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NOTE

Preparation and Characterization of Cathode Material for Magnesium Cells

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A cathode materials for magnesium cells $MgTi_2O_5$ was synthesized *via* sol-gel method in this work. TG/DTA, XRD, SEM were used to characterize the reaction and product. The results showed that the product is well-crystallized and gets uniform particle size. Simulation cells were assembled employing the product as positive electrode. Charge/discharge cycle test show that the product has good reversibility. The discharge capacities for $MgTi_2O_5$ is 50.2 mAh g^{-1} after 50 cycles. The use of $MgTi_2O_5$ as active cathode materials for magnesium batteries in general shows great promise for secondary batteries.

Key Words: Magnesium battery, $MgTi_2O_5$, Positive electrode.

Following the achievements in the field of lithium-ion batteries, there have been more and more attempts over the years to develop rechargeable magnesium batteries, due to its natural abundance, relatively low price and higher expected safety[1-6]. One of the encountered difficulties is the low mobility of Mg^{2+} in the host lattice, since Mg^{2+} has high surface charge density. Consequently, fast Mg^{2+} transport in the intercalation host is required at ambient temperature.

Recently, it has been suggested that $Li_4Ti_5O_{12}$ can store large amounts of charge per unit mass as cathode materials for rechargeable lithium batteries *via* a highly reversible redox reaction. The similar ion sizes of monovalent lithium and divalent magnesium cations may lead, from a steric point of view, to an analogous insertion electrochemistry. Here, it is proposed that $MgTi_2O_5$ is feasible as potential cathode materials for magnesium batteries. In this work, we report the synthesis of $MgTi_2O_5$ samples by sol-gel method and investigated electrochemically as a magnesium host.

All the chemicals *viz*., $Mg(NO_3)_2{\cdot}6H_2O$, $Ti(OBu)_4$ and alcohol used in the experiment are of analytical grade.

General procedure: $MgTi_2O_5$ was synthesized *via* sol-gel method. First, $Mg(NO_3)_2{\cdot}6H_2O$ and $Ti(OBu)_4$ in a stoichiometric ratio were dissolved in alcohol with magnetic stirring. After a clear solution formed, while stirring for 2 h and then a gel formed in an air oven at 100 ºC. The gel was decomposed at 350 ºC in an air for 4 h and the obtained product was ground, pressed into pellets and sintered at 700 ºC or 24 h in an air.

Detection method: Thermogravimetry/differential thermal analysis thermal analysis was carried out on Shimadzu TA-60 with the heating temperature from 20-900 ºC at 20 ºC/min in N_2. XRD (X-ray Power Diffraction) patterns for all the samples were measured by Rigaku D/max-3B X-ray Diffractometer with CuKα radiation (λ = 0.15406 nm). The morphology of the samples was observed by Jeol JSM-5600LV scanning electron microscope.

Electrochemical Mg-ion intercalation performances of the samples were evaluated in Mg test cells. The cathode materials were prepared by mixing the samples with acetylene black and polytetrafluoroethylene (PTFE) with a weight ratio of 85:10:5 in ethanol to ensure homogeneity. After the ethanol was evaporated, the mixture was rolled into a sheet and the sheet was cut into circular strips of 8 mm in diameter. The strips were then dried at 100 ºC for 10 h. Magnesium metal was used as an anode. The electrolyte was composed of 0.25 M $Mg(AlBu_2Cl_2)_2$/THF solution and performed in a cylindrical glass cell. Test cells were assembled in an argon-filled dry glove box. The galvanostatic charge/discharge tests were performed with a Land CT2001 battery tester at 25 ºC.

TG/DTA analysis: Fig. 1 shows DTA curves of the gel. There are two endothermic peaks at 147.18 ºC, 442.07 ºC and one exothermic peak at 611.81 ºC in DTA curves. Thermogravimetry curve revealed there are three reaction stages. Firstly, the gel loses crystal water at 147 ºC. At the temperature range from 300-500 ºC, the gel further decomposes. at above 610 ºC, the gel produces a pure $MgTi_2O_5$, whose structure is characterized by X-ray diffraction the third stage was to be exothermic.

