SUPPLEMENTAL INFORMATION

Novel, Electrolyte Solutions Comprising Fully Inorganic Salts with High Anodic Stability for Rechargeable Magnesium Batteries

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Synthesis of MACC Salts

In a typical preparation of an electrochemically active MACC solution such as 0.267 M Mg_2AlCl_7, one may undertake the following reaction:

$$2MgCl_2 + 1AlCl_3 \rightarrow Mg_2AlCl_7,$$

using ~0.508 g $MgCl_2$ powder (Sigma, 99.99%) and ~0.356 g $AlCl_3$ (Sigma, 99.999%) with 10.0 ml of tetrahydrofuran (THF, Novolyte). Subsequently stir and heat to ≥30.0 degrees Celsius for several hours after which solution may be returned to room temperature. The resulting solution is light yellow with no precipitation. Similar methods and formulations extend to other ethereal solvents.

Experimental

Voltammetry was performed on a PAR VersaStat 3 in a beaker cell utilizing Pt metal working electrodes and Mg (Goodfellow, 99.9%) metal as the counter and reference electrodes. Chronopotentiometry tests utilize a two electrode cell (Cu vs. Mg) and a current density of 0.5 mA/cm^2. 20% of an initial deposit of about 2.5 micron Mg was cycled 50 times at ambient conditions. Maccor Series 4000 was utilized as a current source.

Cell assembly was conducted inside an argon filled glove-box using custom made pouch cells. Magnesium metal (Goodfellow) serves as the negative electrode while the positive electrode consists of a composite (70% Mo_6S_8, 10% Timcal Super C65 Conductive Carbon black, 20% binder, Kynar HSV PVdF) cast from 1-Methyl 2-Pyrrolidinone (Sigma) slurry and dried at 120 degrees Celsius under dynamic vacuum (10 - 100 mTorr) overnight. The electrolyte comprises 0.25M MACC (3:2) in 1, 2 dimethoxyethane (DME, Novolyte). The Mo6S8 cathode was synthesized according to a reported recipe. [1] The cycling regime consisted of constant current discharge a rate of approximately C/15 to a cut-off voltage of 0.5 V followed by constant current charging at a rate of C/15 to 2 V for 7.5 cycles. Subsequently the rate was increased to C/2 for 15 cycles. Note: C/2 refers to a rate at which a full load or unload of the active material is accomplished in 2 h and providing 129 mAh/g. Current interrupts were applied for five minutes every two hours to monitor open circuit potential. Cycling was performed on an Arbin BT2000.

Scanning electron (SEM) micrographs were obtained on a JSM-6700F scanning electron microscope operating at an accelerating voltage of 10.0 kV. Powder samples were pressed into carbon tape for mounting on sample stubs; electrodes were mounted directly on the stubs. Energy dispersive X-ray spectroscopy (EDS) measurements were carried out using an Oxford Instruments X-Max 80 mm^2 silicon drift detector.

References

1. Lancry, E., Levi, E., Mitelman, a, Malovany, S., & Aurbach, D., *Journal of Solid State Chemistry*, 2006, **179**, 1879–1882.