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13. ABSTRACT (Maximum 200 words)

Attempts to achieve low temperature nuclear fusion can be traced to the work of Paneth, Peters, and Tanberg in 1927 [1]. Martin Fleischmann, who in his early career was a colleague of Paneth, teamed with Stanley Pons at the University of Utah to pursue this goal by the electrolysis of heavy water with a palladium cathode. Palladium is a metal which strongly absorbs hydrogen isotopes. The question they sought to answer was whether the internal pressure generated by this absorption would be sufficient to overcome the coulomb repulsion of deuterium nuclei. In 1989 they claimed success in fusing deuterium nuclei to form helium [2]. Their main evidence for this claim was excess heat which was orders of magnitude higher than could be explained by any known chemical reaction.

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INTERACTION OF TITANIUM WITH HYDROGEN ISOTOPES

FINAL PROGRESS REPORT

JOHN DASH

APRIL 27, 2001

U.S. ARMY RESEARCH OFFICE

GRANT NUMBER DAAG55-97-1-0357

PORTLAND STATE UNIVERSITY

1. FOREWORD

Attempts to achieve low temperature nuclear fusion can be traced to the work of Paneth, Peters, and Tanberg in 1927 [1]. Martin Fleischmann, who in his early career was a colleague of Paneth, teamed with Stanley Pons at the University of Utah to pursue this goal by the electrolysis of heavy water with a palladium cathode. Palladium is a metal which strongly absorbs hydrogen isotopes. The question they sought to answer was whether the internal pressure generated by this absorption would be sufficient to overcome the coulomb repulsion of deuterium nuclei. In 1989 they claimed success in fusing deuterium nuclei to form helium [2]. Their main evidence for this claim was excess heat which was orders of magnitude higher than could be explained by any known chemical reaction.

Great interest was generated by this claim, but most scientists who attempted to reproduce it were not successful. For example, about 200 scientists at Los Alamos National Laboratory attempted but only three claimed success [3]. The interest of the mainstream scientific community waned, but scientists around the world who had success continued to pursue this research. For example, the Eighth International Conference on Cold Fusion (ICCF8) was held in Italy in May 2000. There were 41 participants from Italy, 40 from the USA, 24 from Japan, 12 from Russia, and smaller numbers from 14 other countries [4]. Of the 110 abstracts which were submitted for presentation, 77 were accepted. The proceedings of this conference contain much evidence (detection of helium, elemental transmutations, and excess heat) which should be proof enough that low temperature nuclear fusion is a reality. ICCF9 will be held in Beijing at Tsinghua University, which is one of the most prestigious universities in China, in 2002.

2. LIST OF ILLUSTRATIONS

3. STATEMENT OF THE PROBLEM STUDIED

Titanium, which strongly absorbs hydrogen isotopes, is an attractive low-cost alternative to palladium, which is currently the most costly noble metal. In fact, titanium forms TiH_2 (or TiD_2) whereas palladium forms PdH (or PdD) which contain about half as much of the hydrogen isotopes as the titanium compounds. Our preliminary experiments indicated that an electrolytic cell with a titanium cathode ran at a temperature 4 C higher than a control cell of the same size containing a platinum cathode, even though the power input

Calculations showed that the cell with the titanium cathode was producing about one watt excess thermal power compared with the control cell [5]. This means that the 10 mg titanium cathode produced about 83,000 joules more energy than was consumed by electrolysis. This is far more energy than could be produced by any known chemical reaction. For example, the conversion of 10 mg of titanium to TiO_2 would produce about 190 joules, which is more than two orders of magnitude less than the observed output.

The titanium cathode was examined before and after electrolysis with a scanning electron microscope (SEM), and microchemical analysis was performed with an energy dispersive spectrometer (EDS). New surface features which were not present before electrolysis were analyzed and found to contain unexpected elements (S, K, Ca, V, Cr, Fe, Ni, and Zn). The most likely source of these new elements is electroplating of impurities. However, this explanation is not satisfactory for several reasons:

1. The most abundant metallic impurity in the electrolyte is Pt from the anode, and Pt deposits at a much lower potential than any of the unexpected elements which were actually observed. If electroplating was the source of the unexpected elements cited above, then Pt also should have been found.
2. The titanium cathode erodes at the rate of about one atomic layer per second. This was determined from the loss of mass which occurred during electrolysis. It would be difficult to deposit any impurity when erosion is occurring at such a high rate. This is probably why Pt was not observed.
3. The unexpected elements occur only in localized concentrations. It is not likely that such a distribution could be produced by electroplating.

