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METAL-LIKE THERMAL CONDUCTIVITY POSSESSED BY ATMOSPHERE ASSISTED SYNTHESIS OF SPARK PLASMA SINTERED MAYENITE ($Ca_{12}Al_{14}O_{33}$)

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Supporting Information

1. Experimental details

Reagent grade calcite ($CaCO_3$, Merck Pvt. Ltd - India) and γ alumina (SRL Pvt. Ltd – India) were used as reagents. The precursors were weighed in the molar ratio of 12:7 and mixed well-using agate mortar and pestle. The mixture was held in a tubular furnace at 1350 $^{\circ}$C for 24 h. The processing atmosphere was varied and the powders prepared in air, argon, and nitrogen atmospheres are referred to in the manuscript as M/air, M/Ar, and M/N_2 respectively.

Mayenite was prepared by the following overall reaction:

$$12CaCO_3 + 7Al_2O_3 \rightarrow Ca_{12}Al_{14}O_{33} + 12CO_2$$

The obtained mayenite powder particles were ground and sintered into pellets using spark plasma sintering at 1250 $^{\circ}$C at 50 MPa pressure maintaining a holding time of 10 min, 0 min, 20 min respectively for M/air, M/N_2, and M/Ar. The powder was filled in a graphite die and a graphite foil was placed between powder and inner die surface for easy removal of sintered pellets. The density of the sintered pellets was measured by the geometrical method due to the hygroscopic nature of mayenite.

The X-ray diffractometer working in Bragg-Brentano geometry was used with Cu K_α radiation ($K_{\alpha1}$ = 1.5406 Å) to obtain diffractograms of both powder and pellet samples for phase composition analysis. The data were recorded in the 2θ range of 15°- 60° with a step size of 0.02° and the patterns were analyzed using X'Pert HighScore Plus software.

The microstructures of the sintered samples were visualized using field emission scanning electron microscopy, SEM-EDX (FE-SEM, Hitachi S-4800) in secondary electron mode. The sintered pellets were polished according to standard ceramographic practice to a 0.5µm finish using diamond paste and thermally etched at 1150 °C for one hour in air, argon, and nitrogen for M/air, M/Ar, and M/N_2 respectively to image the microstructures.

The laser flash technique was used to measure the thermal conductivity of mayenite samples. The laser shots were taken at different temperatures between room temperature and 1000 °C and the corresponding thermal diffusivity values at respective temperatures were obtained for the three samples. The specific heat capacity (C_p) values from 50 K to 298 K were obtained from the work done by E.G. King [1] on the heat capacities and entropies for crystalline calcium and magnesium aluminates at low temperatures. The C_p at higher temperatures (i. e., 298 K to 1000 K) were calculated by differentiating the enthalpy equation as elaborated in the work by Bonnickson [2] given by the following equation;

$$H_T - H_{298.16} = 301.96\ T + (32.75 \times 10^{-3}\ T^2) + (55.30 \times 10^{5}\ T^{-1}) - 111{,}491\ \text{cal/mol} \quad (1)$$

The calculated C_p is given by equation (2)

$$C_p = (65.5 \times 10^{-3}\ T^2) - (55.30 \times 10^{5}\ T^{-2}) + 301.96\ \text{cal/deg-mol} \quad (2)$$

The C_p values for the temperatures at which thermal diffusivity values had been measured were calculated and k was obtained using the formula

$$k = \alpha \rho C_p \quad (3)$$

where k is the thermal conductivity (W/(m.K)), α is the thermal diffusivity (m^2/s), ρ is the density (kg/m^3) and C_p is the specific heat capacity (J/(kg·K)) of the sample.

References

1. E. G King, U.S Bur. Mines Tech. Paper, 1955, 59, 218-219.

2. K. R Bonnickson, U.S Bur. Mines Tech. Paper, 1955, 59, 220-221.

Table S1. Lattice parameters of powder samples of mayenite

Sample	Lattice parameter (Å)
M/air	11.984 ± 0.02
M/N_2	12.027 ± 0.02
M/Ar	12.033 ± 0.03

Table S2. Sintering conditions and physical properties of mayenite

	Sample	Holding time	Density(g/cc)
SPS at 1250°C	M/air	10	2.69
	M/N_2	0	2.64
	M/Ar	20	2.61