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THERMAL-SPAYED, THIN-FILM PYRITE CATHODES FOR THERMAL BATTERIES – DISCHARGE-RATE AND TEMPERATURE STUDIES IN SINGLE CELLS

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Abstract

Using an optimized thermal-spray process, coherent, dense deposits of pyrite (FeS_2) with good adhesion were formed on 304 stainless steel substrates (current collectors). After leaching with CS_2 to remove residual free sulfur, these served as cathodes in Li(Si)/FeS_2 thermal cells. The cells were tested over a temperature range of 450°C to 550°C under baseline loads of 125 and 250 mA/cm^2, to simulate conditions found in a thermal battery. Cells built with such cathodes outperformed standard cells made with pressed-powder parts. They showed lower interfacial resistance and polarization throughout discharge, with higher capacities per mass of pyrite. Post-treatment of the cathodes with Li_2O coatings at levels of >7% by weight of the pyrite was found to eliminate the voltage transient normally observed for these materials. Results equivalent to those of standard lithiated catholytes were obtained in this manner. The use of plasma-sprayed cathodes allows the use of much thinner cells for thermal batteries since only enough material needs to be deposited as the capacity requirements of a given application demand.

Introduction

The Li(Si)/FeS_2 couple is the main electrochemical system used in thermally activated ("thermal") batteries. These are primary batteries that utilize a molten salt as the electrolyte. Thermal batteries are used primarily as power sources in missiles and weapons applications. The batteries have an almost unlimited storage lifetime as long as hermeticity is maintained. However, once the electrolyte is melted by an internal pyrotechnic, the batteries become active and are capable of delivering higher current and power. This is made possible by the high intrinsic ionic conductivity of the molten salts used in the separator. The electrolyte that is commonly used is the LiCl-KCl eutectic that melts at 352°C. A battery built with pyrite (FeS_2) cathodes and this electrolyte functions over a temperature range of 400°C to 550°C.

All the cell components are made by cold pressing of powder mixes into discs. The cells are then stacked as needed to obtain the desired voltage. [The Li(Si)/FeS_2 couple, for example, provides a nominal 2-V output at 500°C.] The disadvantage of using pressed powders is that there is a minimum finite thickness that can be used before parts become difficult to handle. It is almost impossible to prepare pellets that are 3" or more in diameter with a thickness of 0.020" or less. Consequently, for some limited-life applications, batteries are made with much more active material than is necessary simply because of these mechanical constraints.

Thermal (plasma) spraying of active cathode materials is appealing because only the actual amount of needed material has to be deposited. With thermal-sprayed electrodes, there is no need for expensive dies for pressing of powders. If good adhesion is realized on large sheets of current-collector material—typically, 304 stainless steel—electrodes can simply be punched in the desired sizes. Since the plasma-spray process is carried out under close computer control, the thickness and uniformity of the electrode deposits can be readily controlled. The use of thinner electrodes also reduces the mass of pyrotechnic needed for activation. Therefore, a significant reduction in volume and weight can be realized, which translates into a higher energy density and specific energy.

In earlier work, we demonstrated that pyrite can be deposited as thin (0.015"-thick) films on 304

stainless steel substrates.[1-6] Subsequently, we were similarly able to deposit films of CoS_2.[7] In both cases, the addition of elemental sulfur to the feedstock was necessary to repress the thermal dissociation of the metal disulfides.

Pyrite decomposition becomes significant at temperatures above 550°C, according to equation 1.

$$FeS_2 \text{ ------> } FeS + 0.5S_{2(g)} \quad (1)$$

Similar decomposition occurs for CoS_2 above 650°C, according to equation 2.

$$3CoS_2 \text{ ------> } Co_3S_4 + S_{2(g)} \quad (2)$$

Sulfur acts according to LeChatlier's Principle to drive the reactions to the left while the sulfides are passing through the plasma. In addition, the sulfur coating acts as a thermal barrier, to lower the temperature of the sulfide particles by evaporation during plasma spraying.

The use of sulfur has the disadvantage of requiring the removal of excess material—typically, by leaching with CS_2—before the electrodes can be tested in a battery. Otherwise, the free residual sulfur would boil away during battery activation and exothermally react with the Li(Si) anode, generating enough heat to cause a potential thermal runaway. The adhesion of the deposit to the stainless steel substrate was inadequate in earlier work to allow punching of coupons for testing after the sulfur is removed. However, under optimized plasma-spraying conditions, much better adhesion to the stainless steel current collectors has been realized.