During the past three years we have continued our studies of the electrolysis of heavy water with palladium and titanium cathodes. The work on palladium cathodes was characterization by SEM, EDS, and secondary ion mass spectrometry (SIMS). Multiple cell experiments were performed, using titanium cathodes, to determine what factors are important in obtaining excess heat reproducibly. Improved calorimetry was employed to increase the accuracy of heat measurements. Neutron activation analysis (NAA) and inductively coupled plasma mass spectrometry (ICPMS) were performed in addition to SEM and EDS analysis.

During this time period there were reports that radioactive elements deposited on the cathode could be rapidly transmuted into stable elements by the electrolysis of water [5]. If true, this would be a very significant discovery. A new graduate student was interested in research on this topic, and he wrote his M.S. thesis on the results of his experiments.

4. SUMMARY OF THE MOST IMPORTANT RESULTS

A. There are changes in the isotopic ratios in both palladium and titanium cathodes after electrolysis in heavy water. For example, Fig.1 (top) shows SIMS spectra taken from a Pd sample which had not been electrolyzed. The six stable isotopes occur in their expected relative ratios. After

electrolysis for six minutes in heavy water, a Pd cathode made from the same batch shows isotopic inversions of ^{108}Pd with ^{106}Pd and ^{110}Pd with ^{104}Pd, Fig.1 (bottom). After electrolyzing a Pd cathode from the same batch in light water, the SIMS spectra showed no isotopic inversions [6]. SIMS is a surface analytical technique. Our previous results showed that an isotopic inversion disappeared after sputtering about 0.3 μm from the Pd surface, indicating that the inversion occurs only at the surface[7].

Changes in the isotopic ratios for titanium after electrolysis in heavy water, obtained by ICPMS, are shown in Fig.2. This shows that the ratios of ^{50}Ti to each of the other stable isotopes is reduced after electrolysis by amounts ranging from 4 to 13%, which is an average over the bulk of the cathode [8].

We realize that our claim of changes in isotopic ratios is extraordinary. Therefore, we have preserved our samples so that our results can be checked by any independent laboratory.

B. In order to achieve more accurate calorimetric results, we acquired a Seebeck envelope calorimeter, which contains many thermocouples connected in series to measure the total heat output of a source contained in the inner cavity. This calorimeter was calibrated by using a cell with Pt electrodes and acidified light water. The calorimeter output signal varied linearly with power input to the electrolysis cell over the range of interest. Using a cell with a Pt anode, a Ti cathode, and acidified heavy water electrolyte, excess heat was observed when the calorimeter output signal significantly exceeded the calibration line at the same input power. Fig.3 shows an example of power output from a cell with a Ti cathode compared with the power input. The cell runs at constant current.

Excess heat is generated constantly after 60 minutes, and the amount gradually increases to the steady state value of about 130 mW +/- 17 mW after about 200 minutes. This cell gave statistically significant excess heat for about 82 hours, after which it was stored for future study.

C. Multiple cell experiments were used to determine if reproducible results could be achieved [9]. Seven cells were connected in series. In one of these both the anode and the cathode were platinum, and in the others the anode was Pt and the cathode was Ti. Another cell of the same type was used to monitor ambient temperature. All cells had double walls, with a sensor to measure temperature located in the air space between the walls.

