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Second Technical Report

1st December 1992 - 31st January 1993

Principal Investigator: Dr Robin John Neat

LITHIUM POLYMER BATTERIES FOR SPACE POWER APPLICATIONS

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AEA Industrial Technology
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Executive Summary

This report documents the results of the work performed by AEA Technology under contract number F61708-92-C0039 over the period 1st December 1992 to 31st January 1993 and represents deliverable item 003.

The US Air Force is undertaking a number of projects aimed at developing new and improved secondary battery systems for the provision of baseload power in satellites.

This project has the objectives of evaluating the Lithium Polymer Battery (LPB), developed by AEA Technology, against a GEO satellite duty cycle, and subsequently developing the battery system with a view to improving on its performance.

A Mark I LPB cell has been defined as a lithium metal anode, lithium-ion conducting polymer electrolyte (PEO_{12}:$LiClO_4$) and a composite cathode based on V_6O_{13}. The chosen operating temperature is 120°C.

The space power duty cycle employed for the evaluation is a fixed discharge time of 72 minutes and a total charging time of 10 hours.

In this report evaluation results are presented covering the variables of depth of discharge (DoD) and discharge current density for cells with cathode capacities of 2.5 mA h cm^{-2} and 1.15 mA h cm^{-2} using a 'mixed' mode charge. LPB cells cycled at 20% DoD have delivered ~ 90 cycles (for a 2.5 mA h cm^{-2} capacity cathode) and over 110 cycles for a 'thin' cathode (1.15 mA h cm^{-2}).

A build-up in cell impedance has been identified with a performance limitation of the Mark I LPB.

The use of a 10 hour constant voltage charge appears to be detrimental to the long term cycling of the Mark I LPB cell.

Several of the evaluation experiments are currently being repeated and results will be included in the third technical report.

CONTENTS

1. Objectives of Research and Statement of Work

1.1 Introduction and Objectives

The provision of baseload power for space vehicles remains a major technological challenge. The most common solution, used exclusively in satellites, is the combination of photo-voltaic cells and secondary batteries. The performance of current state-of-the-art secondary batteries is poor; they are bulky and heavy.

It is clear that an improvement in secondary battery technology would provide a significant payoff in terms of performance, survivability and affordability. Currently the majority of satellites fly with Ni-Cd batteries. These suffer from being both heavy and bulky (energy density 25-30 W h kg^{-1} and 50-60 W h $litre^{-1}$).

The US Air Force is undertaking a number of projects aimed at developing new and improved secondary batteries for satellite power applications. This report documents work carried out by AEA Technology under contract F61708-92-C0039 which has the object of evaluating the AEA-developed Lithium Polymer Battery against the GEO satellite duty cycle, and subsequently undertaking a development program to improve the battery's performance.

The Lithium Polymer Battery (LPB) is an all solid-state system which combines a lithium ion conducting polymer electrolyte with two lithium ion reversible electrodes. The polymer electrolyte also acts as a mechanical separator for the two electrodes. In most cases the anode is a metallic lithium foil. The cathode is typically a reversible intercalation compound such as V_6O_{13} or TiS_2 in the form of a composite backed by a metal foil current collector. LPB cells are fabricated by lamination of the three component layers; lithium foil, polymer electrolyte and composite cathode. The LPB concept has a number of advantages based on the all-solid-state construction; high energy density, high power density, good shelf life and excellent safety characteristics.

This project is the first phase of a three-phase program designed with the objective of translating the promise of the LPB technology into prototype battery units.

1.2 Statement of Work

The statement of work (SOW) specifies the requirements for AEA Technology to evaluate the lithium polymer battery against the GEO satellite duty cycle, and subsequently undertaking a development program to improve the battery's performance. AEA Technology is performing three work packages in order to achieve the objectives of the program. These are illustrated in the work breakdown structure in Figure 1.

Work package 1 will be performed from the program start for nine (9) months, although the electrical testing phase may be allowed to continue if results are promising. Work package 2 will run from month nine (9) to month twenty three (23) and is designed to follow on from work package 1. Work package 3 will be performed from month three (3) to month (21) and is designed to run concurrently with work packages 1 and 2.

Work Package 1.

AEA Technology will draw up a specification for the Mark I LPB which will be the most advanced version of the V_6O_{13}-based LPB at the start time of the contract. AEA Technology will use its facilities in the Lithium Battery Section of the Applied Electrochemistry Department to test the cycle performance of the Mark I LPB cell against the following variables: a range of depths of discharges (20%, 40%, 60%, 80% and 100%) of the cell's theoretical capacity; a range of cathode capacities (1.0, 1.5, 2.0, 2.5 and 3.0 mA h cm^{-2}); a range of discharge current densities (0.2, 0.5, 1.0 and 2.0 mA cm^{-2}), and different charging techniques (constant current, constant potential and mixed mode). The results of the testing will be presented as a benchmark performance and included in a technical report. The length of cycle testing will be constrained by the testing equipment available.

Work Package 2.

AEA Technology will study the cycling performance of the Mark I LPB in which V_6O_{13} is replaced as the active cathode material by: $LiMn_2O_4$ and related spinels (e.g. $Li_2Mn_4O_9$); MnO_2; V_2O_5 and TiO_2. Following initial evaluation of the compounds listed above, AEA Technology will attempt to optimise the Mark I cell configuration and/or composition to yield cycle performance which is superior to that of the V_6O_{13}-based Mark I cell. The testing and evaluation of these alternative cathode material cells will be restricted to combinations of parameters (i.e. depth of discharge, current density and cathode capacity) for which it is possible to predict superior performance.

Work Package 3.

AEA Technology will apply several investigative analytical techniques which it has developed, or is in the process of developing, to identify LPB performance limiting phenomena. AEA Technology will build LPB cells which contain a reference terminal and use three terminal a.c. impedance analysis to monitor changes in the overall cell impedance, the lithium/electrolyte impedance and the electrolyte/cathode impedance during cycling. Using the resulting data AEA Technology will attempt to modify the LPB cell configuration and/or composition to remove or reduce any identified performance limiting factors. AEA Technology will section LPB after testing, and use a scanning electron microscope to examine the physical condition of the cell. EDX analysis will also be used to examine the chemical composition of the cell layers. Using the resulting data AEA Technology will attempt to modify the LPB cell configuration and/or composition to remove or reduce any identified performance limiting factors. AEA Technology will use a post test X-ray diffraction technique to examine the crystal structure of the active cathode material. Using the resulting data AEA Technology will attempt to modify the LPB cell configuration and/or composition to remove or reduce any identified performance limiting factors.

1.3 The Current Report

This report is the second technical report which documents the work performed over the period 1st December 1992 to 31st January 1993 and is deliverable item 003. The report covers the second two months of effort under Work Package 1, and preliminary experiments in Work Package 3.

2. Status of Research Effort - Progress against Objectives

2.1 Status of Work in Work Package 1

2.1.1 Performance Update on Mark I LPB cells described in the First Technical Report

The Mark I LPB cell has been defined as a lithium metal anode, a lithium-ion conducting polymer electrolyte (PEO_{12}:$LiClO_4$) and a composite cathode based on V_6O_{13}. A detailed description of the Mark I LPB and the experimental procedures employed in the fabrication of tests cells can be found in the First Technical Report (AEA-InTec-1161).

The initial space power duty cycle agreed for this evaluation project consists of a fixed discharge time of 72 minutes and a total charging time of 10 hours. In order to undertake Task 1.1 a cathode capacity of 2.5 mA h cm^{-2} was initially chosen. This gives a theoretical capacity of 100 mA h for the 40 cm^2 Mark I LPB cell. The discharge current has been varied to correspond to DoDs of 20%, 40%, 60%, 80% and 100% as indicated in Table I below.

Table I

DoD	Discharge current / mA	Current density / mA / cm^2	Discharge rate
20%	16.7	0.42	C/6
40%	33.3	0.83	C/3
60%	50.0	1.25	C/2
80%	66.7	1.67	C/1.5
100%	83.3	2.01	C/1.2

In the first sequence of experiments a 'mixed' mode of charging has been selected.This consists of a constant current charge to 3.25 V followed by a potentiostatic hold at 3.25 V such that the total charge time is 10 hours. A lower voltage limit of 1.5 V has been used to determine the end of life in all cases.

The cycle performance data is presented in two forms; discharge energy versus cycle number and end of discharge voltage versus cycle number.

20% Depth of Discharge

The updated cycling performance data for the group of three Mark I LPB cells (P003, P004 and P005) discharged to 20% depth of discharge are given in Figure 2. After 85 cycles there was approximately a 5% loss in the discharge energy from the initial value, while the end of discharge voltage has decreased by ~ 0.13 V. A comparison between the 50th and 85th discharge cycles for cell P003, Figure 3, illustrates that the discharge process continues to occur on two voltage plateaux. Unfortunately a power supply problem led to a test rig crash in which the computer lost control of the cycling potentiostats. As a consequence the cells were subjected to an extended period (~ 60 hours) of charging, and subsequently failed to sustain the discharge current for the full 72 minutes. A.c. impedances measurements taken on the three cells at that time, and shown in Figure 4, indicated large increases in cell resistance over the initial cell impedance values ($< 1\ \Omega$). A repeat of the 20% DoD experiment is currently in progress.

