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Effect of compression on the behaviour of lead-acid batteries

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Abstract

Mechanical pressure applied to the plate group is known to increase the cycle life of lead-acid batteries. However, the mechanism of the active material stabilisation process is not completely understood yet. The evolution of mechanical pressure in a lead-acid battery during a cycle and through cycle life is also a topic with still many open questions. Additionally, the separation systems available today are not able to exert high compression on the electrode plates: glass mats crush when compressed too much and the gel design does not allow any significant pressure generation at all. Daramic have developed the new acid jellying separator (AJS) allowing the application of mechanical pressure on the plate group. This paper reports on

1. The capacity evolution during cycling for cells with different separation systems namely AGM, gel and AJS, under a variety of initial compression levels.
2. The evolution of mechanical pressure on the cell walls during a cycle and through cycle life for different separation systems, and initial compression levels.
3. The condition of the active materials at the end of life for cells cycled under compression.
4. The effect of the addition of phosphoric acid to the electrolyte.
5. The effect of the application of mechanical pressure on the corrosion of pure lead based on cyclic voltammetry measurements under compression.

The results of these studies are presented together with some conclusions about the mechanism and effect of compression on a VRLA battery.

Keywords: VRLA batteries; AJS separator; Compression; Mechanical pressure; Active material; Corrosion

1. Introduction

The cycle life of valve regulated lead-acid (VRLA) batteries is often limited by a capacity loss of the positive plate. In addition to the problem at the grid/positive mass interface, changes in the whole crystalline structure of the positive active mass make a major contribution to this capacity loss [1].

The so-called softening process, which is due to a loss of contact between the lead dioxide particles as the active mass tends to swell, seems to be the major reason for the decrease of capacity, especially under high rate discharge load [2,3]. This phenomenon has been the subject of many investigations during the last few years [4–10].

Since 1983, mechanical pressure applied to the plate group of a lead-acid battery has been known to improve the cycle life of the battery [11]. The reason for the improvement of the cycle life seems to be a stabilisation of the positive active material preventing swelling of the active mass and thus reducing the capacity loss due to limited conductivity [12]. However, the application of mechanical pressure to the plate group of conventional VRLA batteries was limited until now because of the lack of a separation system that is able to withstand the mechanical pressure required and to transfer it to the plate group over the whole cycle life.

With the aim of overcoming this problem, a new separator called acid jellying separator (AJS) was developed by Daramic [13]. The new AJS aims at combining the advantages of the gel system with the potential of applying high pressure to the plates. In fact, the AJS system prevents stratification of acid and liquid [14] and still has good mechanical properties after many cycles in the battery, as illustrated in Fig. 1.

Within the framework of a project supported by the ALABC and the European Union, the new material, and

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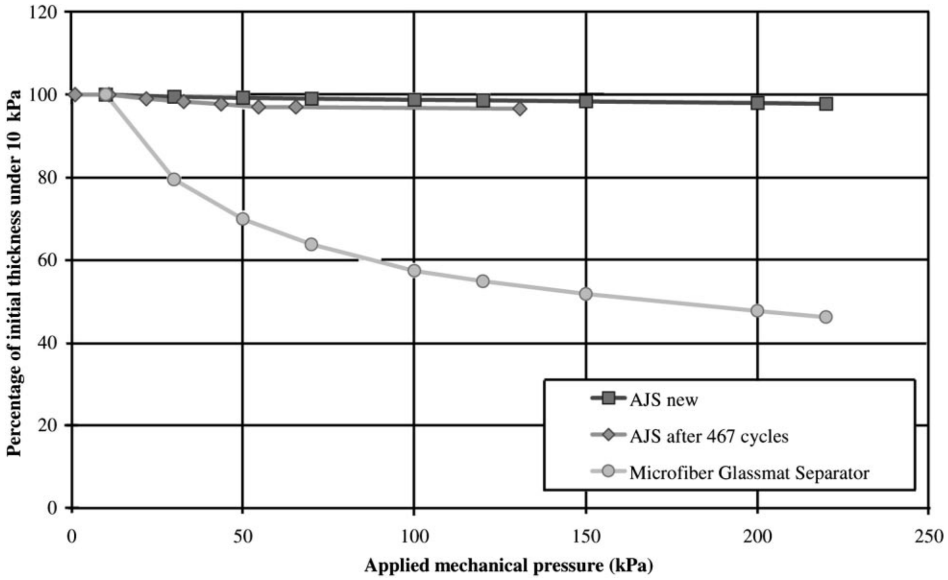


Fig. 1. Relative deformation of the separator versus mechanical pressure.

its behaviour in VRLA batteries compared to conventional systems, were studied in detail [15].

This paper gives an overview of the knowledge gained about the development of mechanical pressure in VRLA cells and batteries. Also, the effect of mechanical pressure on the performance of the VRLA system is demonstrated as a function of the separation system and the electrolyte composition (with or without phosphoric acid). Finally, the mechanisms of processes causing mechanical pressure changes in the lead-acid system are discussed.

2. Experimental

Cells and batteries with 48 A h nominal C/5 capacity and different separation systems and electrolyte compositions were tested. The initial conditions of the tests are summarised in Table 1.

The cells were submitted to 25 C/2 cycles followed by two C/5 capacity checks with the following regime: *charge*: IU: 14.4 A, 10 h, 2.4 V for AGM, 2.45 V for AJS and gel; *discharge*: 20 A until 1.6 V (C/2); 9.6 A until 1.75 V (C/5).

During the C/5 cycles, the gas emission of the cells was recorded as well as the evolution of the mechanical pressure.

Some cells were removed from cycling because they showed hints of internal short circuits. After disassembly the active material was analysed and inspected by means of an optical microscope.

The batteries were cycled at the C/2 rate with the following regime: *charge*: IU: 14.4 A, 8 h, 2.4 V/cell for AGM, 2.45 V/cell for AJS and gel, constant current phase: 400 mA for 2 h; *discharge*: 20 A until 1.6 V/cell (C/2).