High purity titanium 0.5 mm thick, in the as received (cold rolled) condition was used for the cathodes. Experiments with titanium cathodes in the as received condition yielded excess heat in four of nine

cases. In an attempt to improve reproducibility, the effect of additional cold rolling on excess heat production was studied. With the Ti thickness reduced from 0.5 to 0.4 mm (20% reduction), it was found that 12 of 21 gave statistically significant excess heat. The magnitude of this excess thermal power varied from 0.1 to 0.5 watt, and the duration was about 20 hours. Failure occurred due to erosion, which is probably caused by high internal stresses produced when TiD_2 forms on the cathode surface. At 50% reduction in thickness, four of 11 cells gave excess heat, and the maximum excess thermal power was about 0.2 watt. Statistically significant excess heat was obtained in only one of nine experiments using cathodes which had been cold rolled to more than 50% reduction in thickness. Fig. 4 summarizes the data on excess thermal power as a function of reduction in thickness by cold rolling.

D. Professor Emeritus H. Kozima, Physics Dept., Shizuoka University, Japan, has developed a theory which is based on thermal neutrons from the ambient background which are trapped in the surface atoms of certain solids. In the excited state, these neutrons may hop to other nuclei and catalyze fusion reactions. Since he joined our laboratory in Sept. 2000, he has had four papers concerning refinements of his theory accepted for publication.

5. LIST OF ALL PUBLICATIONS AND TECHNICAL REPORTS

1. D. S. Silver and J. Dash, "Surface Studies of Palladium after Interaction with Hydrogen Isotopes", Proc. 7th Int. Conf. on Cold Fusion, pp. 351-355, Vancouver, B.C., April 1998 (Published by Eneco, Salt Lake City, 1998).

2. M.J. Klopfenstein and J. Dash, "Thermal Imaging during Electrolysis of Heavy Water with a Titanium Cathode", ibid, pp. 98-102.

3. D. Silver and J. Dash, " SEM and EDS Characterization of Palladium Cathodes after Electrolysis in Light and Heavy Water", Proc. Conf. on Microscopy and Microanalysis, pp. 596, 597, Portland, OR, Aug. 1999 (Springer, 1999).

4. J. Warner and J. Dash, "SEM and EDS Characterization of Titanium Cathodes before and after Electrolysis in Heavy Water", ibid, pp. 598, 599.

5. J. Warner and J. Dash,"Heat produced during the Electrolysis of D_2O with Titanium Cathodes", Proc. 8th Int. Conf. on Cold Fusion, pp. 161-167, Italy, May 2000 (Published by Italian Physical Society, 2000).

6. G. Goddard, J. Dash, and S. Frantz, "Characterization of Uranium Codeposited with Hydrogen on Nickel Cathodes", Trans. American Nuclear Society 83, 376-378 (2000).

7. H. Kozima, J. Warner, G. Goddard, and J. Dash, " Reality of the Super Nuclear Reaction in Metal Hydrides and Deuterides – Verification by Numerical Calculations for PdH(D)", accepted for publication in Fusion Technology.

8. H. Kozima, "Neutron Bands in Metal Hydrides – Effects of Occluded Hydrogen on Nuclear Reactions in Solids", accepted for publication in Fusion Technology.

9. H. Kozima, K. Yoshimoto, H. Kudoh, M. Fujii, and M. Ohta, "Analysis of Zn and Excess Heat Generation in Pd/H_{2}(D_{2}) System by the TNCF Model, accepted for publication in J. New Energy.

10. H. Kozima, M. Ohta, M. Fujii, K. Arai, and H. Kudoh, " Possible Explanation of ^{4}He Production in a Pd/D_{2} System by the TNCF Model", accepted for publication in Fusion Technology.

11. D. Silver, "Surface Studies of Metals after Interaction with Hydrogen Isotopes", Ph.D. Dissertation, Portland State University (1998).

12. J. Warner, "Observation of Heat Produced and Chemical Changes Caused by Electrolysis of Titanium with Heavy Water", M.S. Thesis, Portland state University (1998).

13. G. Goddard, "Characterization of Uranium Codeposited with Hydrogen on Nickel Cathodes", M.S. Thesis, Portland State University (2001).

6. LIST OF PARTICIPATING SCIENTIFIC PERSONNEL

1. David Silver completed his doctoral dissertation on this research in Jan. 1998. He then continued the research as a postdoctoral research assistant until May 1998.