Powder X-ray diffraction: The XRD pattern of $MgTi_2O_5$ (Fig. 2) revealed that the crystal structure of the sample was pure $MgTi_2O_5$ crystal, which belonged to orthorhombic crystal system compared with the standard PDF cards (#35-0792). The lattice is base-centred orthorhombic lattice and the cell parameters: a = 0.9750 nm, b = 0.9980 nm, c = 0.3748 nm, interplanar distance: 0.3499 nm.

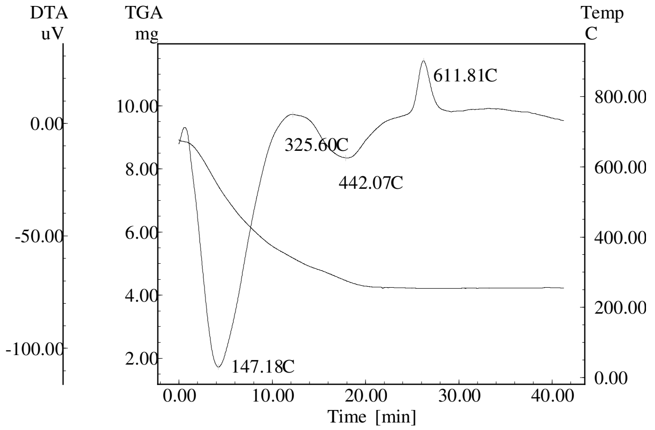

Fig. 1. TG/DTA curves of the gel

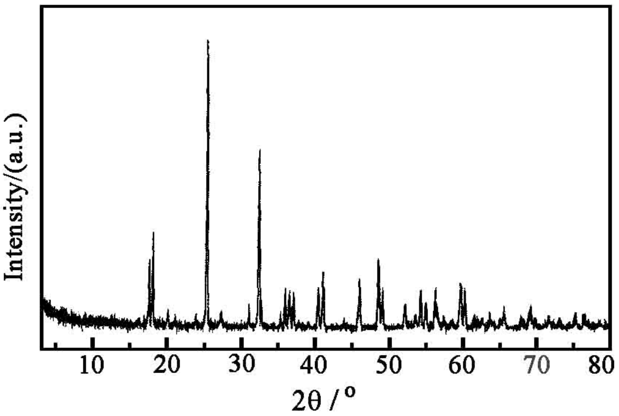

Fig. 2. XRD of $MgTi_2O_5$

SEM: Fig. 3 showed microstructure of the fracture surfaces of the specimen pellet heat-treated at 700 °C. The sample has a spherical morphology that is 5 µm in diameter and the small particles show good crystallinity and homogeneity.

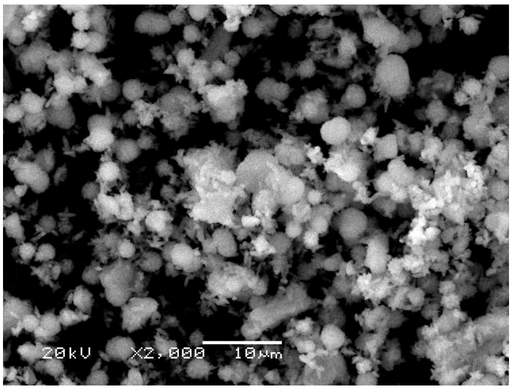

Fig. 3. SEM images of $MgTi_2O_5$

Electrochemistry: The initial galvanostatic discharge/charge curves for $MgTi_2O_5$ test cells are measured at a current density of 0.02 mA/cm^2 in the potential range of 0.5-2.0 V at 25 °C. Cell exhibits one charge plateaus around 1.5 V. The discharge capacities for $MgTi_2O_5$ is 50.2 mAh g^{-1} after 50 cycles.

Conclusion

A new cathode material $MgTi_2O_5$ was synthesized *via* sol-gel method. It was found to electrochemically absorb and desorb magnesium. The discharge capacities for $MgTi_2O_5$ is 50.2 mAh g^{-1} after 50 cycles. The use of $MgTi_2O_5$ as active cathode materials for magnesium batteries in general shows great promise for secondary batteries.

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