Table 1
Cells and batteries tested

	Separation system	Electrolyte	Mechanical pressure
Cell 1	Gel (Darak 2000)	With phosphoric acid	30 kPa[a] (constant distance[b])
Cell 2	AGM		30 kPa[a] (constant distance[b])
Cell 3	AJS		80 kPa[a] (constant distance[b])
Cell 4	AJS		30 kPa[a] (constant distance[b])
Cell 5	AJS		60 kPa (springs[c])
Cell 6	AJS	With phosphoric acid	30 kPa[a] (constant distance[b])
Battery 1	AJS	With phosphoric acid	80 kPa[a] (constant distance[b])
Battery 2	AJS	With phosphoric acid	60 kPa (springs[c])
Battery 3	AGM		60 kPa (springs[c])
Battery 4	AGM		Free[d]
Battery 5	Gel (Darak 2000)	With phosphoric acid	Free[d]
Battery 6	Gel (Darak 2000)	With phosphoric acid	Constant nominal thickness

[a] The initial mechanical pressure applied to the battery case.

[b] Constant distance fixed between aluminium plates.

[c] The initial mechanical pressure is applied through the use of springs and stays almost constant during the cycle life.

[d] Without any external mechanical pressure applied.

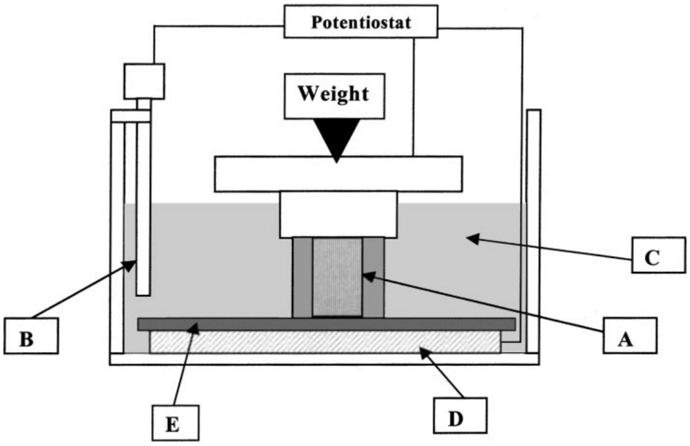


Fig. 2. Apparatus for the measurement of the effect of mechanical pressure on the corrosion behaviour of pure lead. A: pure lead electrode embedded in Teflon; B: reference electrode $Hg/Hg_2SO_4/H_2SO_4$; C: sulphuric acid electrolyte 1.28 g/cm^3; D: counter electrode, pasted negative electrode (1 A h); E: AJS separator. Cyclic regime: charge 30 s at 1400 mV; discharge 30 s at 900 mV.

The effect of mechanical pressure on the corrosion of pure lead electrodes was studied by means of cyclic polarisation. The apparatus and conditions are described in Fig. 2.

3. Results and discussion

3.1. The effect of mechanical pressure on the initial performance and cycling behaviour

Fig. 3 shows the evolution of the C/5 capacity measured during the capacity checks after 25 C/2 cycles.

With the end of life being defined as falling short of 80% of their initial capacity, the cells had the following cycle life.

Separation system	Initial mechanical pressure (kPa)	Cycle life
AGM	30	≈250
AJS	30	≈540
AJS	80	≈1005

Compared to compressible AGM, a marked improvement of the cycle life is achieved with AJS cells under the same

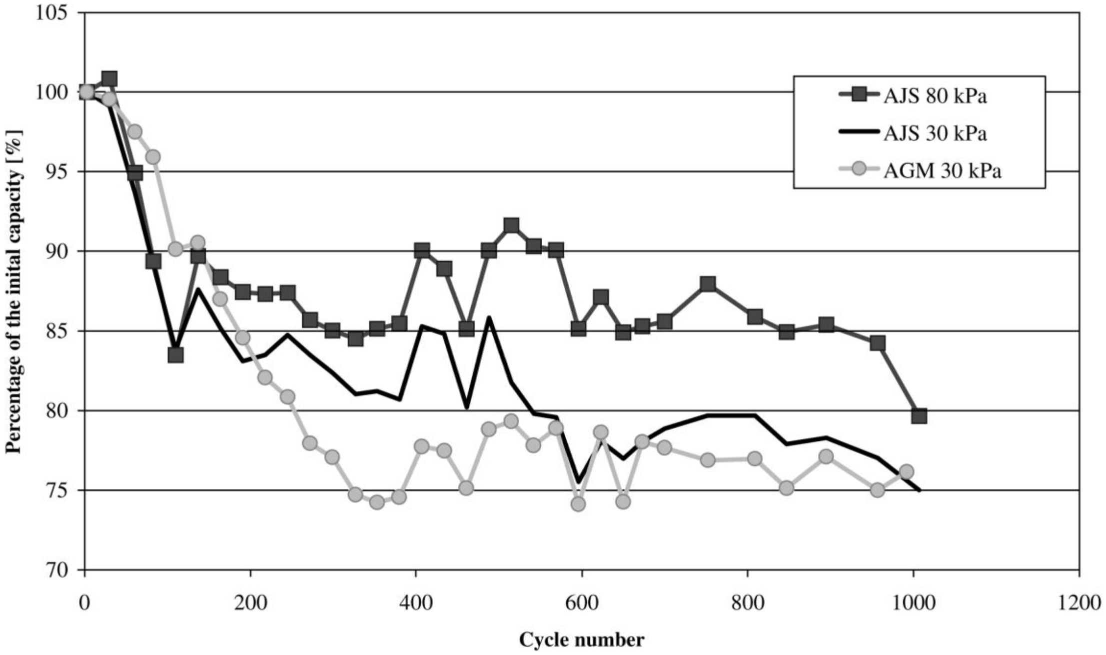


Fig. 3. C/5 capacity evolution of cells with different separation systems and initial external mechanical pressure.

