Report from Visit to Blacklight Power on Friday January 17, 2014

Nick Glumac, Ph.D.

I. Summary

During the visit to Blacklight Power, experiments were conducted in four general classes: high-current initiation of H_2O-based fuel pellets, similar initiation within a calorimeter under a helium atmosphere, high voltage discharges in water inside a bomb calorimeter, and DSC testing of a solid fuel. Multiple tests were performed for each class, including controls. Sample preparation and processing were observed for nearly all tests. All tests provided evidence for phenomena that are not readily explained with conventional thermochemistry. No experimental flaws could be identified which would lead to simple, alternative explanations.

II. Tests Observed and Results

This section will be broken into four parts, each discussing a different class of experiments. The experimental approach will be reviewed, followed by the results.

II.A Open Air, High-Current Initiated Plasmas

Several samples were exposed to the high current from a commercial spot welder (Acme Electric Welder Company model 3-42-75, 75 KVA with Warren control, Model 108). The system used copper alloy electrodes rods (RWMA class 2 copper/ C18200, 0.6 to 1.2% Cr, 5/8" OD) that were replaced after every test. The operating voltage was 6 volts RMS (60 Hz AC), though some voltage deviations were observed during the actual initiation event, possibly due to power-wave reflection from the circuit interruption by the high pressure of the blast following the initiation. Current was measured with a Rogowski coil, and data were sampled at 80 KS/s using a Labview interface to the sensors (Additional details are given in Sec. IIB). The control test using a nickel sheet provided a baseline for the event, and these data are shown in Figure 1.

As seen in the figure, the event lasts roughly 30 ms. There is a tail on the current trace that lasts at least a few seconds. The current during this tail portion is less than 1% of the peak current and integrates to less than 1 joule. In the control test, the voltage stays within the +/- 6V bounds but shows interruptions at times, possibly due to the welder controller that shorts the power at controlled intervals to control the energy delivered to the sample. The current follows a regular AC curve (with the exception of a short interruption at 5 ms after contact). The peak current is roughly 20 kA. For the control run, the integral of VI dt yields 817 Joules.

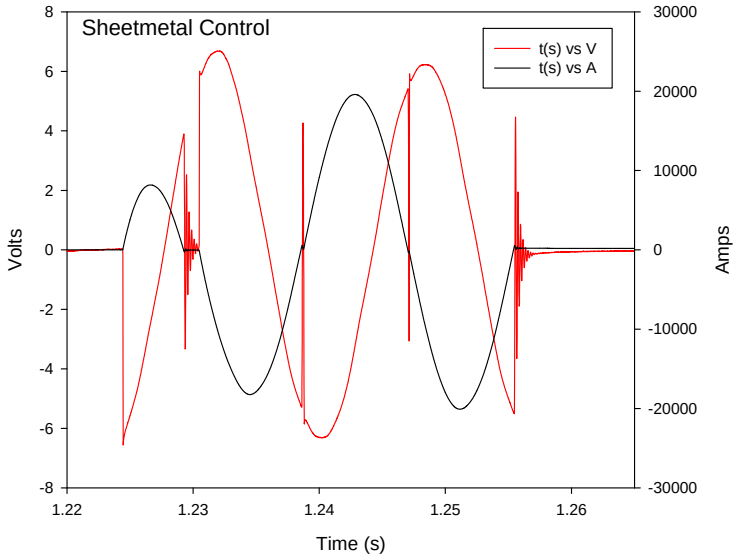

Figure 1: Nickel sheet control on the instrumented spot welder.

In addition to the nickel sheet control, eight tests were run, and the conditions were as follows:

1) Setaram 30 microliter Aluminum crucible. Empty.

2) Crucible with 30 mg deionized water.

3) Crucible with Cu, CuO, and 15 mg water mixed.

4) Crucible with 30 mg water and large mesh copper (100 mg).

5) A pressed silver pellet with no additives.

6) A silver pellet with $MgCl_2$ and 30 uL water, pressed.

7) A gold 0.010” diameter wire.

8) A repeat of test 6 with the sample prepared immediately before the run.