40% Depth of Discharge

Figure 5 shows the updated cycling performance data for 3 Mark I LPB cells (P007, P009 and P010) discharged to 40% depth of discharge. Although the energy loss for LPB cells P009 and P010 over the first 40 cycles was only 3%, both cells exhibited a more rapid decline over the next 20 cycles during which there was a further 5% loss of energy. LPB cell P009 developed a short after 68 cycles and was removed from test.The testing of P010 was terminated after approximately 70 cycles following the rig crash discussed above.

60% Depth of Discharge

The updated cycling performance data for a group of Mark I LPB cells discharged to 60% depth of discharge are given in Figure 6. Although the initial performance of P012 and P013 is very encouraging (60% utilisation at the C/2 discharge rate), with less than a 1% reduction in energy output during the first 27 cycles, the end of discharge voltage decreases steadily over this period. After 28 cycles both cells failed to sustain the discharge current for the full 72 minutes before the cell voltage fell below 1.5 V. A comparison between the final and initial a.c. impedance data for P012 and P013, Figures 7 and 8, indicates a relatively small increase in internal resistance. The poorer performance of P011 compared to P012 and P013 further illustrates the problem of cell-to-cell irreproducibility which is inherent in the hand construction methods currently employed.

Figure 9 illustrates an updated direct comparison between the cycling performance data for

20% (P003), 40% (P010) and 60% (P012) depth of discharge. For the Mark I LPB with a cathode capacity of 2.5 mA h cm^{-2}, cycling under the specified duty cycle with a 'mixed' method of charging, only the 20% DoD regime can be considered a possible option for the provision of extended cycling, and remains under investigation.

2.1.2 Performance with Constant Voltage Charging

In order to undertake Task 1.4 two alternative methods of charging, constant voltage and constant current, are being compared to the 'mixed' mode described above. The cycling performance data for two Mark I LPB cells with capacity 2.5 mA h cm^{-2} discharged to 20% DoD and charged with a 10 hour constant voltage charge at 3.25 V are shown in Figure 10. LPB cell P038 developed a short after 12 cycles. Although the energy loss of LPB cell P037 was less than 1.5% over the first 20 cycles it failed after 37 cycles. A.c. impedance measurements, Figure 11, again indicate a significant increase in cell resistance at the end of cycle life. A comparison with the cycling performance of Mark I LPB cells under 'mixed' mode charge, cells P003, P004 and P005 described above (see Figure 2), indicates constant voltage charging appears to reduce cycle life. The decrease in cycle life may be attributed in part to the large initial currents that flow at the start of a constant voltage charge, typically of the order of 1 A (25 mA cm^{-2}), that possibly result in disruption of the electrode interfaces. For a 40% DoD employing constant voltage charging, Figure 12, cell failure occurs within the first 15 cycles. No evidence has been obtained to suggest the use of constant voltage cycling will lead to an improvement in cycling performance compared to the 'mixed' mode of charging. The constant current charging mode is being evaluated at the present time and will be reported in a subsequent technical report.

2.1.3 Performance with Lower Cathode Capacity

The investigation of the effect of cathode capacity and current density on cycle life, as described in Tasks 1.2 and 1.3 respectively, has been initiated by the fabrication of a 'thin' composite cathode (i.e. capacity 1.15 mA h cm^{-2}). This was achieved by doctor blade casting the standard Mark I cathode mix, as described in the First Technical Report, using a blade gap of 0.8 mm. The resulting cathode coating had the same composition as the original 2.5 mA h cm^{-2} capacity cathode. The discharge current has been varied to correspond to DoDs of 20%, 40%, 60%, 80% and 100% as indicated in Table II. It should be noted that although the discharge rate for a Mark I LPB cell containing the 'thin' cathode cycling with a particular DoD is the same as for the 2.5 mA h cm^{-2} cathode (given in Table I) the current density is lower. Cycling performance results for 20% and 40% DoD with a 'mixed' mode charge are reported here.

Table II

DoD	Discharge current / mA	Current density / mA / cm^2	Discharge rate
20%	7.7	0.19	C/6
40%	15.3	0.38	C/3
60%	23.0	0.58	C/2
80%	30.7	0.77	C/1.5
100%	38.3	0.96	C/1.2

20% Depth of Discharge

Figure 13 shows the cycling performance data for a group of three Mark I LPB cells with a 'thin' cathode (capacity 1.15 mA h cm^{-2}) discharged to 20% depth of discharge. All three cells exhibited an initial energy loss, although the magnitude varied from cell to cell. For example, over the first 40 cycles the energy loss ranged from less than 1% for P044 to approximately 4% for P041. The variability in cycling behaviour is further illustrated by the longer term cycling; LPB cells P043 and P044 showed a gradual decline in energy output from cycles 40 to 115 (~5% and 8% respectively) whereas for P041 the energy loss was only ~ 0.5%. As expected the energy output from these cells is proportionally lower than that obtained from those LPB cells (see Figure 2) containing the 2.5 mA h cm^{-2} cathode cycling under the same conditions. Unfortunately, after 115 cycles the cycling rig crash resulted in LPB cells P041, P043 and P044 being overdischarged to a such an extent that they were unable to perform any further discharge cycles. At this point in the cycle life the energy output from both P043 and P044 was decreasing reasonably rapidly, as illustrated in Figure 13, but that of P041 was essentially constant. As it is unclear why the behaviour of P041 differed from that of P043 and P044, and whether it could maintain the observed level of energy output it is proposed to repeat this experiment. Given the poorer cycling performance of P043 and P044 compared to P003, P004 and P005 one may tentatively conclude there is little benefit in using a lower capacity cathode for 20% DoD cycling.

40% Depth of Discharge

The cycling performance data for a group of three Mark I LPB cells with a 'thin' cathode (capacity 1.15 mA h cm^{-2}) discharged to 40% depth of discharge are given in Figure 14. An initial energy loss 2% to 4% was observed over the first 5 cycles. LPB cell P045 developed a cell short after 34 cycles resulting in failure. LPB cells P047 and P048 continued to cycle with constant energy output until cycles 50 and 80 respectively, before entering a region in which there was a steady decline in both energy output and end of discharge voltage that ultimately resulted in cell failure. Figure 15 illustrates the observed increase in cell resistance in the cycled P047 LPB cell. Although the cycle life of these cells is greater than that measured for the 2.5 mA h cm^{-2} cathode cycling under the same 40% DoD conditions, see Figure 5, the overall cycling performance appears to be limited to a maximum of ~ 100 cycles.

2.2 Status of Work in Work Package 3

The purpose of Work Package 3 is to identify and understand the performance limitations of the Mark I LPB, or subsequent improvements, thereby enabling a reduction or removal of any such limiting factors. This Work Package will employ three analytical techniques; post-test SEM/EDX analysis, three terminal a.c. impedance and post test X-ray diffraction studies, and is designed to run in parallel with the LPB cell cycling experiments performed in Work Packages 1 and 2.

Scanning electron microscopy (SEM) is a versatile technique to probe near surface phenomena. However, until recently this was confined to materials which were not beam sensitive. The introduction of cryogenic techniques has ensured sensitive materials, such as oils, emulsions, plastics and polymers can be stabilised by lowering their temperature, so enabling information to be obtained about their physical and chemical characteristics. Post test SEM analysis of the LPB poses technical problems because it requires an 'in situ' fracture to expose the laminated cell structure without leading to delamination of the individual layers. A cryogenic sectioning technique has been developed by AEA Technology which can be used to fracture a LPB cell within an SEM chamber. These sections can then be examined visually to investigate any possible morphological changes within the cell during cycling. Any interesting features observed can then be analysed using the EDX attachment which provides elemental analysis data. The present project will be carried out using an S570 Hitachi scanning electron microscope equipped with a Cryotrans CT1000 system which includes a cryogenic specimen transfer unit. The microscope was also fitted with an EDX energy dispersive X-ray system and a back scattered electron detector.

Task 3.2 has been initiated by examining an uncycled Mark I LPB cell, P027, with a cathode capacity of 2.5 mA h cm^{-2}. Figure 16 shows a scanning electron micrograph of the cross-section of P027. The individual component layers can be easily distinguished. This will provide a control against which the various features observed in the micrographs of cycled cells will be compared.

A common feature of the LPB cells removed from cycling has been the observed increase in the cell resistance compared to the initial value. We have obtained a number of examples of this characteristic (several of which are described in the previous section), such that it can be identified with a cycle performance limitation. In the following months we will now be employing three terminal a.c. impedance measurements to identify the possible origins of this impedance build-up during cycle life (e.g is it related to the electrolyte, the anode/electrolyte interface etc.).