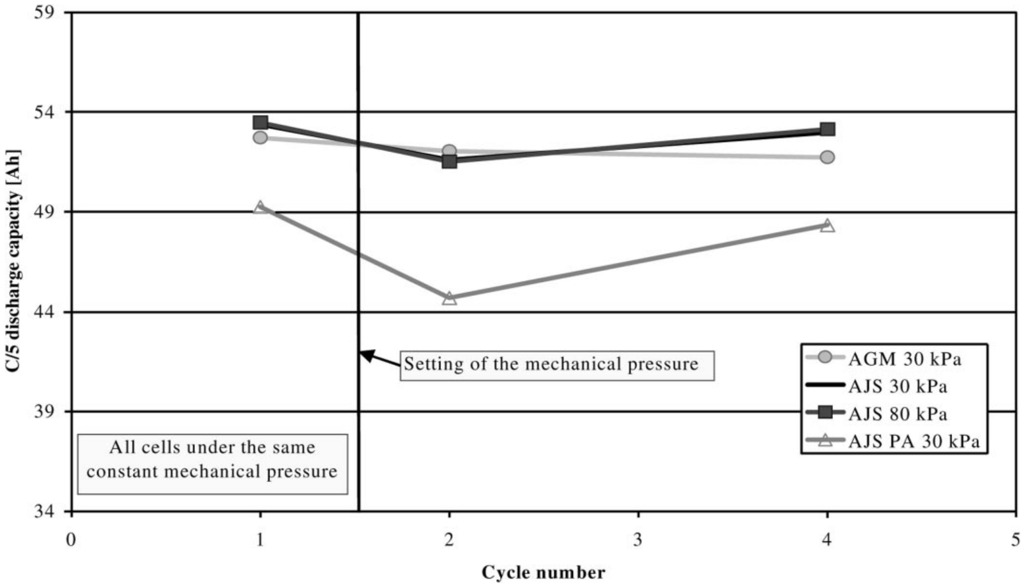


Fig. 4. C/5 capacity of cells at the beginning of life before and after compression.

initial mechanical pressure. By increasing the mechanical pressure from 30 to 80 kPa, an additional improvement in cycle life was observed. In total, four times more cycles (1005 versus 250) could be achieved with AJS at 80 kPa as with AGM at 30 kPa.

Regarding the initial performance, Fig. 4 shows the C/5 capacity of the cells. The cells were subjected to a different initial mechanical pressure after the first cycle. The mechanical pressure level seems to have no influence on the cell performance in the very first cycles since both AJS cells at 30 and 80 kPa show the same behaviour (identical curves in Fig. 4).

The cell with phosphoric acid has a lower C/5 capacity than the cells containing no phosphoric acid.

Concerning the further development of the capacity quite early in the cycle life, Fig. 5 represents the evolution of the C/2 capacity of the gel and AJS batteries. For the gel system, one battery is free of constraint and the second is fixed at its nominal interplate spacing. For the AJS system, one battery is submitted to 60 kPa constant pressure by means of springs

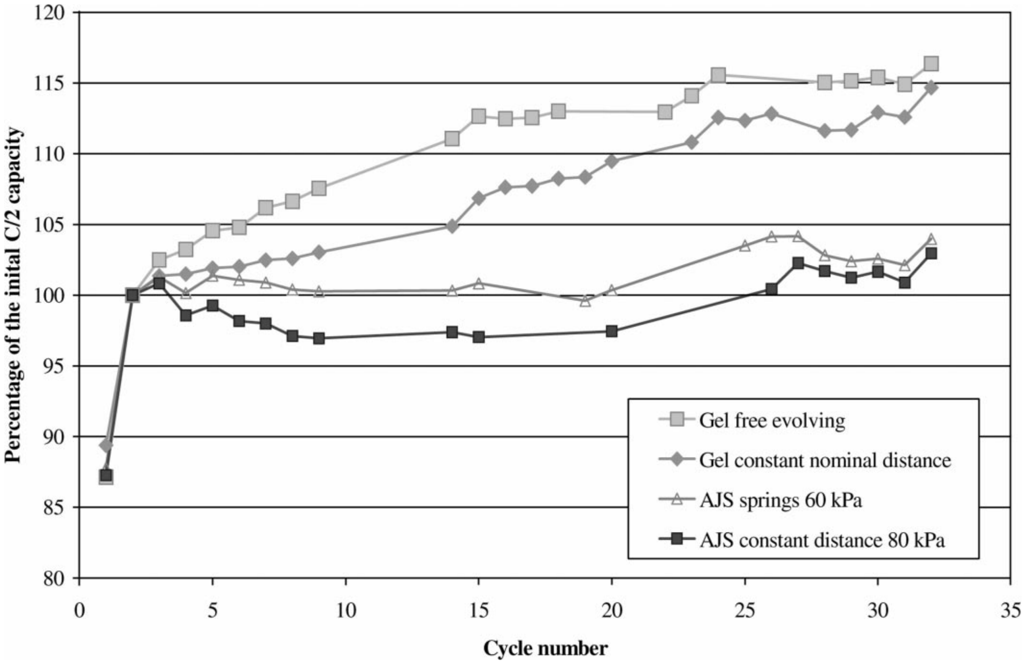


Fig. 5. C/2 capacity evolution of batteries under different mechanical pressures at the beginning of life.

and the other was put under 80 kPa initial external mechanical pressure and subsequently maintained at constant distance.

The application of a mechanical pressure seems to prevent the frequently observed capacity increase in the early stages of cycle life. A positive effect of the application of mechanical pressure is not visible during the first 30 cycles. Both AJS versions, subjected to 60 and 80 kPa, respectively, have similar capacities.