Control tests (Tests 1, 5, and 7) produced no appreciable acoustic or light-emitting event. Traces were similar to the nickel sheet control test. In the wire test, no wire explosion or disruption in conductivity was observed. The current/voltage trace from test 5 is shown below:

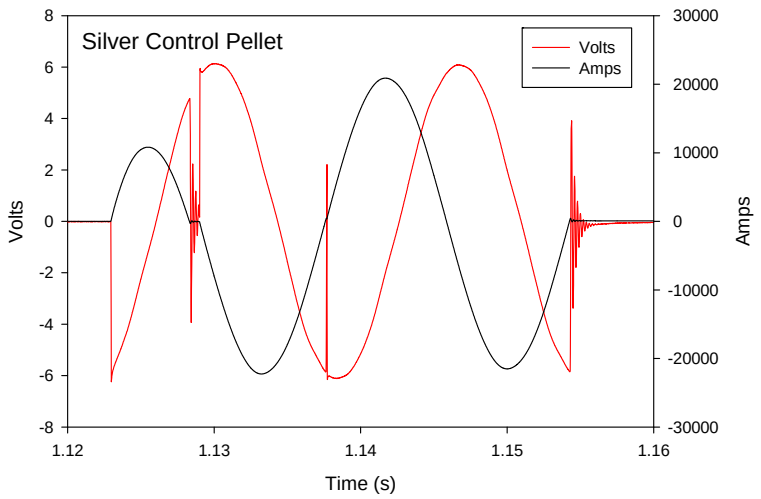

Figure 2: Traces from the test using a silver control pellet.

This trace is very similar in all aspects to the nickel control. Integrated power was 595 J. Tests in which there was water vapor present showed significantly different behavior, both qualitatively and quantitatively. Tests were accompanied by a loud and sharp noise, qualitatively similar to that of small firecracker. In addition, there was typically a fairly bright flash of light. In some cases, there was visible ejection of bright streaming particulates. The current/voltage traces were also significantly different. For example, test 8 (for which test 5 served as the control) is shown in Figure 3 below.

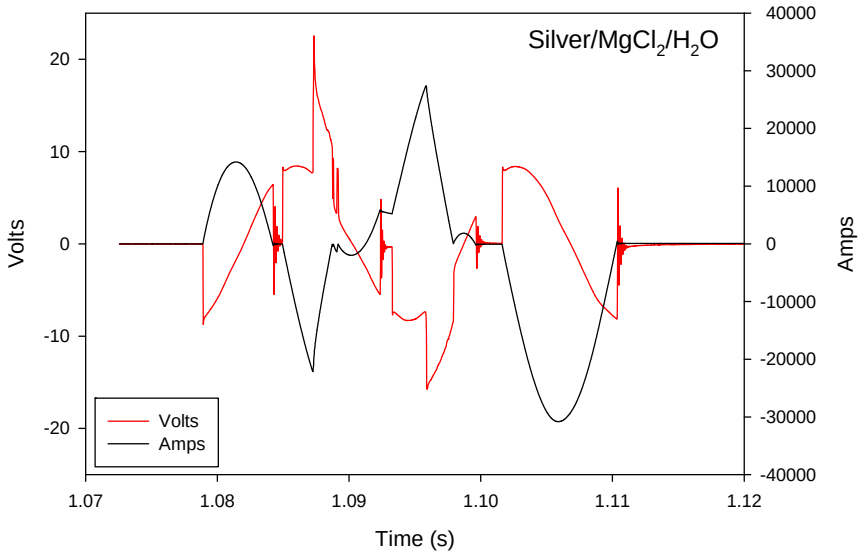

Figure 3: The current and voltage traces for Test 8 (Silver/$MgCl_2$/H_2O). Integrated power was 1582 Joules.

The event duration (set by the instrument) is the same. However, there are much larger deviations from the baseline AC traces. The current exceeds 30,000 amps at peak. Disruptions in the AC profile are more numerous and more pronounced. This effect is especially pronounced for the current trace, which showed only minor deviation from a sinusoidal profile in the control experiments. The voltage trace also spikes well outside its +/-6V envelope, reaching above 20 volts at one point, possibly due to power-wave reflections with mechanical disruptions of the power-flow from the pressure of the ensuing reaction of the H_2O-based solid fuel. Similar traces were observed for all reactive tests (Tests 2, 3, 4, 6, and 8). But, there were qualitative differences in the output from the tests. For instance, test 3 was louder than Test 2. However, these observations were difficult to quantify. In addition to tests 6 and 8, there was one other pellet made with the same composition, but which did not ignite during testing.

Electrodes were generally observed to be heavily pitted after a reactive test, with pits perhaps one or two mm in diameter, and typically just under 1 mm deep. Similar results for identical active tests run under an argon atmosphere were reported by BLP.