An additional issue relates to the voltage transient that occurs on activation of thermal batteries with pyrite cathodes. Normally, the pyrite catholytes are lithiated to fix the activity of Li in the cathode. This fixes the potential, so that there is no voltage excursion during activation due to thermal effects. [The voltage of the Li(Si)/FeS_2 couple shows a positive dependency with increase in temperature.] Any free sulfur contributes to the voltage "spike" because of the higher voltage of the Li(Si)/S_2 couple (over 2 V) vs. the Li(Si)/FeS_2 couple.

We have been exploring the optimization of the thermal-spray process to improve both the mechanical and electrochemical properties of the pyrite deposits. In previous work, testing was done at only a single current density. In this work, we report on the electrochemical characterization of plasma-sprayed pyrite deposits formed under optimized conditions. These were tested in single cells over a temperature range of 450°C to 550°C at several current densities. The results were compared to those obtained using standard pressed-powder parts. We also investigated the post-treatment of the deposits (after removal of free sulfur) with Li_2O coatings, to minimize the voltage transient that is normally observed with these materials. The use of alternate thermal-barrier coatings is briefly discussed, as a replacement to the sulfur coating currently used

Experimental

Thermal Spraying

The pyrite powder was from American Mineral and was –325 mesh (~44 µm in diameter). It was leached in HCl solution (1:1 v/v) to remove acid-soluble impurities and was then washed with deionized water. After vacuum drying at 120°C, the pyrite powder was mixed with sulfur powder and ball milled, to improve the flowability and provide a thermal-barrier coating.

Plasma spraying was carried out with a Metco 9MB plasma spray system (Metco, Westbury, NY). A dc arc current of 200 A to 400 A was generated in the copper nozzle. The pyrite powder was fed radially into the plasma stream near the nozzle exit at a rate of 1 kg/h to 5 kg/h. The plasma output was directed to the supported 1.25"-dia. by 0.005" thick 304 stainless steel substrates, positioned 5 mm to 10 mm away.

Materials

Flooded anodes of 25% LiCl-KCl eutectic electrolyte and 75% active anode (44% Li/56% Si, Foote) were used for all tests. The final anode pellet weight was 0.93 g. The presence of electrolyte aids in pelletizing and improves the electrochemical performance. The separator was formulated with 35% Maglite 'S' MgO (Merck) and LiCl-KCl eutectic and weighed 1.0 g. A standard catholyte was formulated with 73.5% FeS_2 (-325 mesh, HCl purified, Climax), 25% separator, and 1.5% Li_2O (Cerac) as a lithiation source. The standard cathode pellet weighted 1.03 g. All processing of powders and materials and cell assembly was conducted in a dry room maintained at <3% relative humidity.

Apparatus and Testing

The single cells were 1.25" (3.18 cm) in diameter and were tested in a glovebox under high-purity argon that contained <1 ppm each water and oxygen. The single cells were tested under computer control using a programmed galvanostat (PAR 371). A constant-current load of 1.0 A (125 mA/cm^2) was used as a background in most cases. Every 30 s, the current density was doubled for 0.5 s, to obtain polarization information. Supplemental tests were also conducted at a background current of 2 A (250 mA/cm^2), with pulses of 4 A (500 mA/cm^2). The cells were tested over a temperature range of 450°C to 550°C, to bracket the normal operating range of a thermal battery.

Steady-state voltage readings were taken with an HP3456A digital voltmeter (DVM) interfaced to an HP3497A data acquisition unit that mutiplexed several channels. Pulse readings were taken with HP3458A high-speed DVMs at a 1.2 kHz rate. The cells were typically discharged to a 1.0-V cutoff.

Results and Discussion

Reproducibility

In earlier work with plasma-sprayed pyrite cathodes made in the presence of sulfur, single cells were tested without removal of residual sulfur.[5] In the present work, the free sulfur was removed by leaching with CS_2.

The repeatability of performance of $Li(Si)/FeS_2$ cells made with CS_2-leached plasma-sprayed pyrite cathodes is shown in Figure 1 for a temperature of 450°C and Figure 2 for a temperature of 550°C.[*] A background current density of 125 mA/cm^2 (1 A) was used for both conditions.