It is concluded that mechanical pressure applied to the walls of a VRLA cell or battery increases its cycle life if the separator is able to withstand the pressure over a large number of cycles. The new AJS material has this property and leads to very good cycling results.

3.2. *Evolution of the mechanical pressure*

The application of mechanical pressure is expected to reduce the ''breathing effect'' of the active material, thus limiting the progressive destruction of the positive electrodes [16]. The force transmitted to the walls of the cells and batteries was recorded during cycling and Fig. 6 shows the typical evolution of the mechanical pressure during one cycle for two different separation systems.

The measurements on cells kept at a constant distance show that the mechanical pressure on the cell walls increases during discharge and decreases during charge.

During *discharge*, the transformation of lead dioxide to lead sulphate on the positive electrode is correlated with a volume increase by a factor of 1.9 [11]. On the negative electrode, the transformation of lead to lead sulphate is also related with a volume increase ($V_{\mathrm{PbSO_4}}/V_{\mathrm{Pb}} \approx 2.6$). The increase in mechanical pressure for the cell containing the compressible AGM separator is smaller than for the AJS cell. The compressible AGM separator leaves some space for the active material to expand. Therefore, in contrast to the incompressible AJS separator, the force of expansion is not fully transmitted to the cell walls.

During *charge*, the mechanical pressure is a superimposition of:

- the volume shrinkage of the active materials (conversion of lead sulphate to lead and lead dioxide) which reduces the mechanical pressure;
- the gas evolution which produces a positive contribution to the mechanical pressure.

Fig. 7 shows the graph for the gas volume released by the valve and the corresponding pressure measured at the walls of the AGM cell. The valve opens after 140 min of charge and closes again after 200 min of charge. Before the valve opens, the increase of the internal gas pressure in the cell causes an increase of the pressure registered on the cell walls (the valve opens at a gas overpressure of about 7–11 kPa).

The evolution of the mechanical pressure during cycle life is presented in Fig. 8. In order to avoid any superimposition of gas pressure, the mechanical pressure for each capacity check was recorded at the end of discharge.

The mechanical pressure at the end of discharge increases steadily during the whole cycle life as a result of the irreversible expansion of the active material. For the cell with a compressible AGM separator, the pressure increase is relatively small. For the cells with the incompressible AJS, the increase of the mechanical pressure is much higher and the trend is similar for both the cell initially subjected to 30 kPa and that initially subjected to 80 kPa. The compressible AGM separator allows some expansion of the active

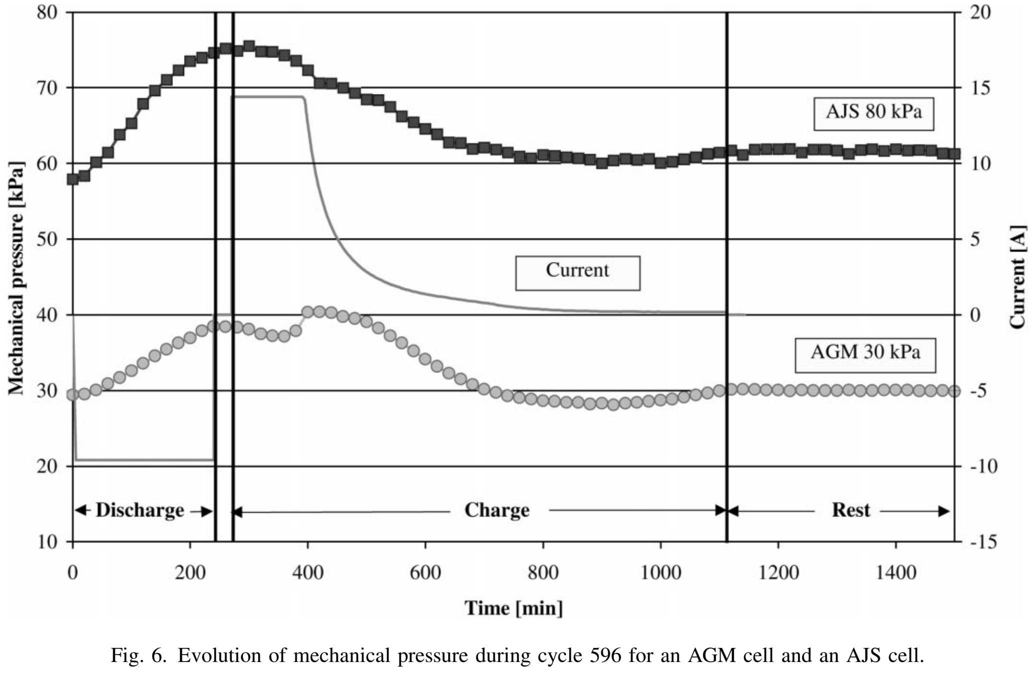


Fig. 6. Evolution of mechanical pressure during cycle 596 for an AGM cell and an AJS cell.