2. Michiaki Ishimura, Ph.D., served as postdoctoral research assistant from Dec. 1998 until June 1998.

3. William Milmoe, B.S., served as research assistant from Sept. to Dec. 1997.

4. William Brown served as research assistant during July and Aug. 1998 and during July and Aug. 2000. He is an undergraduate student in physics.

5. Grant Noble, M.S., served as research assistant from Dec. 1997 until June 1998.
6. Michael Klopfenstein, B.S., served as research assistant from Oct. 1997 until June 1998.

7. Jon Warner completed his M.S. thesis on this research in 1998. He is currently writing his Ph.D. dissertation on results obtained since he completed the M.S. degree.

8. Gregory Goddard completed his M.S. thesis on this research in Jan. 2001.

9. Conrado Cano, B.S., has served as research assistant since Sept. 2000. He intends to use this research for his M.S. thesis.

10. Hideo Kozima, Visiting Professor of Physics, has served as our consultant on theory since Sept. 2000.

7. BIBLIOGRAPHY

1. F. Close, "Too Hot to Handle" (Princeton University Press, 1991), pp. 19-21.

2. M. Fleischmann and S. Pons, "Electrolytically Induced Nuclear Fusion of Deuterium", J. Electroanalytical Chem. 261, 301(1989).

3. E. Storms, Private Communication.

4. F. Scaramuzzi, Proc. 8th Int. Conf. on Cold Fusion, pp. XVII, XVIII, Italy, May 2000 (Published by Italian Physical Society, 2000).

5. G. Miley, "Scientific Feasibility Study of Low-Energy Nuclear Reactions for Nuclear Waste Amelioration", Proposal No. 99-0222, submitted to DOE Nuclear Energy Research Initiative, 1999.

6. D. S. Silver, "Surface Studies of Metals after Interaction with Hydrogen Isotopes", Ph.D. dissertation, Portland State University, 1998, pp. 79-81.

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8. M. F. Klopfenstein and J. Dash, "Thermal Imaging during Electrolysis of Heavy Water with a Ti Cathode", Proc. 7th Int. Conf. on Cold Fusion, p. 98, Vancouver, B.C., April 1998 (Published by Eneco, Salt Lake City, 1998).

9. J. Warner, "Observation of Heat Produced and Chemical Changes Caused by Electrolysis of Titanium with Heavy Water", M.S. Thesis, Portland State University (1998).