The reproducibility was very good for both temperatures and was comparable to that of the standard cells with pressed-powder parts. The cell polarization (resistance) was quite flat for the entire discharge in both cases.

Relative Performance

Figure 3 compares the performance of the plasma-sprayed cathode and the standard pressed-powder cathode in single-cell tests at 450°C under a 125 mA/cm^2 steady-state load and 250 mA/cm^2 (2 A) pulses.

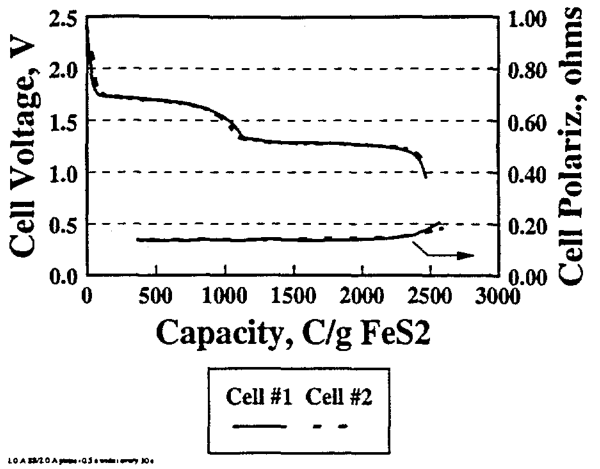

Figure 1. Reproducibility of Performance of Li(Si)/LiCl-KCl/FeS_2 Cells with CS_2-leached Plasma-Sprayed Cathodes at 450°C and 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

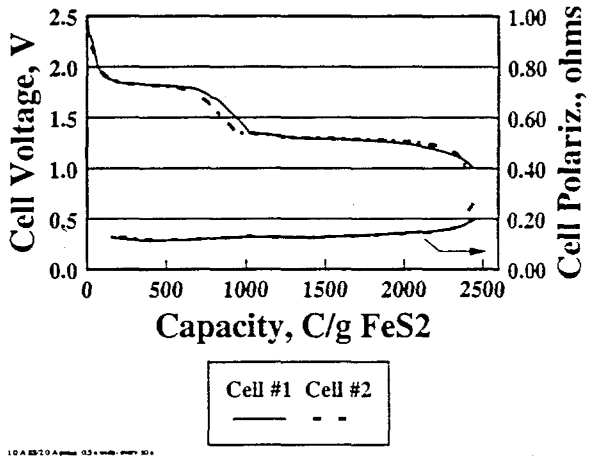

Figure 2. Reproducibility of Performance of Li(Si)/LiCl-KCl/FeS_2 Cells with CS_2-leached Plasma-Sprayed Cathodes at 550°C and 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

The voltage for the cell with CS_2-leached plasma-sprayed pyrite cathode was greater than that of the standard cell for the entire discharge. The large voltage transient at the front end of the discharge is a result of the plasma-sprayed cathode not being lithiated, which gives rise to this behavior, even in the absence of free sulfur.

The corresponding polarization for this cell was similar to that of the standard cell but did not show the resistance hump near 700 C/g FeS_2 associated with the phase transformation to $Li_3Fe_2S_4$ from

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[*] The curves have been normalized to the weight of active FeS_2 that was present. This was determined by weighing the samples after CS_2 leaching prior to testing and subtracting the weight of the 304 stainless-steel current collector.

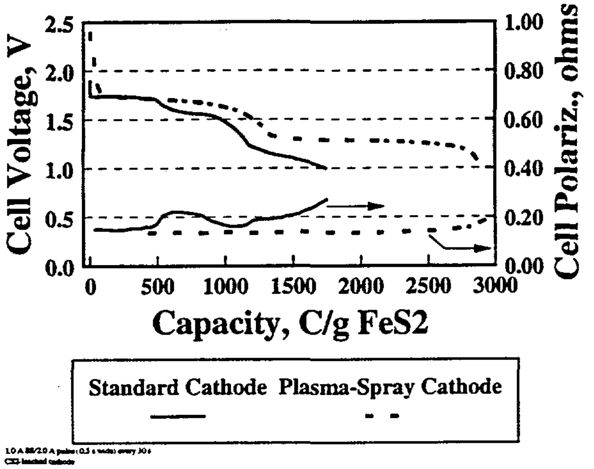

Figure 3. Comparison of the Performance of Li(Si)/LiCl-KCl/FeS_2 Cells with CS_2-leached Plasma-Sprayed Cathodes and Pressed-Powder Cathodes at 450°C and 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