2.3 Discussion and Conclusions

For a Mark I LPB, tested under a space power duty cycle consisting of a 72 minute discharge and 10 hour mixed mode charge, cells cycled at 20% DoD have delivered ~ 90 cycles (for a cathode capacity of 2.5 mA h cm^{-2}) and over 110 cycles for a 'thin' cathode (1.15 mA h cm^{-2}). These cycle numbers do not represent the end of cycle life, but rather the point at which the tests were terminated due to a cycling rig crash. These experiments are currently being repeated. The present space power duty cycle only permits 15 discharge/charge cycles to be completed per week. However, by reducing the charging time to a total of 5 hours this can be nearly doubled to 27 cycles per week enabling the longer term cycling performance to be investigated more quickly. This alternative testing regime could be adopted for those DoD and cathode capacity cycling conditions that had demonstrated the most promising performance under the present standard duty cycle.

The initial cycling performance at 60% DoD, corresponding to a high discharge rate (C/2) with a good utilisation of the active cathode material, is very encouraging with less than a 1% loss in energy output during the first 27 cycles.

A build-up of cell impedance has been identified with a performance limitation of the Mark I LPB. At this time it is unclear whether the origin of this impedance build-up is: i) occurring in the electrolyte layer, ii) the result of an interfacial resistance on one of the electrode surfaces, or iii) occurring within the bulk of the composite cathode. A structural transformation of the $V_6 0_{13}$ cathode material during cycling could lead to a decrease in electronic conductivity thereby increasing the overall resistance of the composite.The successful elimination of the cause of this impedance increase should lead to a significant improvement in the cycle performance at all depths of discharge, such that the dramatic failure observed for example at 60% DoD is removed.

The use of a 10 hour constant voltage charge appears to be detrimental to the long term cycling of the Mark I LPB cell. No cycling performance improvement has been obtained to date by the use of a 'thin' composite cathode (capacity 1.15 mA h cm^{-2}) on discharge to 40% DoD.

In preparation for post test SEM/EDX studies an uncycled Mark I LPB has been sectioned and will allow correlation of visual information before and after failure.

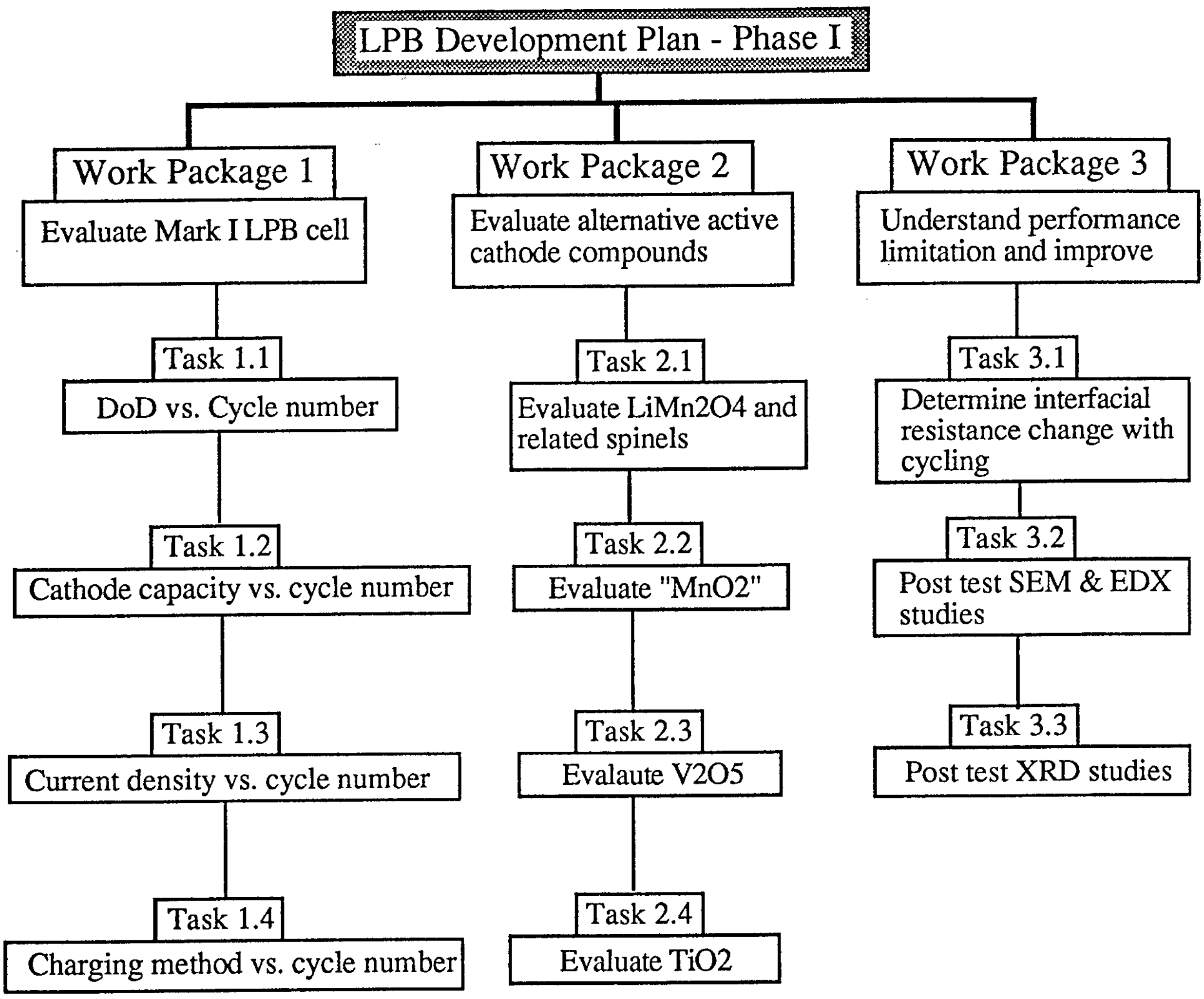


Figure 1 *Work Breakdown Structure.*

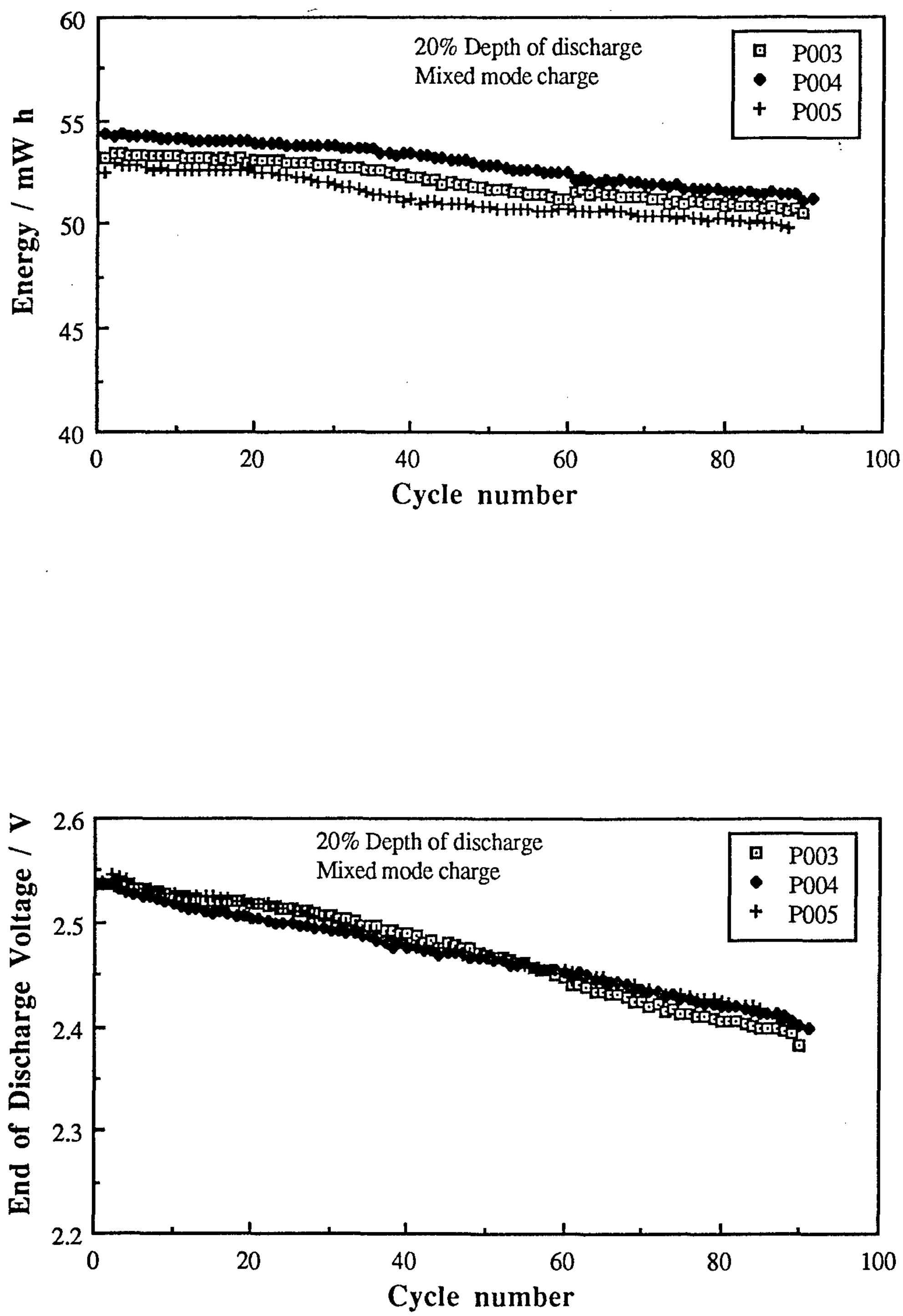
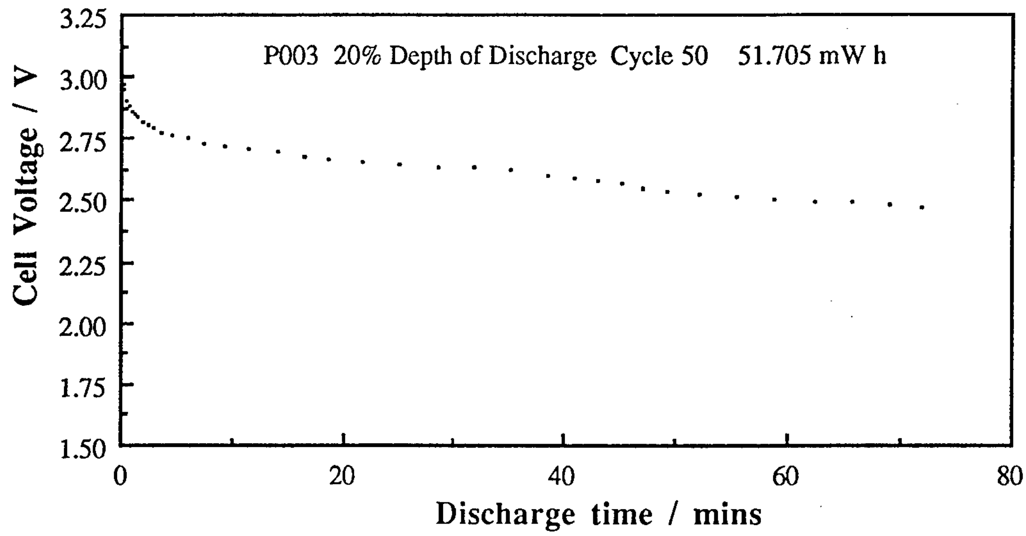