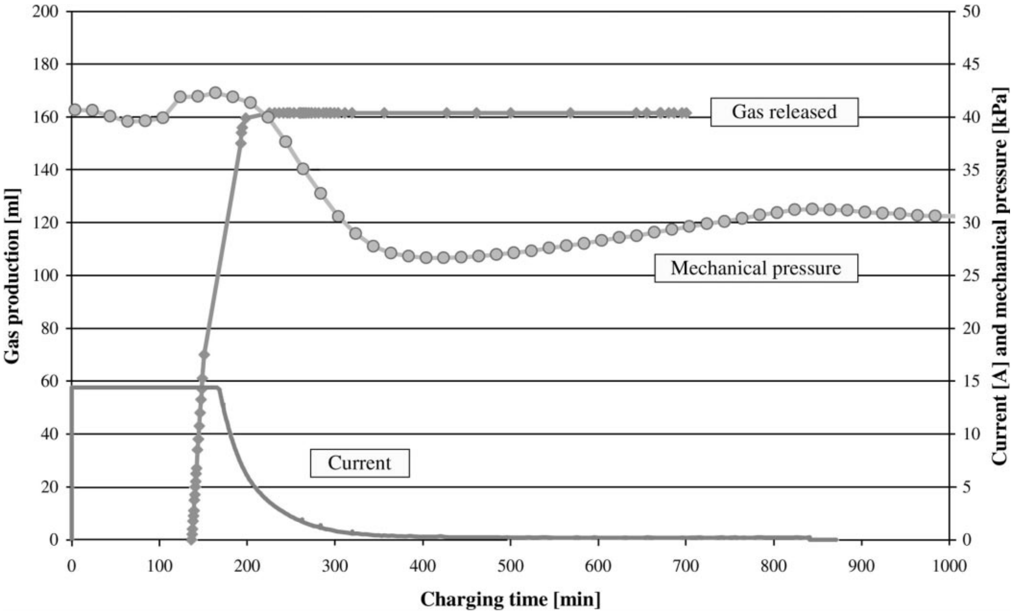


Fig. 7. Gas evacuation and mechanical pressure on the cell walls for AGM cell during charging in cycle 515.

material while the incompressible AJS withstands the expansion and thereby increases the forces transferred to the cells walls.

A linear approximation of the curves in Fig. 8 can be made with the following equations and regression coefficients.

	Equation of the straight lines	Regression coefficient (R^2)
AGM	$Y = 0.0205 \times X + 34.201$	0.6438
AJS 30 kPa	$Y = 0.0843 \times X + 26.435$	0.9751
AJS 80 kPa	$Y = 0.0806 \times X + 50.411$	0.9827

For AJS, the linear approximation shows a good fit with a linear increase of the mechanical pressure. The slope is very similar for cells with 30 and 80 kPa initial pressure. While the mechanical pressure increase is 0.0843 kPa per cycle for the less compressed version, the more compressed version shows an increase of 0.0806 kPa per cycle. Even after 900 cycles and beyond a pressure level of 120 kPa, the mechanical pressure still seems to increase which indicates a further expansion of the active material.

For the more compressible AGM separator, the slope of the curve seems to increase from cycle 450 on. It is possible that the separator has reached a critical thickness

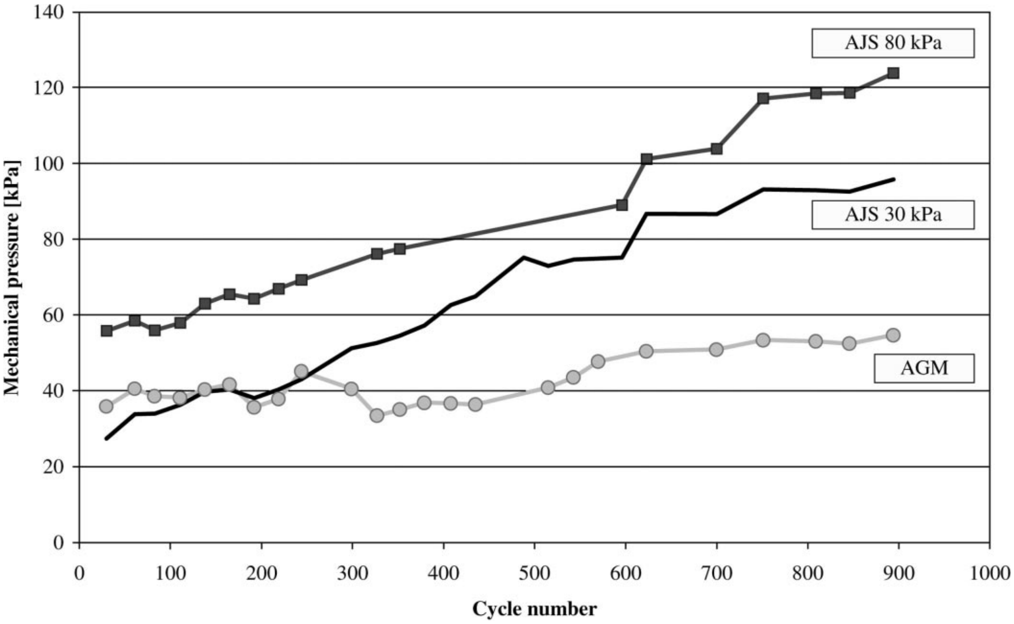


Fig. 8. Evolution of the external mechanical pressure at the end of discharge versus cycle number.

and is not as compressible anymore as it was in the beginning.

These results indicate that the mechanical pressure on the walls of a cell kept at a constant thickness increases during discharge as a result of the expansion of the active material and decreases during charge.

During cycle life, the mechanical pressure recorded at a fixed state of charge shows an almost linear increase for cells with the AJS separator as a result of the irreversible active material expansion. The level of the initial mechanical pressure has only a small effect on the slope of the pressure increase.

3.3. Mechanical pressure and the effect of phosphoric acid

As shown in Fig. 4, the presence of phosphoric acid (PA) in the electrolyte of a VRLA cell leads to a lower C/5 capacity at the beginning of life compared to cells containing no phosphoric acid. Fig. 9 shows the relative difference between the C/2 and the C/5 capacity as an average over the whole cycle life.

The presence of phosphoric acid decreases the low rate capacity (C/5) in comparison with the same system containing no phosphoric acid, while the high rate capacity (C/2) is not affected.