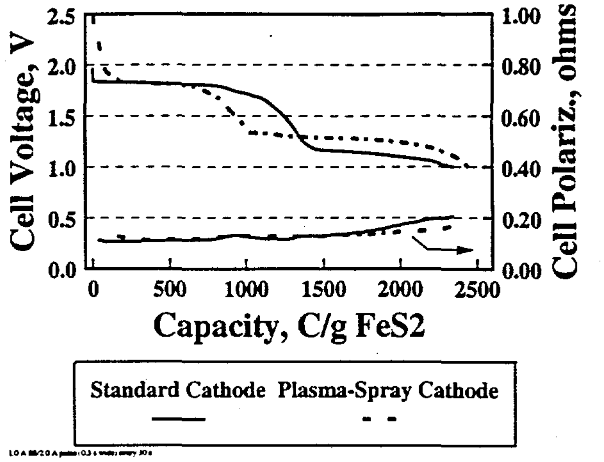

Figure 4. Comparison of the Performance of Li(Si)/LiCl-KCl/FeS_2 Cells with CS_2-leached Plasma-Sprayed Cathodes and Pressed-Powder Cathodes at 550°C and 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

FeS_2. This is believed to be due to the better electrical contact through the plasma-sprayed samples. The standard cathode shows this behavior due to an increase in the interparticle contact resistance which is absent in a high-density, pseudo-monolithic plasma-sprayed deposit. The cell polarization (resistance) was very constant throughout the entire discharge and did not increase significantly until after 2,600 C/g FeS_2 had been removed. This type of behavior is never seen with the standard pressed-powder cathodes.

The corresponding comparison under the same conditions but at 550°C is shown in Figure 4. The length of the first discharge plateau was shortened for the cell with the plasma-sprayed cathode but the second plateau was higher in voltage and longer. The drop in the capacity for the first plateau may reflect some loss of sulfur at the much higher temperature relative to the run at 450°C, so that the actual mass of FeS_2 would have been reduced as a result. (Subsequent thermogravimetric analysis revealed that some residual sulfur remains trapped in the deposit after CS_2 leaching.)

The total polarization of the cell with the plasma-sprayed cathode was comparable to that of the standard cell, except there was no evidence of the small maximum near 960 C/g FeS_2 and the rate of increase of polarization at the end of life was less.

Effect of Load

The effect of load on the performance of the single cells at 450°C is shown in Figure 5 for cells built with the standard pressed-powder cathodes. The polarization hump was not readily evident at the 2-A (250 mA/cm^2) background load, due to parallel discharge processes. The cell lifetime dropped markedly in going from 1 A (125 mA/cm^2) to 2 A (from ~1,750 C/g FeS_2 to ~950 C/g FeS_2).

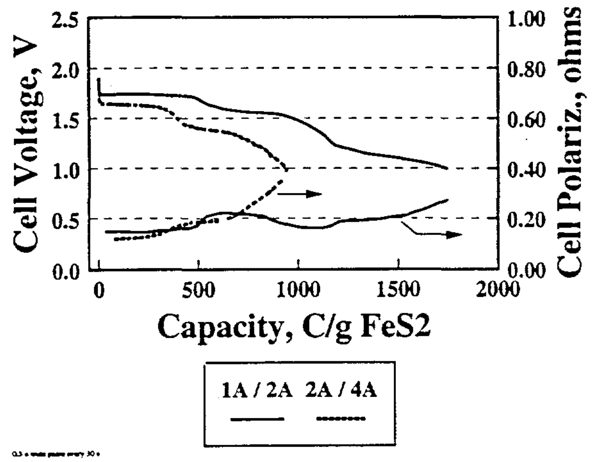

Figure 5. Effect of Load on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with Standard Pressed-Powder Cathodes and Tested at 450°C.

The corresponding data for the cells with the plasma-sprayed cathodes are presented in Figure 6. The differences in performance were not nearly as great for the two loads as was noted for the standard cells (Figure 5). The voltage drop at the higher load was not large and the shapes of the two discharge curves were very similar, being only offset slightly. This is the type of behavior that is preferred from the perspective of a thermal-battery engineer.