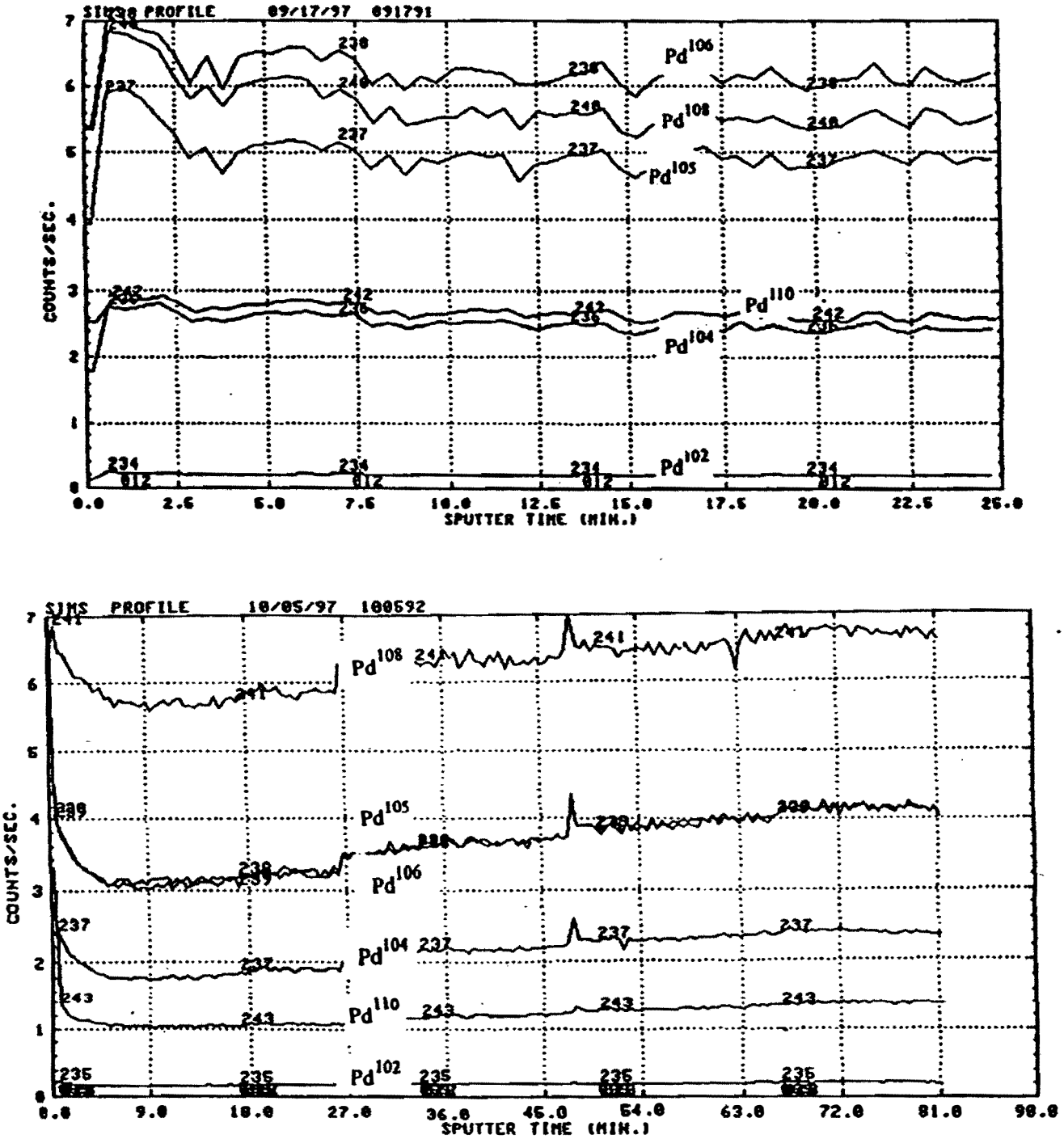
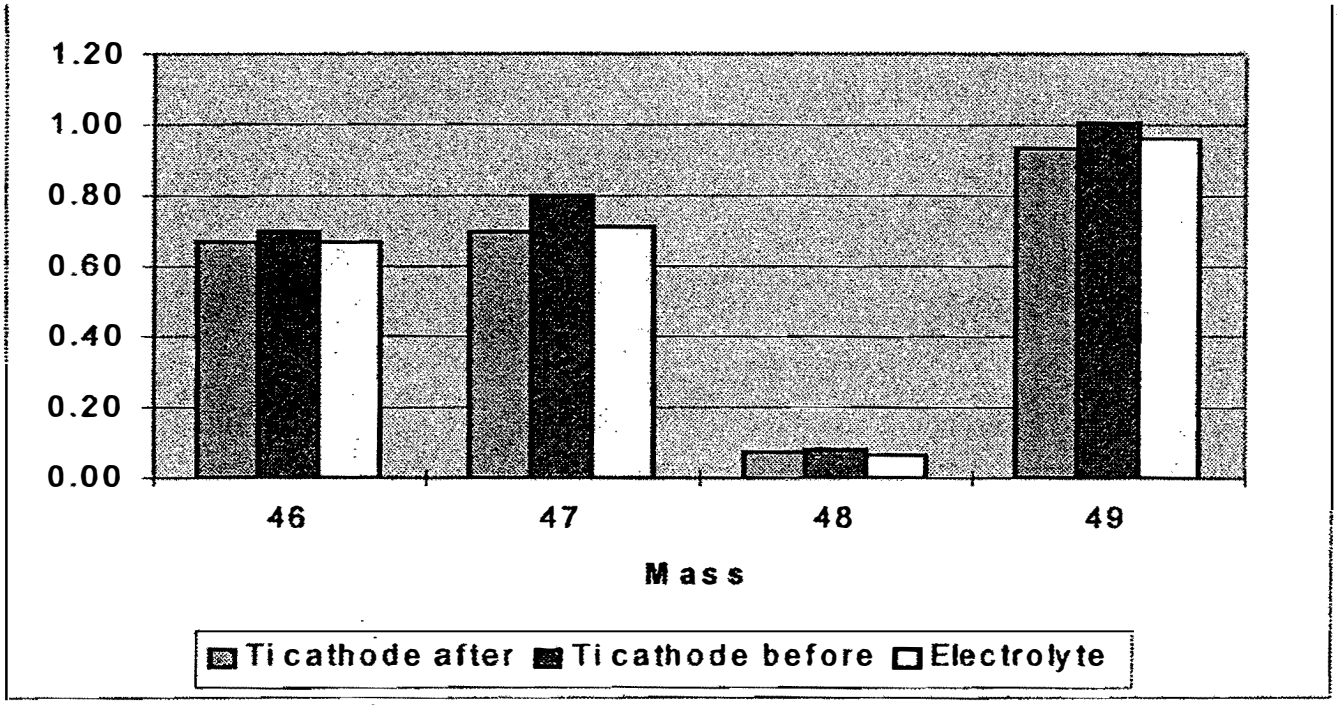