II.B Energy Balances of High-Current Initiation of Plasmas in a Bomb Calorimeter

These experiments were intended to replicate the results of the open spot welder tests in a controlled environment where thermal output could be measured by initiating the energetic plasma within a calorimeter vessel under a helium atmosphere. The calorimeter was a Parr 1341 plain-jacketed calorimeter with a Parr 6775 calorimeter thermometer option. A Parr 1108 oxygen combustion chamber of the bomb calorimeter was modified to permit initiation of the chemical reaction with high current. Copper rod ignition electrodes that comprised 1/2” outer diameter (OD) by 12” length copper cylinders were fed through the sealed chamber containing a nickel sheet as a control resistive load for calibration of the heat capacity of the calorimeter or a test solid fuel pellet wherein the ends had a copper clamp that tightly confined each sample. The calorimeter water bath was loaded with 2,000 g DI water (as per Parr manual). The system was allowed to equilibrate after loading. Helium gas was used as a purge to eliminate possible air reactions and speed thermal equilibration. The power source for calibration and ignition of the solid fuel pellet was a Taylor-Winfield model ND-24-75 spot welder that supplied a short burst of electrical energy in the form of a 60 Hz low-voltage of about 8 V RMS and high-current of about 10,000 to 20,000 A. The input energy of the calibration and ignition of the solid fuel was given as the product of the voltage and current integrated over the time of the input. The voltage was measured by a data acquisition system (DAS) comprising a PC with a National Instruments USB-6210 data acquisition module and Labview VI. The current was also measured by the same DAS using a Rogowski coil (Model CWT600LF with a 700 mm cable). V and I input data was

obtained at 10 KS/s and a voltage attenuator was used to bring analog input voltage to within the +/-10V range of the USB-6210.

Using the nickel sheet sample with a measured energy input by the spot welder, the calibrated heat capacity of the bomb calorimeter and electrode apparatus was prior determined by BLP to be 12,800 J/°C. Three tests were run: a nickel sheet control and two solid fuel tests. Specifically, each sample of solid fuel comprised Cu + CuO + H_2O that was sealed in an aluminum DSC pan (70 mg) (Aluminum crucible 30 µlL, D:6.7x3 (Setaram, S08/HBB37408) and Aluminum cover D: 6,7, stamped, tight (Setaram, S08/HBB37409)). The nickel sheet control data are shown in Figure 4:

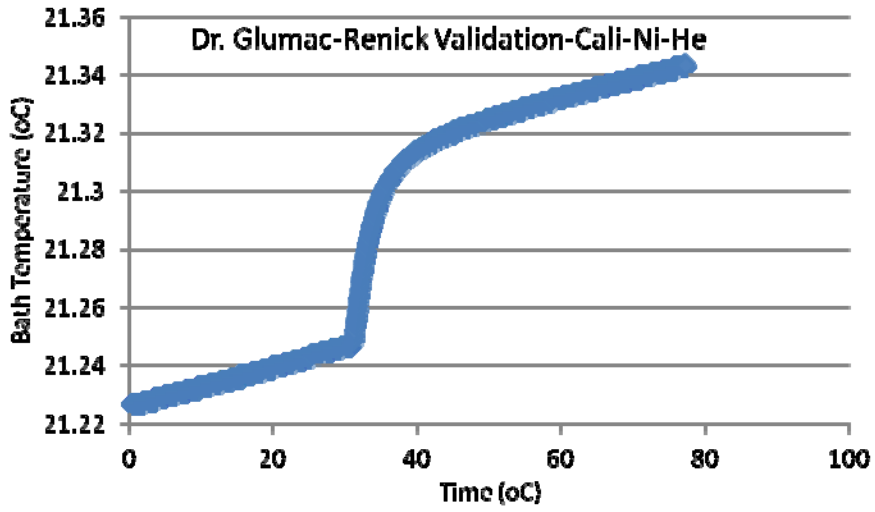

Figure 4: Nickel sheet control run.

For this test, the integrated VI over the waveform duration was 750.00 Joules of energy to the sample. The output calculated from the easily resolvable temperature rise of 0.071 °C and the prior determined heat capacity was 759.91 Joules. The net was only 9.91 J from the expected value of zero net energy. These results suggest an accuracy of better than two percent at this energy input level.

The second and third tests (Figure 5) were designed to be essentially the same reactive variant. 43 mg CuO, 33 mg CuO, and 20 mg H_2O were mixed and sealed in the aluminum Setaram crucible in the second test, and 45/36/19.8 mg in the third. For the second calorimetric test, the energy input was 682.00 J, and the measured output was 1061.15 J on a 0.079 C temperature

rise. The third calorimetric test had 802.00 J in and 1247.57 J out corresponding to a temperature rise of 0.110 °C. The measured net energies from the solid fuels were 379.15 J and 445.57 J, respectively. The 1.56X and 1.80X output gains observed in these tests are far beyond any reasonable assessment of the uncertainty of this experiment.