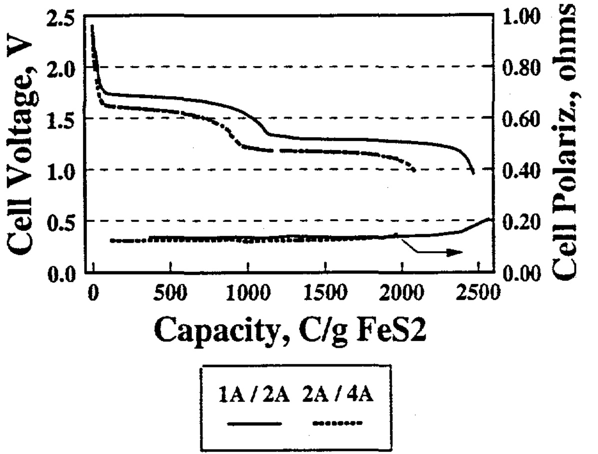

Figure 6. Effect of Load on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with CS_2-leached Plasma-Sprayed Cathodes and Tested at 450°C.

The effect of load on the performance of single cells at 550°C is shown in Figure 7 for cells built with the standard pressed-powder cathodes. There was a slight reduction in the voltage at the higher load but the total polarization was very similar for both loads. The corresponding data for the cells with the plasma-sprayed cathodes are shown in Figure 8.

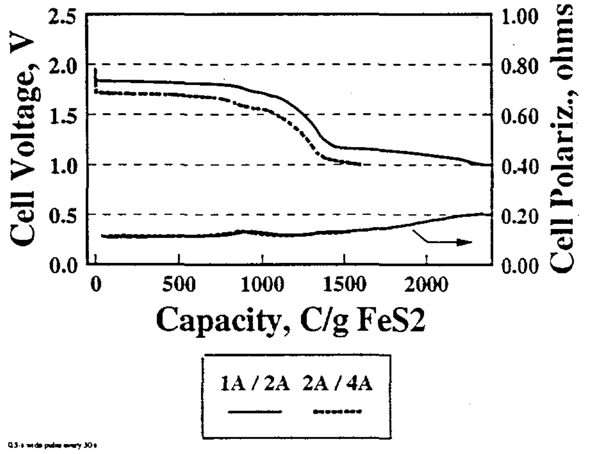

Figure 7. Effect of Load on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with Standard Pressed-Powder Cathodes and Tested at 550°C.

The relative difference in performance at the two loads at 550°C mirrored that observed at 450°C (Figure 6). The total cell polarization remained surprisingly steady over the length of the discharge, which is quite a desirable behavior.

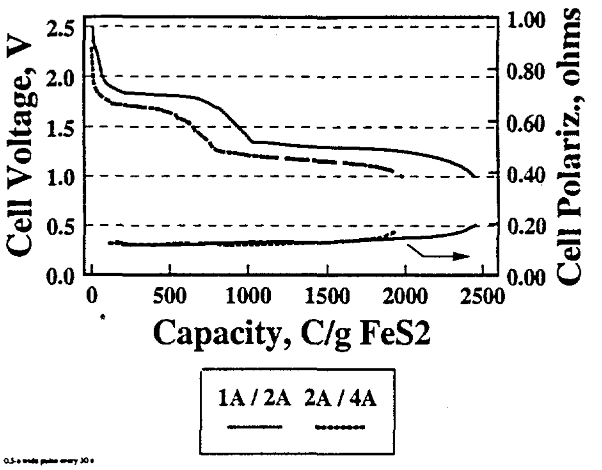

Figure 8. Effect of Load on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with CS_2-leached Plasma-Sprayed Cathodes and Tested at 550°C.

Effect of Temperature

The effect of temperature on cell performance is shown in Figure 9 for a steady-state load of 125 mA/cm^2 and a pulse load of 250 mA/cm^2 for the cells with the standard pressed-powder pyrite cathodes.