Figure 1. SIMS profile of the six palladium isotopes for a region of the palladium sample not electrolyzed (top), and a region of the six minute heavy water electrolyzed sample (bottom). Note the isotopic inversions of Pd^{108} with Pd^{106}, and Pd^{110} with Pd^{104}, for the latter sample.



	Ti cathode before	Ti cathode after	Electrolyte after electrolysis
Ti50/Ti46	0.6960	0.6683	0.6593
Ti50/Ti47	0.8027	0.6978	0.7022
Ti50/Ti48	0.0785	0.0727	0.0685
Ti50/Ti49	1.0111	0.9358	0.9476

Fig. 2. Effect of electrolysis in heavy water with a titanium cathode on isotopic ratios.

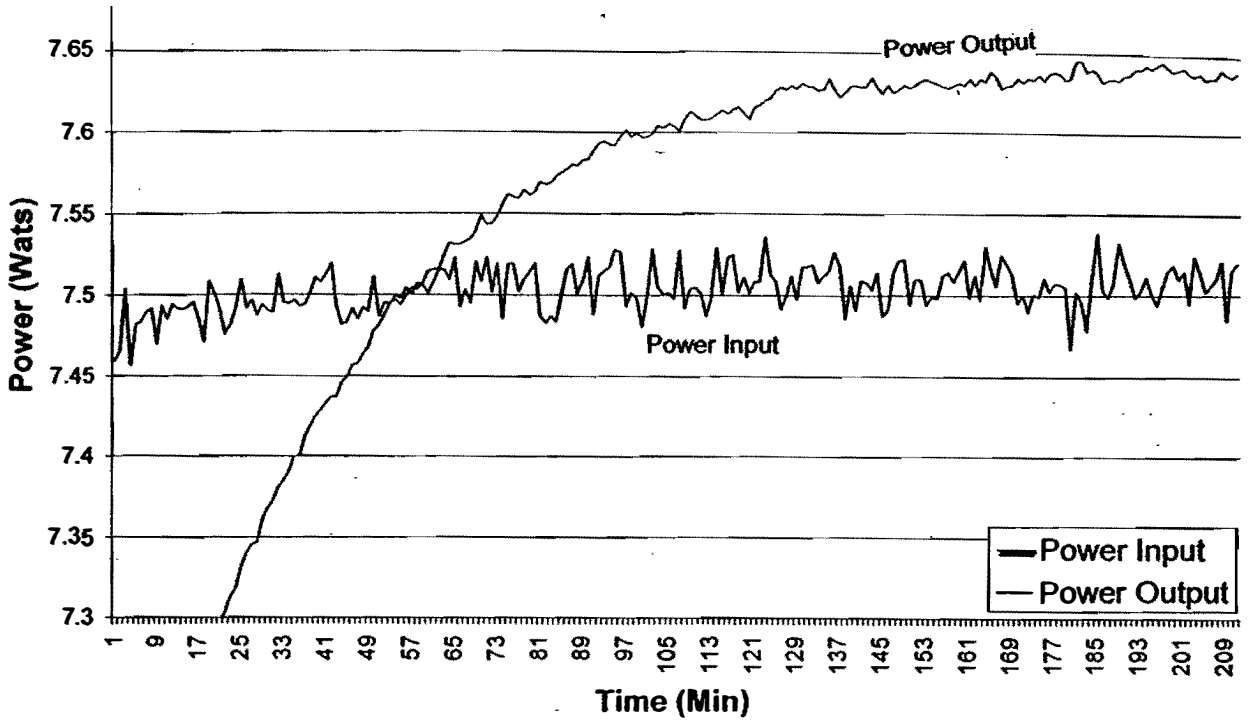


Fig. 3. Seebeck envelope calorimeter results showing excess heat produced by electrolysis of heavy water with a titanium cathode. The standard deviation of the thermal power output is 17 mW.

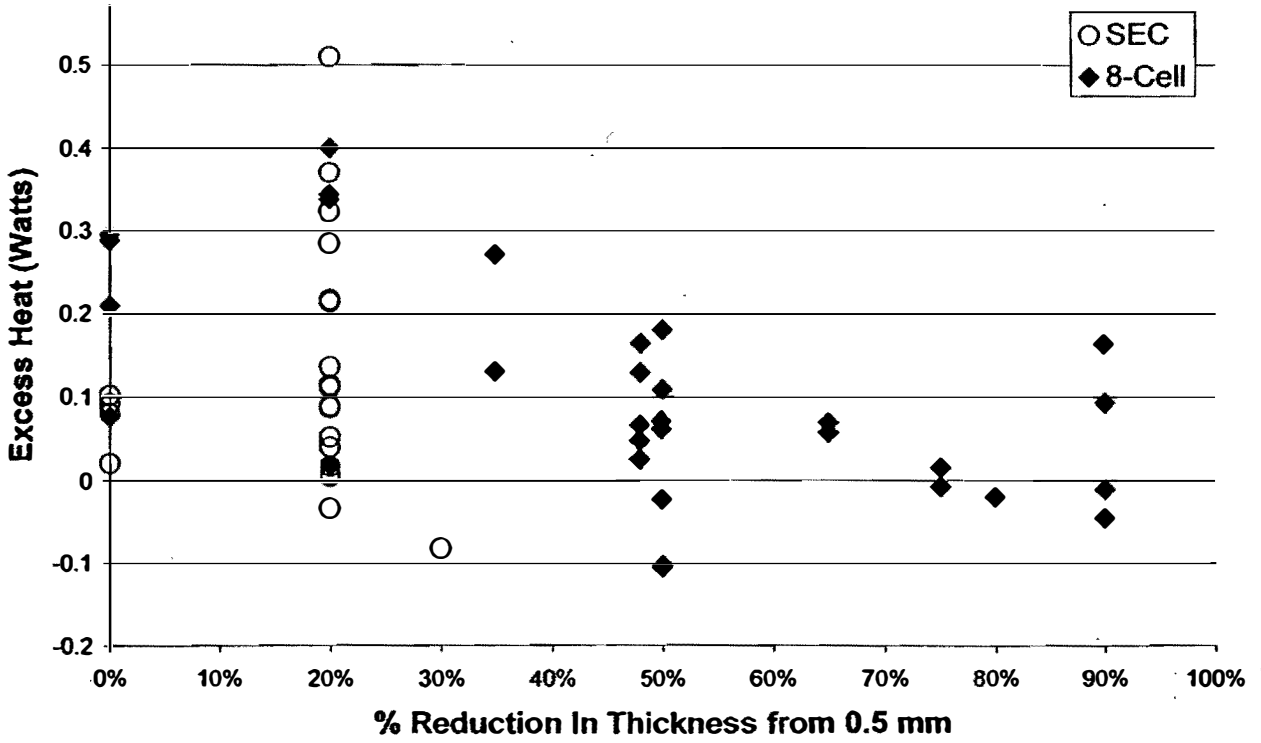


Fig. 4. Excess thermal power as a function of reduction in thickness of Ti cathodes by cold rolling,