Figure 2 *Cycle performance data for the Mark I LPB cell at 20% depth of discharge.*



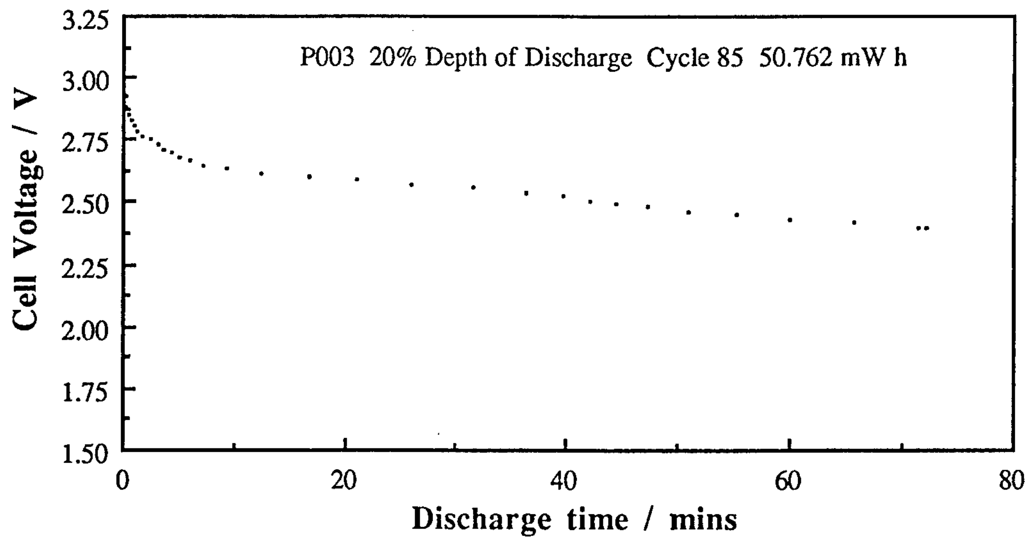


Figure 3 *50^{th} and 85^{th} discharge cycles for a LPB cell at 20% depth of discharge.*

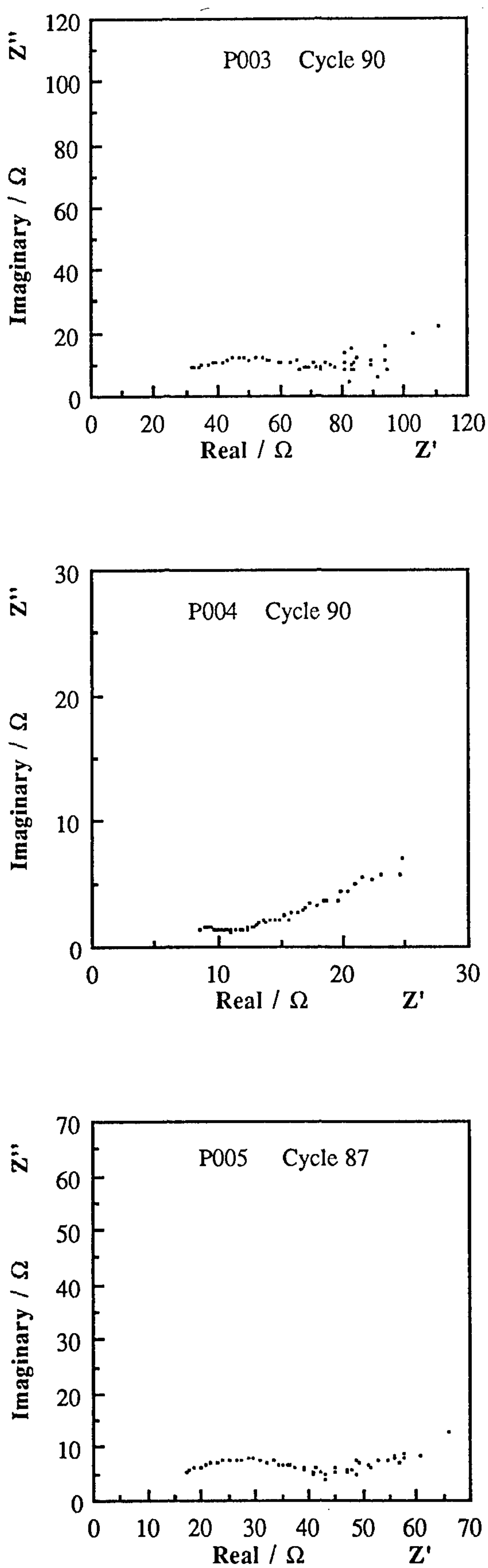
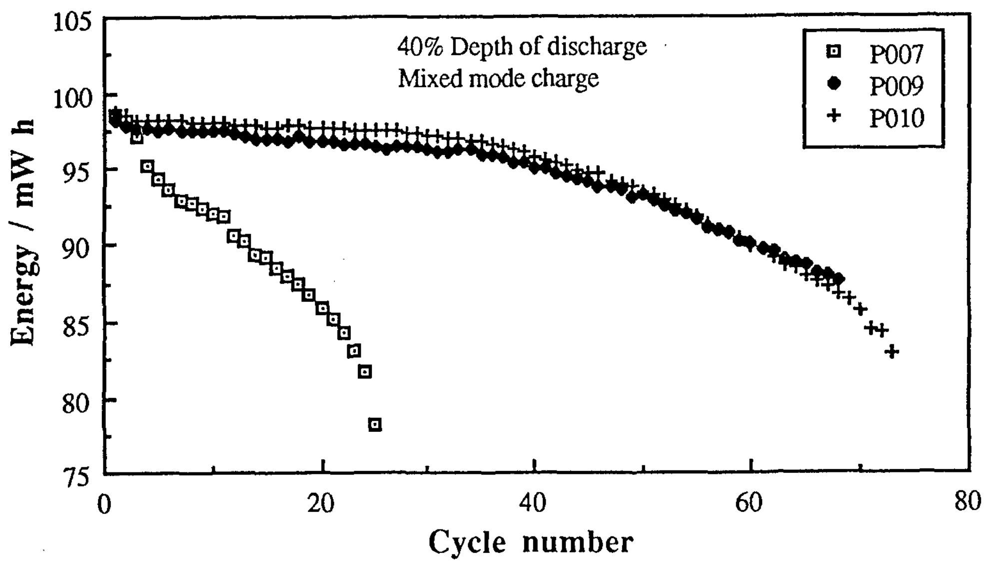