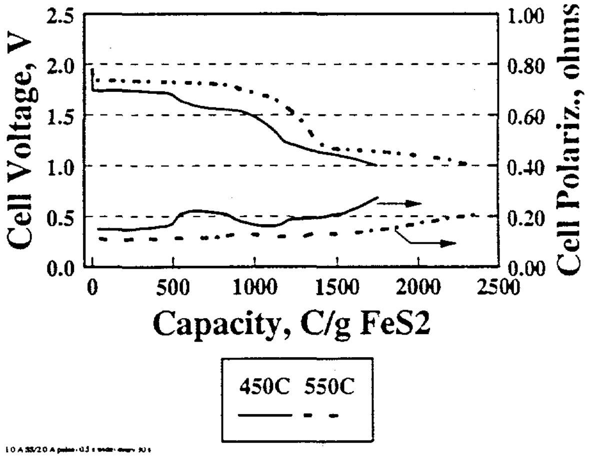

Figure 9. Effect of Temperature on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with Standard Pressed-Powder Cathodes and Tested at 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

As expected, the voltage was higher at the higher temperature, due to the enhanced kinetics and higher thermodynamic emf under these conditions. Another notable feature is the maximum in the total cell polarization at 450°C was substantially reduced at 550°C. The position of the maximum also shifted from near 600 C/g FeS_2 at the lower temperature to 950 C/g FeS_2 at the higher temperature. This reflects more complete conversion to the pyrite to the first discharge phase prior to subsequent reduction to the next discharge phase (Li_2FeS_2).

The corresponding data for the cells with the plasma-sprayed pyrite cathodes are presented in Figure 10. As mentioned earlier, the voltage transient at the start of discharge was pronounced for both temperatures. The relative differences in performance at the two temperatures were much less than for the cells with the pressed-powder cathodes. The total cell polarization remained stable throughout the discharge for both temperatures, except near the end of life. There was no evidence of any maximum in polarization as for the standard cells.

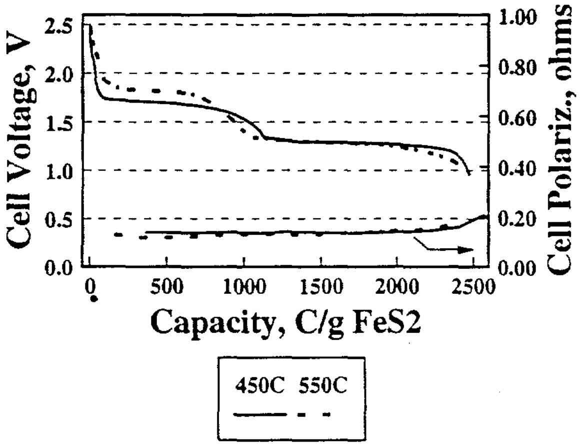

Figure 10. Effect of Temperature on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with CS_2-leached Plasma-Sprayed Cathodes and Tested at 125 mA/cm^2 Background with 0.5 s 250 mA/cm^2 Pulses every 30 s.

The effect of temperature on cell performance for a steady-state load of 250 mA/cm^2 and a pulse load of 500 mA/cm^2 for the cells with the standard pressed-powder pyrite cathodes is shown in Figure 11.

The relative performance at the two temperatures at this higher load reflected that observed at the lower load (Figure 9). The data of Figure 12 for the cells with the plasma-sprayed cathodes show that the behavior at the higher load was similar to that observed at the lower load (Figure 10).

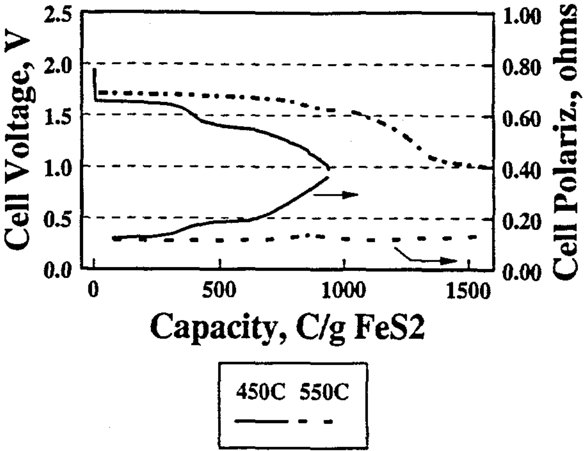

Figure 11. Effect of Temperature on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with Standard Pressed-Powder Cathodes and Tested at 250 mA/cm^2 Background with 0.5 s 500 mA/cm^2 Pulses every 30 s.