The ‘‘breathing’’ of the active material can be correlated with the increase of the mechanical pressure between the beginning and end of discharge. Fig. 10 shows the evolution of the mechanical pressure on the cell walls during cycle 100 for two cells with identical designs apart from the presence of phosphoric acid in one of them. For the cell containing phosphoric acid, the variation of mechanical pressure is much smaller during discharge ($\mathrm{d}P_{\mathrm{dis\,PA}} < \mathrm{d}P_{\mathrm{dis}}$) while both cells deliver the same number of Ah. The action of phosphoric acid reduces the expansion of the active material during discharge. Since phosphoric acid affects only the positive electrode [17], it limits the expansion of the positive active mass as a result of its influence on the crystallisation of lead dioxide. Döring et al. [18] showed that the presence of phosphoric acid adsorbed on the electrode surface leads to the formation of fine crystalline lead dioxide. In the following discharge, the fine crystalline lead dioxide is transformed into fine crystalline lead sulphate which can form a half-permeable layer that protects the electrode against further discharge.

Both the formation of small lead sulphate crystals and the protection of the electrode against further discharge favour a smaller expansion of the positive active material during discharge in the presence of phosphoric acid.

In Fig. 11, the increase of mechanical pressure during discharge versus cycle number is presented.

For all cells except the one containing phosphoric acid, the increase of the mechanical pressure between beginning and end of discharge is almost constant during cycle life with only a very slightly increasing trend. At the beginning of life, the presence of PA results in a much lower increase of the mechanical pressure during discharge ($\mathrm{d}P_{\mathrm{dis\,PA}} < \mathrm{d}P_{\mathrm{dis}}$). However, the variation increases for the cell containing PA and after some 350 cycles both cells under same initial mechanical pressure with and without PA reach the same value. It could mean that the phosphoric acid progressively loses its effect in limiting the ‘‘breathing’’ of the active material.

It appears that the phosphoric acid protects the positive active material against expansion. But its effect seems to be limited in time and to disappear after some hundreds of

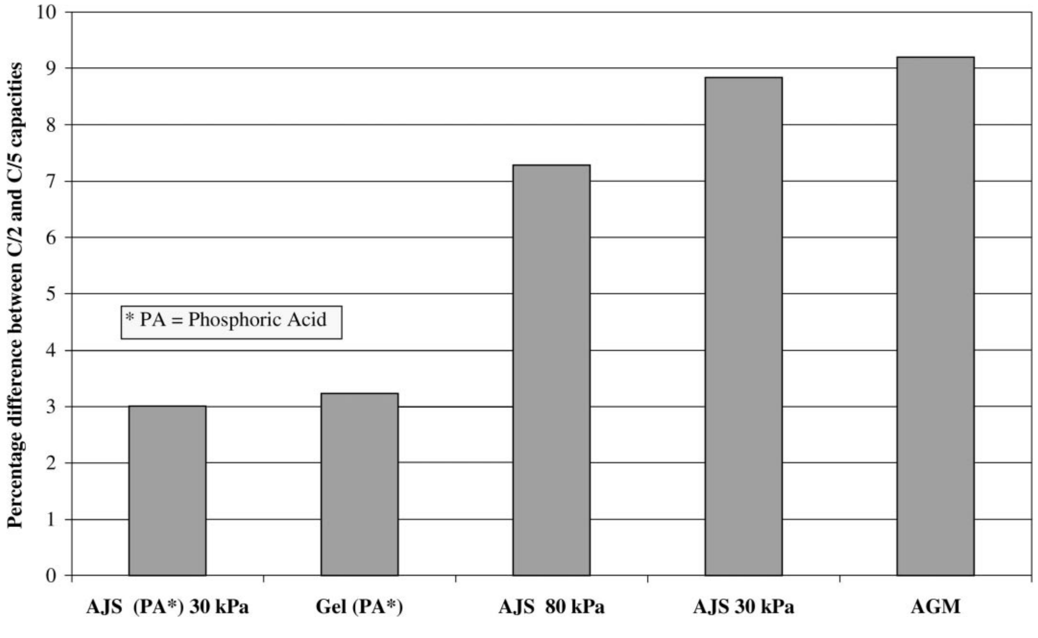


Fig. 9. Difference between C/2 and C/5 capacity for different electrolyte and separation systems.

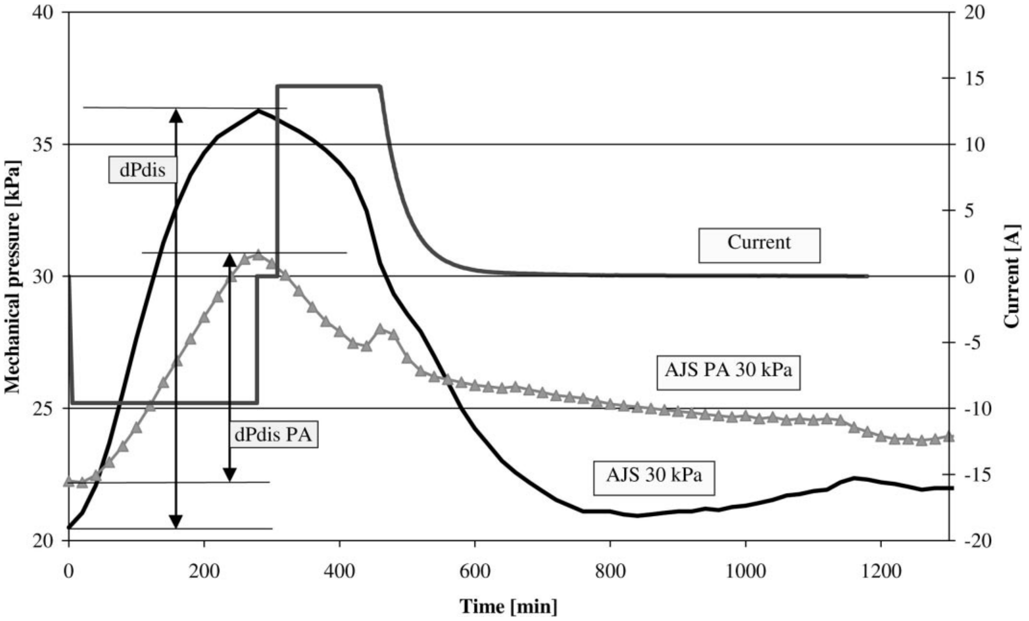


Fig. 10. Evolution of mechanical pressure during cycle 100 for AJS cells with and without phosphoric acid under 30 kPa initial mechanical pressure.

cycles. Additionally, the presence of phosphoric acid causes a lower performance of the cell at low discharge rates.

