of C12A7 at various sintering temperature by SPS.

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The relative density of C12A7 increased from 87.1 to 96.6% with increasing temperature from 1100 to 1200 °C. The fully-densified C12A7 with the relative density of 99.4% was obtained at 1300–1350 °C.

Figure 2 shows the temperature dependence of electrical conductivity of C12A7 sintered at various temperature. The electrical conductivity of all specimens increased with the increasing temperature indicating the semiconductor behavior. The C12A7 specimens sintered at 1350 °C showed the highest electrical conductivity of 1.96 S m⁻¹ at 800 °C. The improvement in electrical conductivity was possible to be due to two reasons. First, carbon diffused into the structure of C12A7 specimens. Second, carbon reduced the extra O²⁻ in the cage structure.

In conclusion, the high density C12A7 with improving electrical conductivity was achieved by SPS at 1350 °C.



Fig. 2. Temperature dependence of electrical conductivity of C12A7.

References.

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