Figure 4 *A.c. impedance data for cycled LPB cells at 20% depth of discharge.*



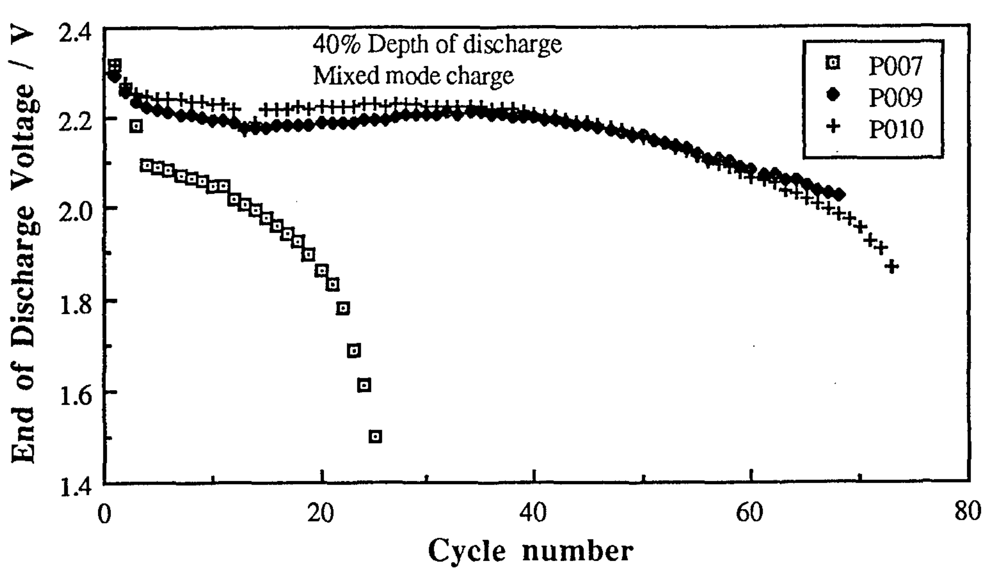
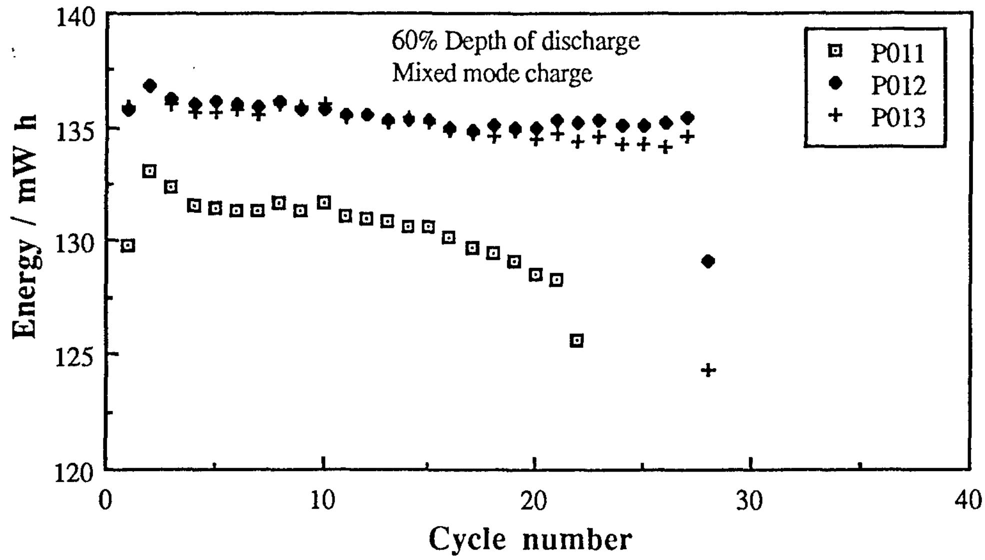


Figure 5 *Cycle performance data for the Mark I LPB cell at 40% depth of discharge.*



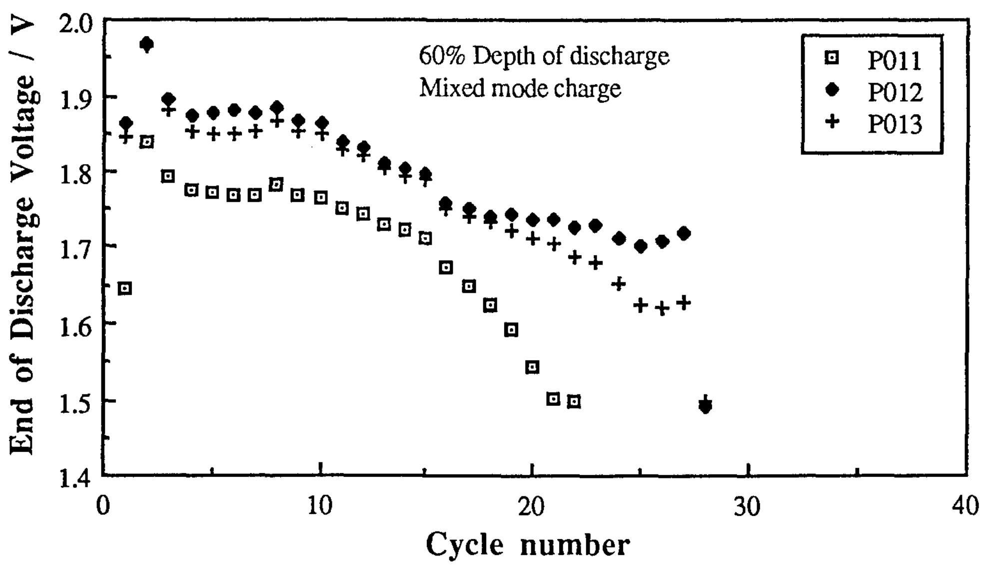
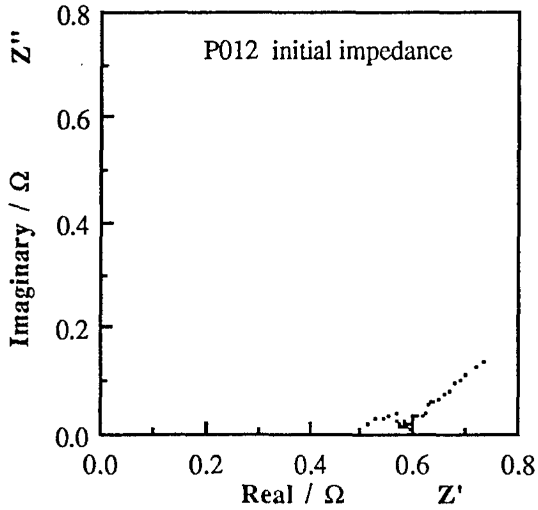


Figure 6 *Cycle performance data for the Mark I LPB at 60% depth of discharge.*



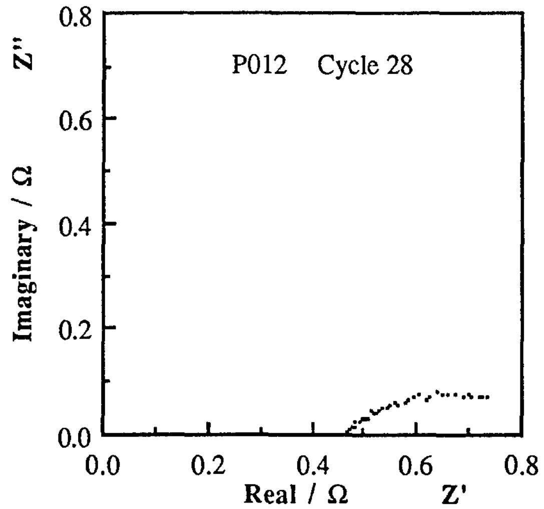
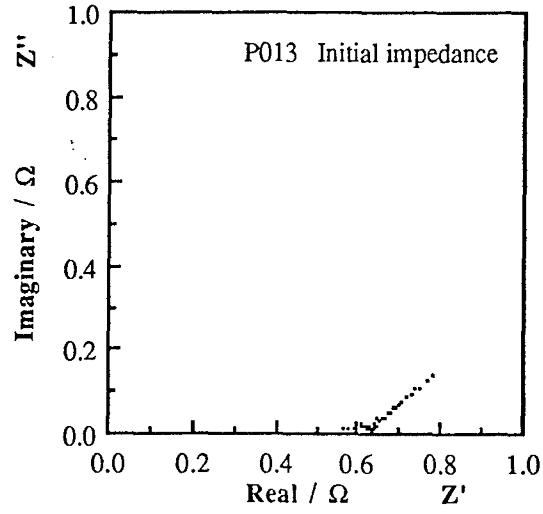