3.4. *How does the application of mechanical pressure lead to a better cycling behaviour?*

The application of mechanical pressure seems to have two separate effects, one on the active material and the other on grid corrosion. The effect on the active material is a superimposition of the effects at the positive and negative active materials.

On the *positive electrode*, the application of mechanical pressure is expected to produce a stabilisation of the electrode structure.

In a recent paper [19], it was suggested that mechanical pressure decreases the oxygen evolution that occurs at the connecting regions between the lead dioxide spheres by decreasing the potential of these necks. This effect could reduce the ‘‘breaking’’ of the positive active material structure, caused by the generation and migration of oxygen.

Table 2 shows the thickness of positive electrodes of new and cycled cells.

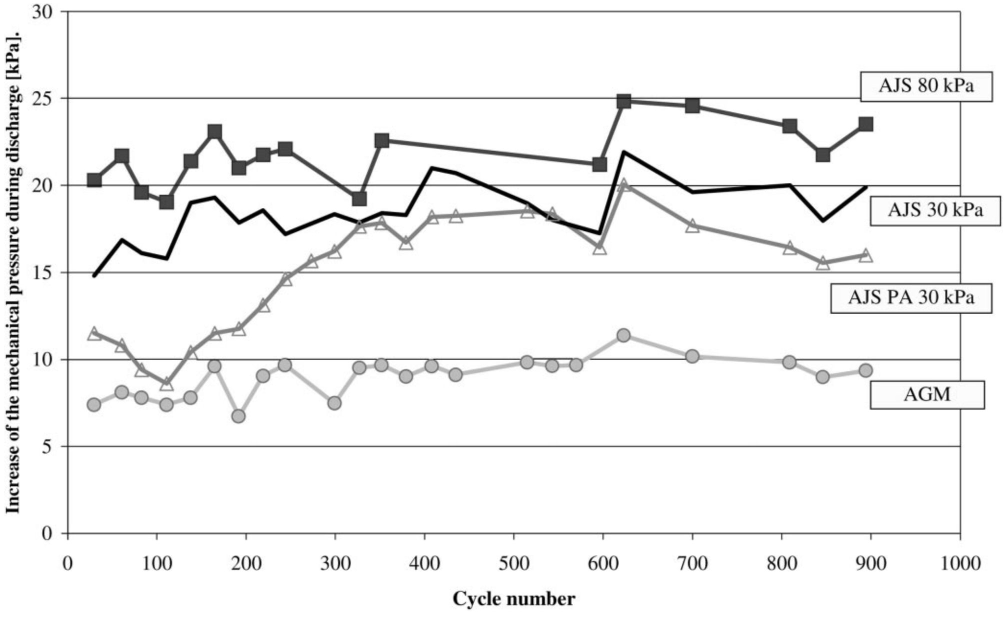


Fig. 11. Increase of the mechanical pressure during discharge versus cycle number.

Table 2
Positive plate thickness and active mass porosity for a new cell and two cells after about 450 cycles with high and low pressure, respectively

	Cell type		
	New	Cycled AJS	Cycled gel
Mechanical pressure		High	Low
Top thickness (mm)	2.85	3.3	4
Middle thickness (mm)	2.85	3.4	4.2
Bottom thickness (mm)	2.85	3.5	4.1
Porosity of the active mass (%)	51–57	58–61	63

The expansion of the positive plate was significantly constrained by the use of AJS and the application of higher mechanical pressure (60 kPa) in comparison to a cycled gel cell under only low mechanical pressure (30 kPa). But as shown above (Fig. 8), the expansion of the active material produces mechanical pressures substantially in excess of 60 kPa when the material is constrained. A small mechanical pressure is not able to fully hinder the expansion since the plates grew by about 20% within 450 cycles under 60 kPa mechanical pressure applied by springs. The application of a mechanical pressure in the range of about 60 kPa is not high enough to prevent the expansion of the positive active mass completely, but it reduces it significantly.

The positive electrodes of both cycled cells show a higher porosity than the new electrode, with a lower porosity for the cell subjected to higher pressure (Table 2 and Fig. 12).

The active material of the AJS cell cycled under mechanical pressure seems to develop a layer structure (Fig. 13) with a porous grey layer (B) framed by the densest layer at the surface (A) and a dense brown central layer (C).

The second effect of the application of mechanical pressure on the positive electrode seems to be a lower corrosion of the positive grid. In a study of lead corrosion, Garche [20] showed that the corrosion rate of lead spines in tubular plates is reduced by the presence of active material and of inert material as well. The force on the corrosion layer imposed by the inert or active material seems to stabilise the corrosion layer mechanically.