The time of the initiation event measured very conservatively by the blast-induced mechanical and corresponding electrical waveform disruption time scale was 1 ms. Considering the under 1 ms release of net energies of 379.15 J and 445.57 J from a fuel volume of about 10 uL, the powers and power densities were 380 kW, 446 kW, 38 GW/liter, and 45 GW/liter, respectively.

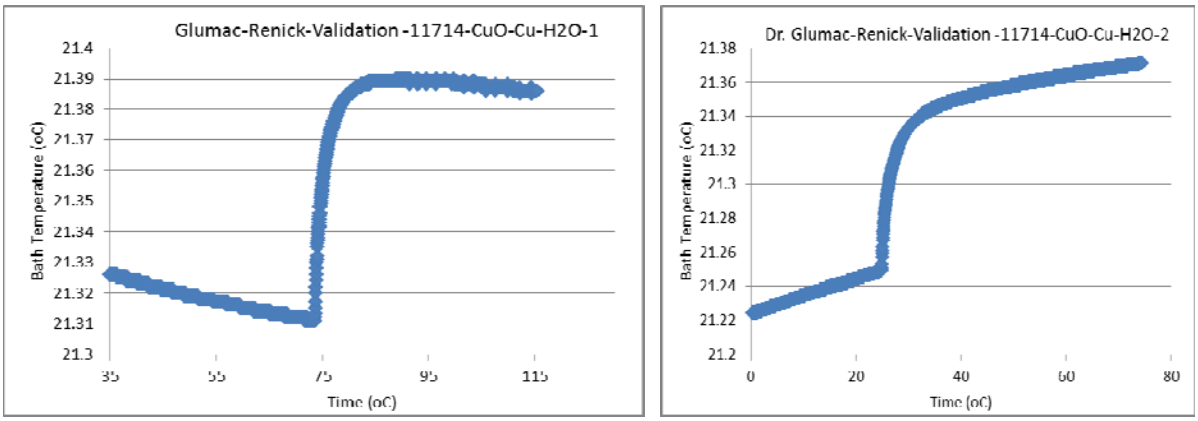

Figure 5: Calorimeter traces from reactive tests (Al crucible, CuO, Cu, H_2O).

II.C High-Voltage H_2O Arc Discharges in a Water Bath Calorimeter

These experiments were conceptually very simple and compelling. A high voltage discharge was directed through water within a calorimeter. The energy balance was performed assuming the energy available is $1/2CV^2$ from the capacitor bank. The system consisted of four nominally 16 uF capacitors, each with capacitance measured independently. The total capacitance was confirmed with a NIST-calibrated multimeter. The capacitors were rated for 4500 V, though less than 3 kV was used in these experiments. Mechanical contact was used to initiate the discharge. This setup would result in energy losses so that the amount of energy dissipated in the discharge would be less than $1/2CV^2$. The electrodes were 1" diameter discs placed 1/8" apart in water. There was shielding and buffering around the electrodes to minimize bulk flow of the calorimeter water. For the tests that we observed, the charge voltage was about 2500 V that provided capacitor-bank energy of about 200 J. The data that were provided to us showed an output of about 300 J. The 100 J Joule difference is well outside the uncertainty of the measurement.

II.D Differential Scanning Calorimetry

DSC measurements were performed on a candidate solid fuel system of iron (II) bromide and copper hydroxide. Samples were prepared as we watched, and the tests were run in a Setaram DSC 131. Standard gold DSC crucibles were used. The heating rate was 10 °C/min and a thermalization period preceded the test. Analysis was performed using the Setaram software, and an indium calibration was conducted as the second test in the series.

The first test involved 5.3 mg of $Cu(OH)_2$ and 11.9 mg $FeBr_2$, mixed and loaded under an argon atmosphere in a glove box. The second test involved 24.4 mg of indium. For the first test, a prominent exotherm was observed upon heating between 140 and 260 °C, with a very small exotherm seen on cooling. Curves are shown in Figure 6:

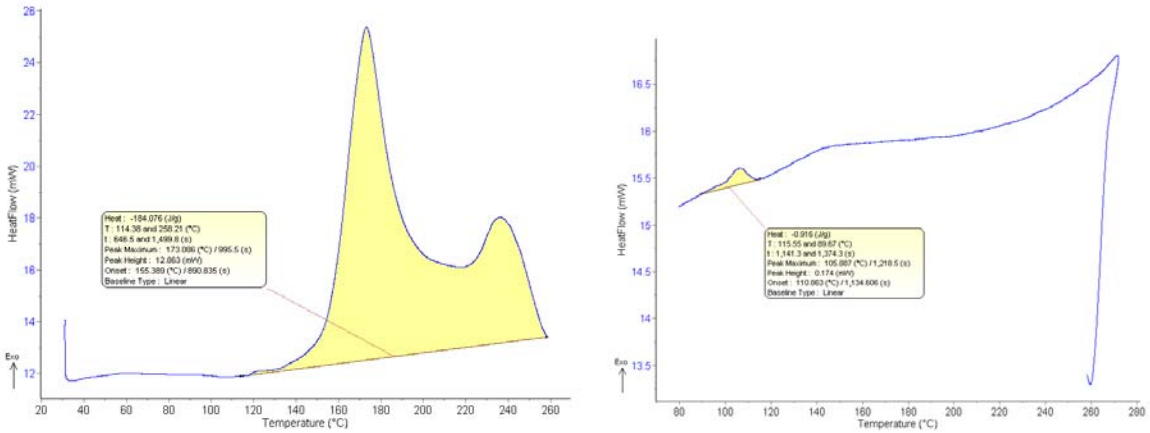

Figure 6: Heating (left) and cooling (right) traces for the reactive test sample.

The calibration sample traces are shown in Figure 7:

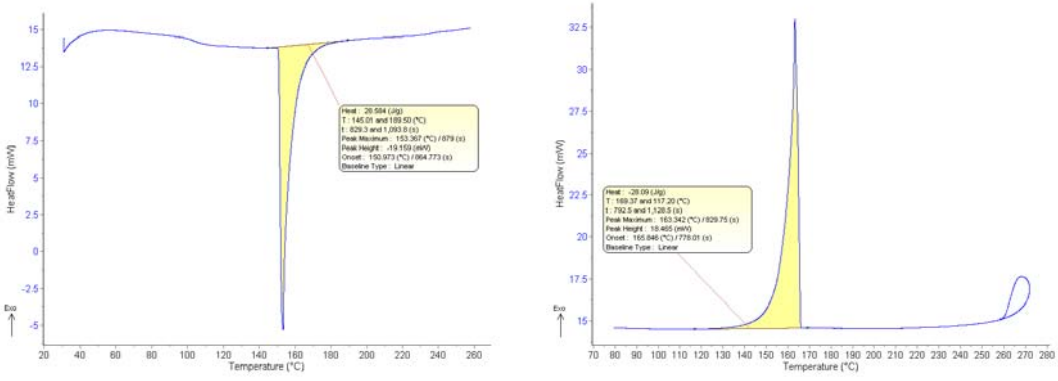

Figure 7: Indium calibration traces; heating (left), cooling (right).

The calibration traces suggested 28.6 and 28.1 J/g, which is in excellent agreement with the accepted value of 28.5 J/g, suggesting uncertainties of a few percent at most. For the test sample, the output was 184 J/g; although, the exotherm was probably not completed at 260 C. Thus, the 184 J/g value underestimates the total energy release for this case. BLP provided the analysis shown in Table 1. According to their analysis, the large energy release cannot be explained by conventional chemistry; the results being at least 280% that of the theoretical energy for any of the product combinations considered.

011714NG-JR-validation, Cu(OH)2 + FeBr2

Reactant	FeBr2.	Cu(OH)2							ΔE, J/g	Excess energy, J/g	Energy Gain
quantity, mg	11.80	5.30							-184.99	-118.81	2.80
quantity, mmol	0.0547	0.0544									
HOF, dH (KJ/mol)	-249.80	-450.00									

Product	Fe(OH)2	CuBr2	Fe2O3	FeBr2. 2H2O	CuBr	Cu2O	FeO	H2O	HBr (g)
HOF, dH (KJ/mol)	-574.00	-141.80	-822.20	-861.40	-104.60	-168.60	-272.00	-285.80	-36.30

Assumed Reaction I	Energy, kJ/reaction	FeBr2 used, mmol	Cu(OH)2 used, mmol	Cu(OH)2 left, mmol	FeBr2 left, mmol	Energy out, J	theoretical energy, J/g
Cu(OH)2 + FeBr2 = Fe(OH)2 + CuBr2	-16.00	0.0544	0.0544	0.0000	0.0004	-0.87	-50.86
10Cu(OH)2 + 9FeBr2 = 2Fe2O3+5FeBr2.2H2O+8CuBr+Cu2O+ 3/2O2	-208.20	0.0489	0.0544	0.0000	0.0058	-1.13	-66.18
3Cu(OH)2 + 3FeBr2 = Fe2O3 + FeO + 3CuBr + 2H2O + 2HBr + 1/2 Br2	47.40	0.0544	0.0544			0.86	

Table 1: BLP Theoretical Enthalpy of Reaction Calculation for Cu(OH)2 and FeBr2.

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2/19/14