Figure 7 *A.c. impedance data for P012 cycled at 60% depth of discharge.*



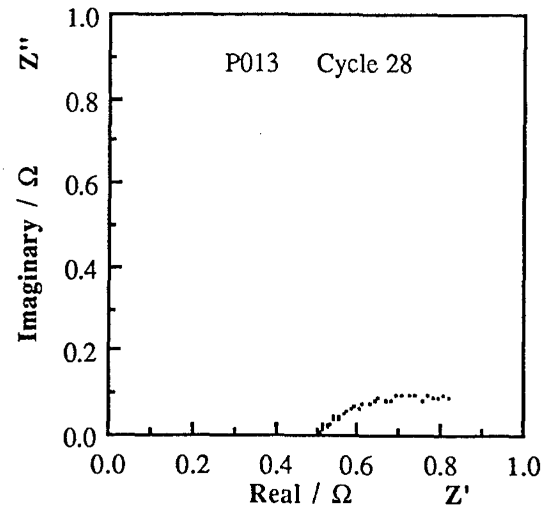
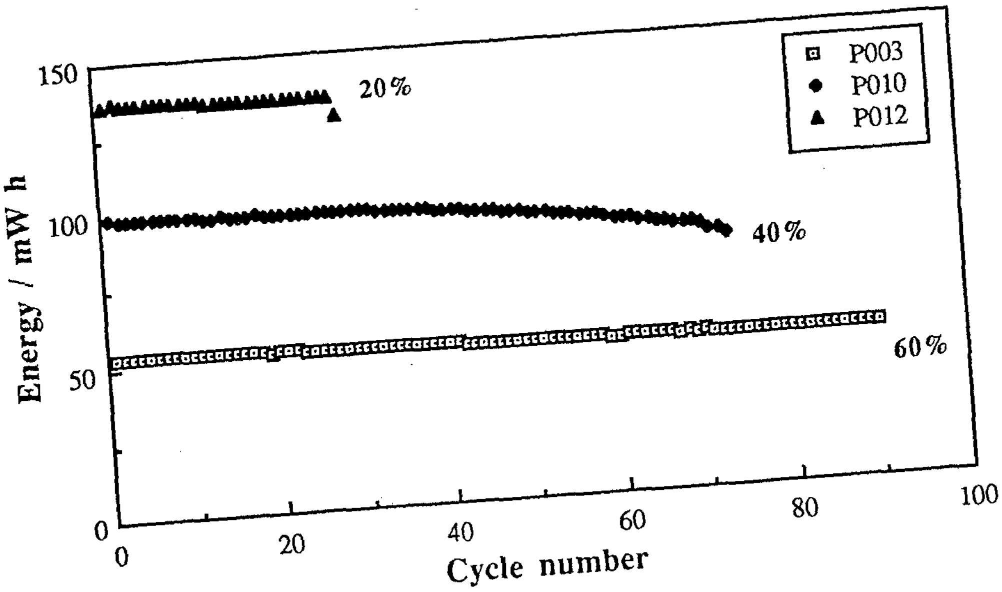


Figure 8 *A.c. impedance data for P013 cycled at 60% depth of discharge.*



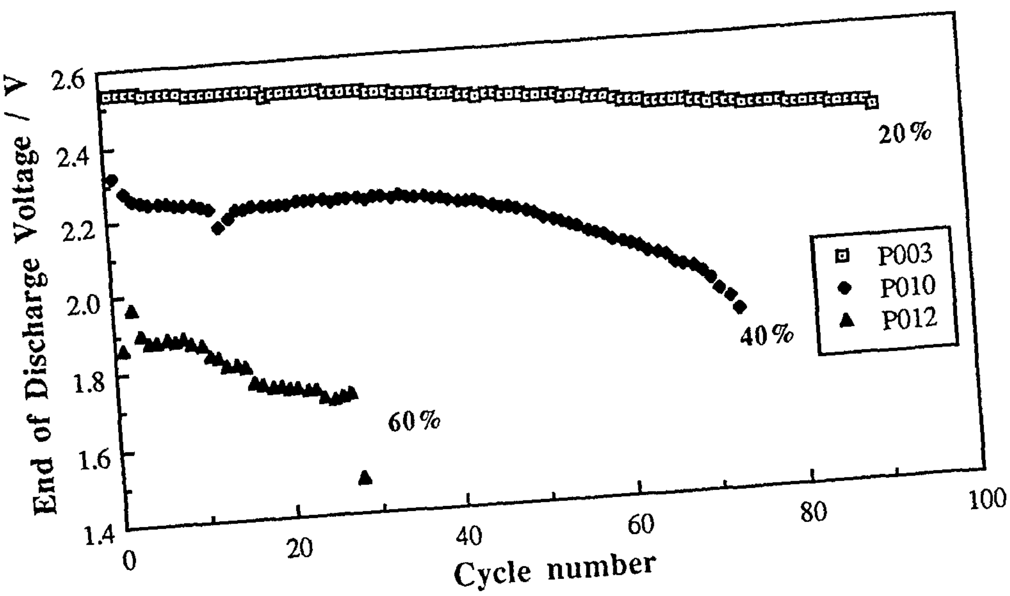
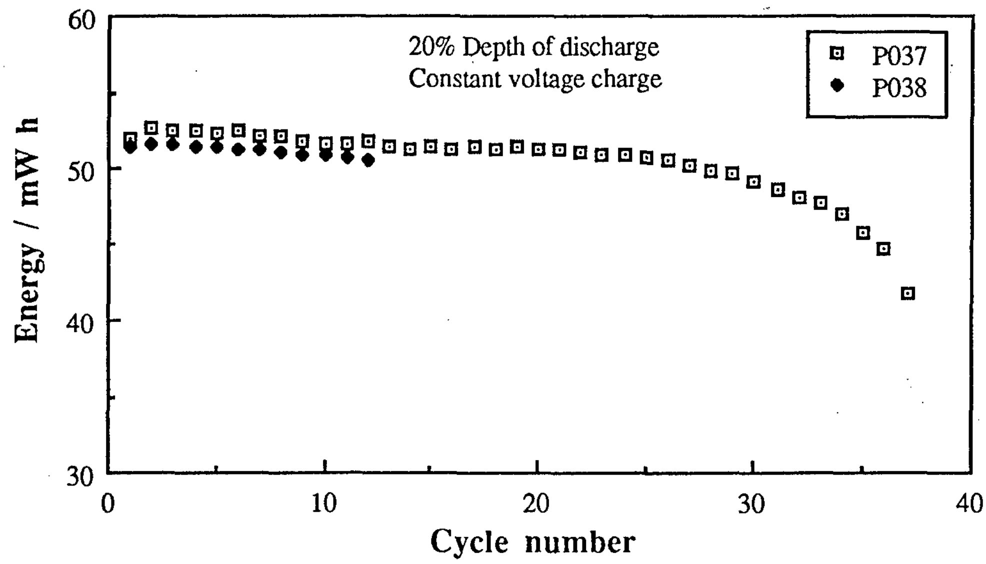


Figure 9 *Comparison of cycle performance data for 20%, 40% and 60% depth of discharge.*



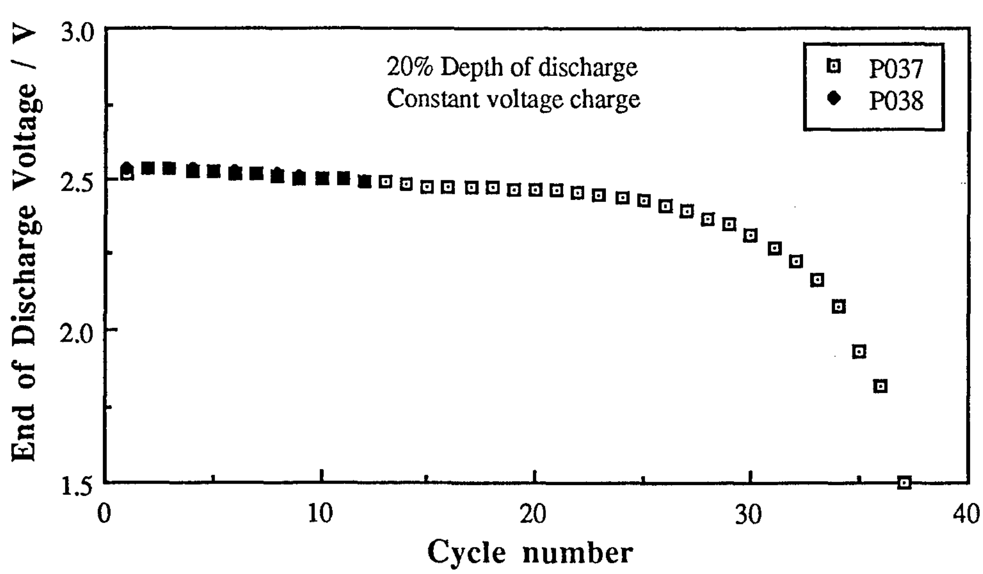


Figure 10 *Cycle performance data for the Mark I LPB cell at 20% depth of discharge with a constant voltage charge.*