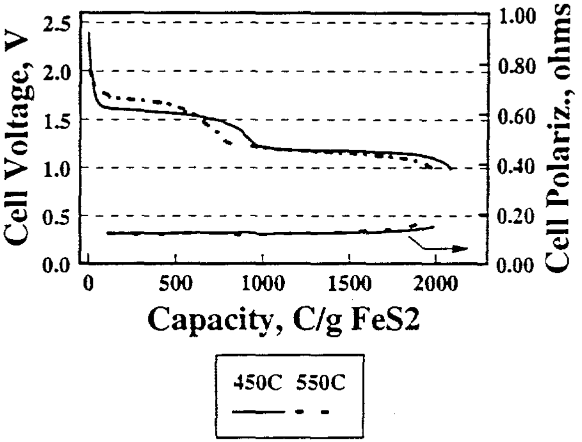

Figure 12. Effect of Temperature on Performance of Li(Si)/LiCl-KCl/FeS_2 Cells Made with CS_2-leached Plasma-Sprayed Cathodes and Tested at 250 mA/cm^2 Background with 0.5 s 500 mA/cm^2 Pulses every 30 s.

The discharge data for the two types of cells tested at two loads and two temperatures are summarized in Table 1. The capacity of the cells with the standard pressed-power cathodes dropped by half at 450°C when the load was doubled. The capacity for the cells with the plasma-sprayed cathodes dropped only 15% under the same conditions. In addition, the capacity obtained with the plasma-sprayed cathodes was much greater—38% at the 125-mA/cm^2 load and 1.5 times greater at the 250-mA/cm^2 load. The drop in capacity for the standard cells at 550°C when the load was doubled was ~38% less than that at 450°C. The decrease in the case of the plasma-sprayed cells was only ~20% under the same conditions. The capacity of both types of

Table 1. Summary of Single-Cell Test Data for Cells with Standard Pressed-Powder and Plasma-Sprayed Pyrite Cathodes.

Temperature, °C	Cathode Type	Steady-State/Pulse Load, A	Capacity to 1.0 V Cut-off, C/g FeS_2
450	Pressed-powder	1.0/2.0	1,750
		2.0/4.0	835
	Plasma-sprayed	1.0/2.0	2,417
		2.0/4.0	2,067
550	Pressed-powder	1.0/2.0	2,424
		2.0/4.0	1,509
	Plasma-sprayed	1.0/2.0	2,440
		2.0/4.0	1,967

cells was comparable at the higher temperature for the light load but the cells with the plasma-sprayed cathodes still outperformed the standard cell at the higher load, with ~30% greater capacity.

Removal of Voltage Spike

The main difficulty in using the plasma-sprayed pyrite electrodes in thermal batteries has been the presence of the voltage transient at the beginning of discharge. This can be eliminated in the case of pressed-powder cathodes by lithiation with Li_2O.[8-10] Post-treatment Li_2O-coating tests were carried out with the plasma-sprayed cathodes after leaching with CS_2) in an attempt to remove the initial voltage transient that occurs during discharge.

Initial tests were performed by sprinkling of Li_2O onto a plasma-sprayed cathode that had been wet with water. The excess material was removed and the sample vacuum dried to remove the water. This was quite effective in removal of the voltage spike but at the expense of increased polarization. The coating was far too thick, with a resultant concentration of 30% or more of the total active FeS_2 present. (In the standard catholyte, the Li_2O content is only 1.5%.) This resulted in a doubling of the cell resistance.

In a second approach, a 5% Li_2O solution was used to coat the plasma-sprayed deposit, which was then vacuum dried to remove excess water. This allowed the deposition of much smaller quantities of material in a more controlled fashion than was possible with the use of Li_2O powder. There was no measurable effect on the voltage spike at low levels of Li_2O (<1% of the mass of the pyrite). However, at levels of 7% Li_2O or more, substantial improvements were realized. The results for a single-cell test at a level of 15.7% Li_2O are shown in Figure 13 for a temperature of 450°C and a load of 125 mA/cm^2. Data are also presented for an untreated plasma sprayed cathode for comparison.

The initial voltage spike was completely eliminated for the cell with the Li_2O coating on the cathode. The overall polarization for this cell was comparable to the cell without the coating. Application of the Li_2O coating by an aqueous process allows much better control of the overall final film thickness. This prevents the rapid buildup of resistance noted for the cells with heavier powdered Li_2O coatings.

The cathode was not preheated for this test. It may be possible to use less Li_2O and preheat the sample to accelerate the lithiation process. More work is underway to optimize the Li_2O-coating procedure while still maintaining satisfactory electrochemical performance.

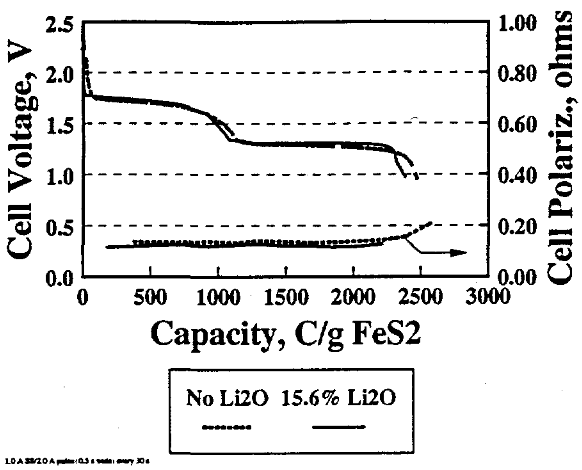

Figure 13. Performance at 450°C of Li(Si)/LiCl-KCl/FeS_2 Cells with CS_2-leached Plasma-Sprayed Cathodes Uncoated and Coated with 15.6% Li_2O. Background Load of 125 mA/cm^2 was Increased to 250 mA/cm^2 for 0.5 s every 30 s.

Future Work

We are currently evaluating the use of other types of coatings to replace the sulfur that is presently used. The need to remove residual sulfur from the pyrite deposits is an undesirable step. Any free sulfur gives rise to an elevated voltage that is also undesirable. A number of halide and mixed-halide salt mixtures are being examined for coating of the pyrite.[11] These materials serve as thermal-barrier coatings for the pyrite, where the heat of fusion acts to protect individual particles from being overheated during passage through the plasma gun. This, in turn, will keep the pyrite particles from exceeding the decomposition temperature under the proper conditions.

By selection of the proper formulation, it will be possible to control the heat of fusion and melting point to custom tailor the coating for individual materials. The preliminary results with pyrite are very promising.

In addition to single cell tests with 1.25"-dia. cells, work is also underway with much larger 3"-dia. cells, to validate the performance over a range of electrode sizes. Complementary battery tests with five or more cells with both sizes of these electrodes are also planned. This is to verify that the performance observed in single-cells will also be realized under dynamic battery conditions, where the temperature can rise from ambient to over 600°C in several hundred milliseconds or less.

Conclusions

The experimental conditions have been optimized for plasma spraying of thin (≤ 0.015"-thick) films of pyrite onto 304 stainless steel substrates for use as a cathode in thermal batteries. When using a sulfur additive (coating), adherent deposits are obtained that are coherent and uniform, with reasonable adhesion. After leaching with CS_2 to remove residual free sulfur, the electrodes were tested in Li(Si)/LiCl-KCl/FeS_2 single cells over a temperature range of 450°C to 550°C at background loads of 125 and 250 mA/cm^2.

Cells tested with standard pressed-powder parts show a maximum in the total polarization (resistance) during a phase transition of FeS_2 to $Li_3Fe_2S_4$. In contrast, cells with plasma-sprayed electrodes show a consistent, uniform polarization without the presence of any maxima. This is attributed to the dense, pseudo-monolithic nature of the plasma-sprayed deposits, which avoids the higher intrinsic resistivity of particle-to-particle contact associated with pressed-powder cathodes. The good adhesion of the plasma-sprayed deposit to the current collector also reduces the interfacial contact resistance.

The cell lifetimes at both discharge loads are substantially greater at 450°C for the plasma-sprayed cathodes, compared to the pressed-powder cathodes. This is especially true at the higher load of 250 mA/cm^2. Differences are much less at 550°C at a load of 125 mA/cm^2, but still much greater for the cells with plasma-sprayed cathodes at the higher load.

The plasma-sprayed pyrite cathodes outperform the standard pressed-powder cathodes overall. Their use in thermal batteries has the advantages of reducing battery weight and volume by minimizing the amount of active pyrite to only that necessary for a given application. This avoids the limitations imposed by mechanical constraints when using pressed-powder parts. This translates into higher specific energies and energy densities for thermal batteries.

Post-treatment of the plasma-sprayed pyrite deposits with a coating of 15.6% Li_2O (relative to the FeS_2 mass) is shown to remove the voltage spike that normally occurs with these materials on activation. Further work in this area and in the use of salt thermal-barrier coatings are expected to improve the overall electrochemical performance of the plasma-sprayed pyrite deposits while at the same time enhancing their mechanical properties.

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