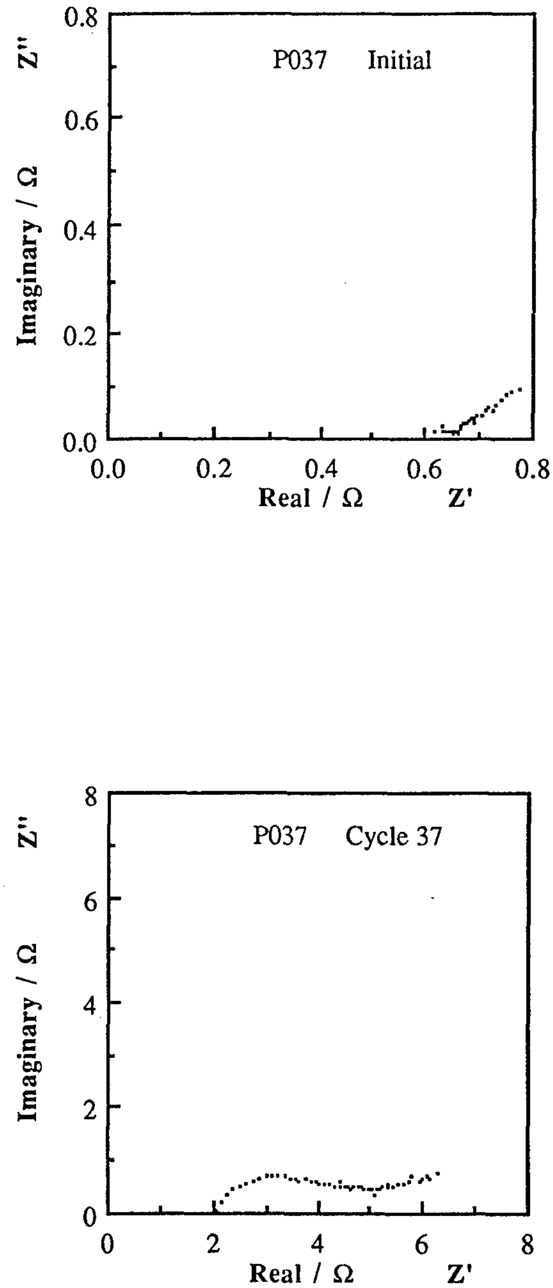
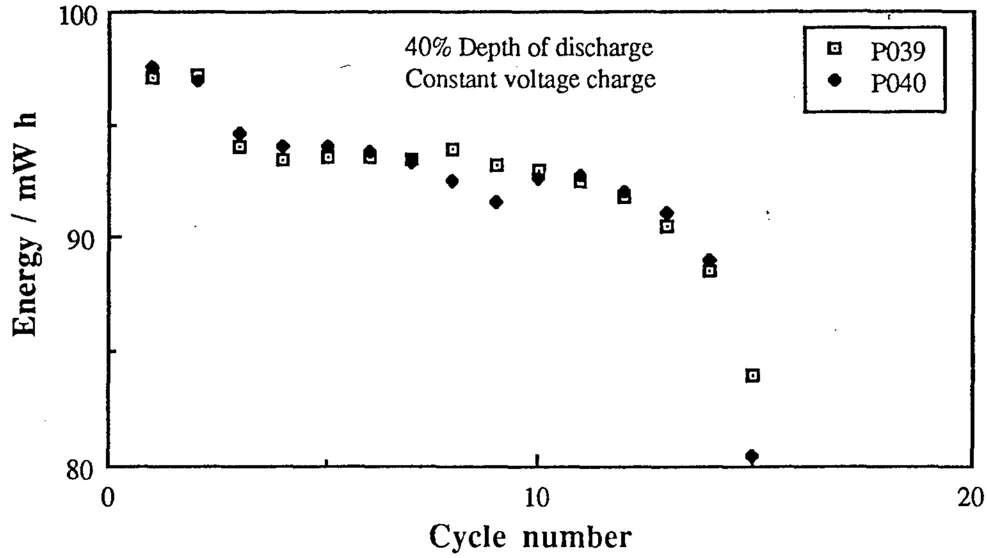


Figure 11 *A.c. impedance data for P013 cycled at 60% depth of discharge*



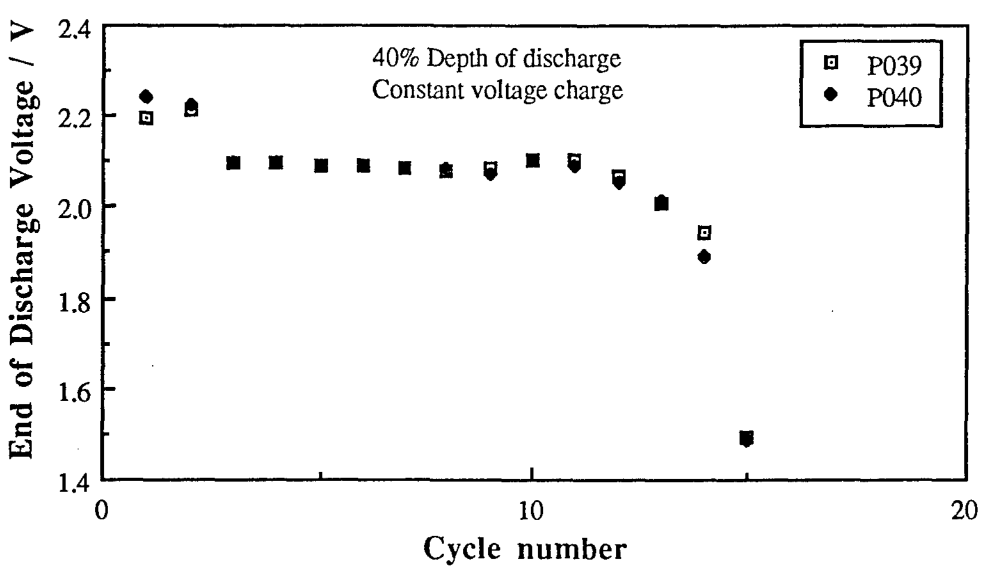
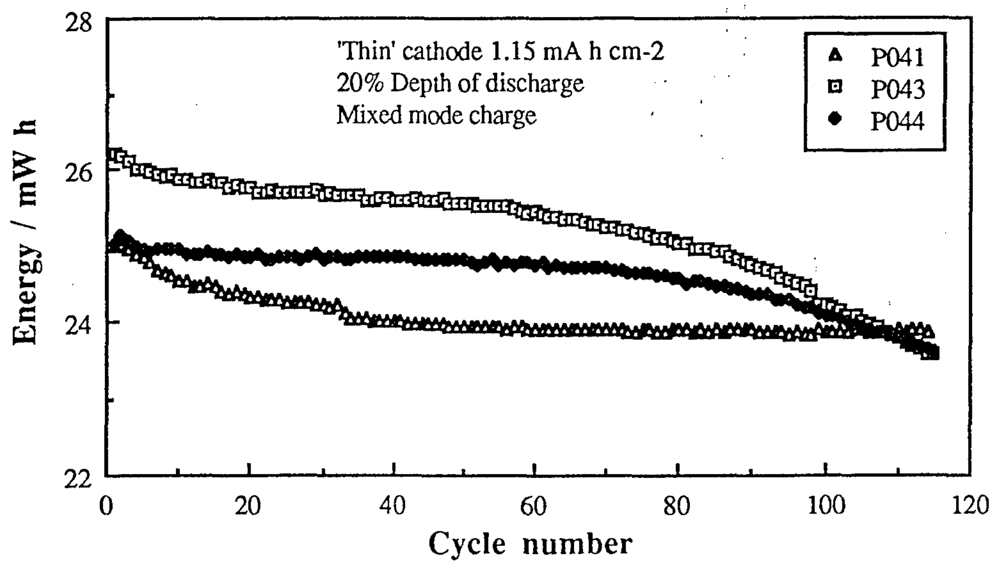


Figure 12 *Cycle performance data for the Mark I LPB cell at 40% depth of discharge with a constant voltage charge.*



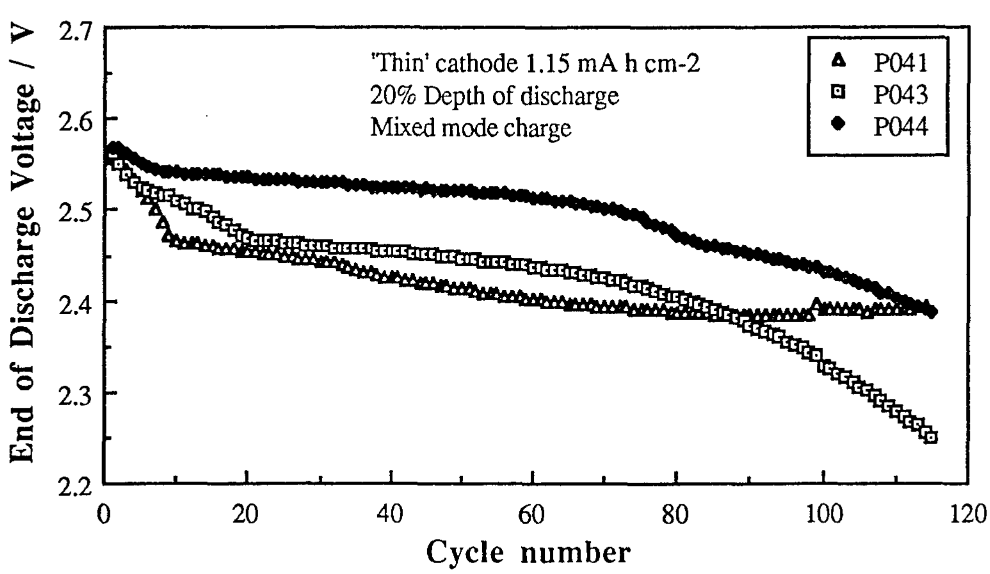
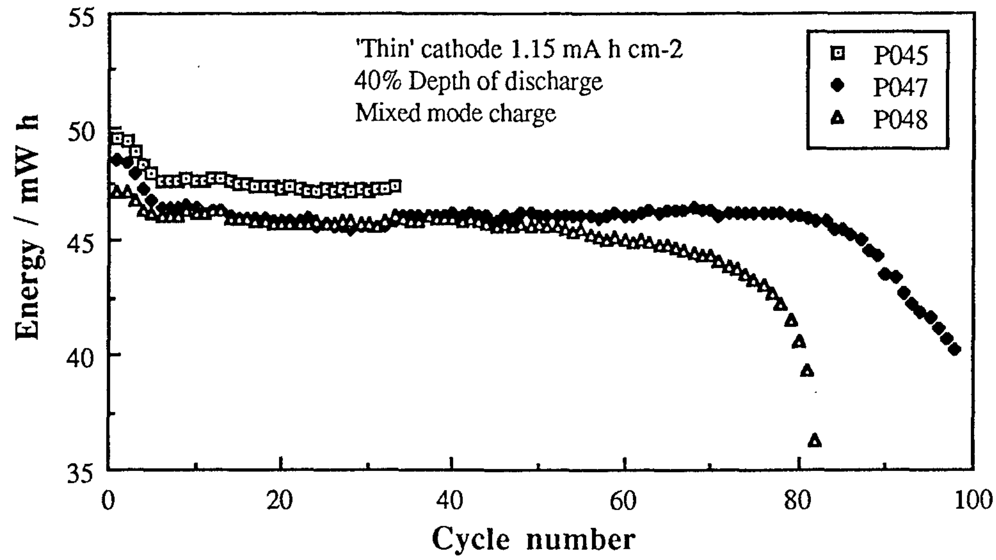


Figure 13 *Cycle performance data for the Mark I LPB cell with a 'thin' composite cathode (1.15 mA h cm^{-2}) at 20% depth of discharge .*



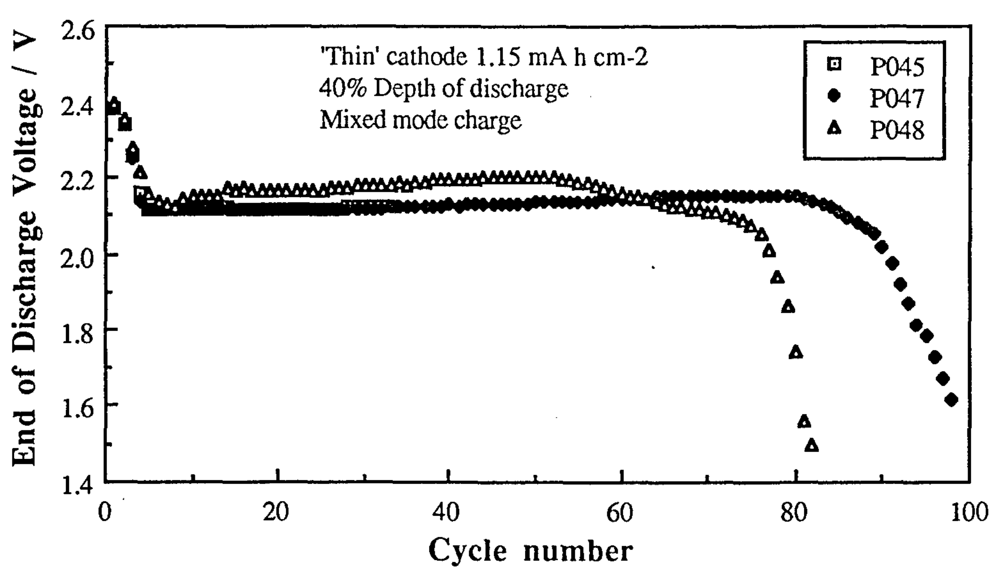


Figure 14 *Cycle performance data for the Mark I LPB cell with a 'thin' composite cathode (1.15 mA h cm^{-2}) at 40% depth of discharge .*

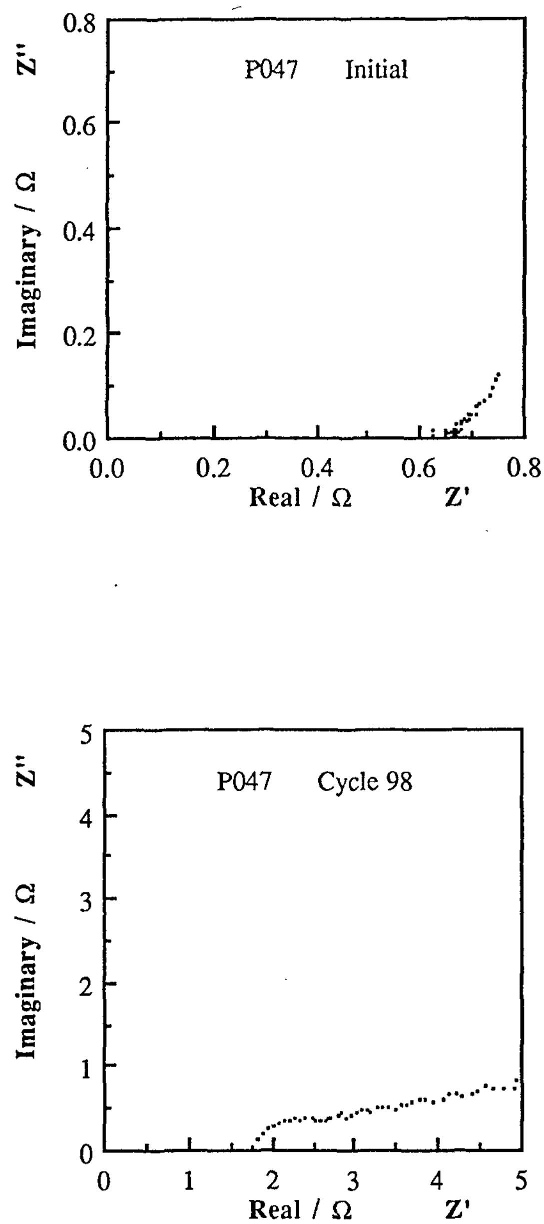


Figure 15 *A.c. impedance data for P047 cycled at 40% depth of discharge.*

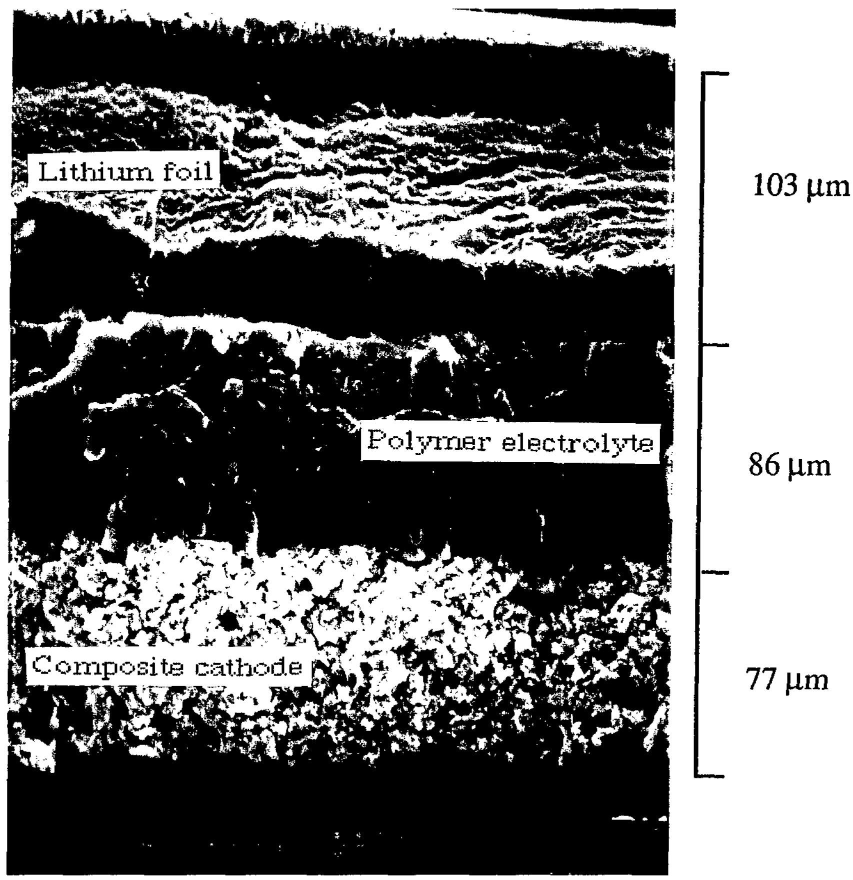


Figure 16 *Scanning electron micrograph of the cross-section of an uncycled Mark I LPB .*

3. List of Publications arising from the Contract Work

AEA Technology, Harwell Laboratory has published no materials resulting from the contract work.

AEA Technology, Harwell Laboratory has no current plans to publish any of the material resulting from the contract work.

4. Professional Personnel

The following is a list of professional personnel who have contributed to the Contract Work. Dr Robin John Neat, Dr William James Macklin, Mr Raymond John Powell and Mrs Alison Jane Walker. None of these personnel were awarded an advanced degree during the duration of the project.

5. Interactions

No interactions between Harwell staff and any organisation, other than the Program Manager and associated staff, has taken place on the subject of the contract work during the duration of the contract.

6. Inventions and Patents

The contract work has not given rise to any inventions and AEA Technology, Harwell Laboratory has not filed or authorised others to file any patents arising from the contract work.

7. Additional Information