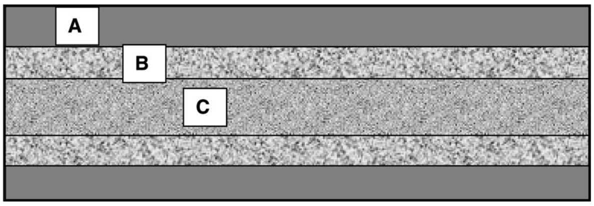
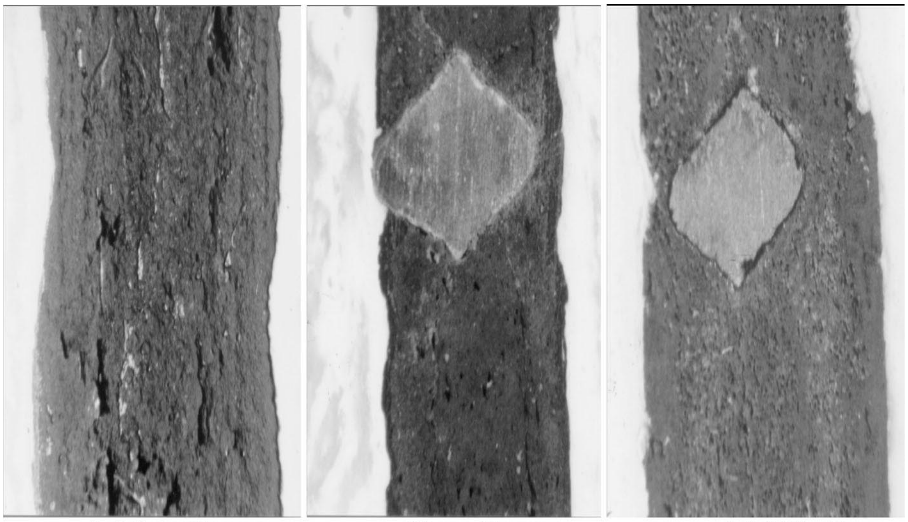


Fig. 13. Schematic representation of the positive electrode structure for an AJS cell cycled under 60 kPa mechanical pressure.

With the apparatus and conditions described above (Fig. 2), the discharge capacity of the corrosion layer on pure lead electrodes with increasing cycling time was recorded (Fig. 14). The mechanical pressure on the lead surface was transferred by the AJS separator.

Faraday's law allows a direct correlation between the discharge capacity of the corrosion layer and its share of ''active material''. Fig. 14 clearly shows that the direct application of mechanical pressure on the surface of the lead electrode slows down the growth of the corrosion layer with increasing cycle number.

Following the theory of Garche [20], a dense corrosion layer grows until it reaches a critical thickness. The critical thickness is reached when the internal mechanical stress relaxes, which results from the higher volume of the corrosion products (PbO_2) in comparison to the volume of lead (factor 1.35). The application of mechanical pressure is able to partially compensate the internal stress of the corrosion layer. In that way, it prevents the cracking of the dense layer and thus increases its critical thickness. Thus, the application of mechanical pressure reduces the corrosion by stabilising the dense corrosion layer.



cycled Gel cell **New cell** **cycled AJS cell**

Fig. 12. Picture of the positive active electrodes of one new and two cycled cells.

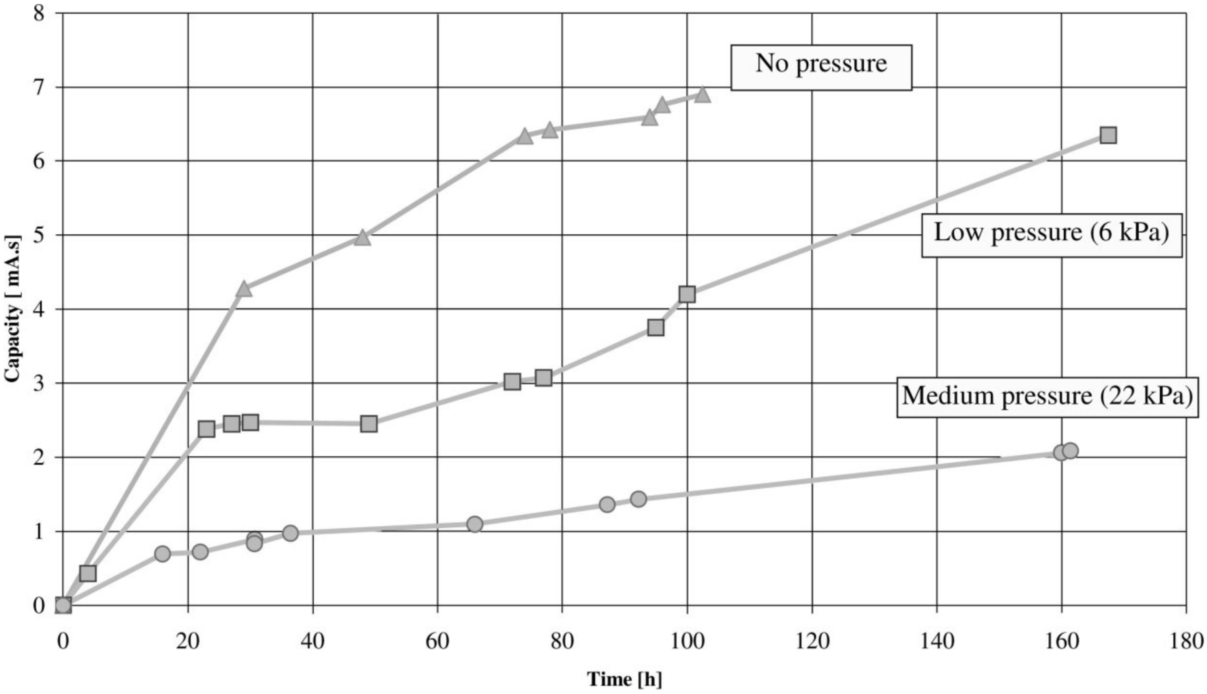


Fig. 14. Discharge capacity of the corrosion layer on pure lead electrodes versus cycling time.

On the *negative active material*, the application of a mechanical pressure seems to produce a compaction. Fig. 15 shows the negative electrode of a new cell and of a cell after about 450 full cycles under 60 kPa external mechanical pressure applied with springs.

Firstly, the plate from the cycled AJS cell has a flat surface. The compressive force has been applied homogeneously across the whole surface.

Secondly, the active material cycled under mechanical pressure does not show the same porosity as the new active material. While the porosity of a new negative plate measured with water is in the range of 55–57%, the value for the cycled plate of an AJS cell is about 40%. The porosity of the negative plate of the gel cell after some 450 cycles is still in the range of 46%. This indicates that the pressure has reduced the porosity of the negative beyond the level to be expected from normal cycling.

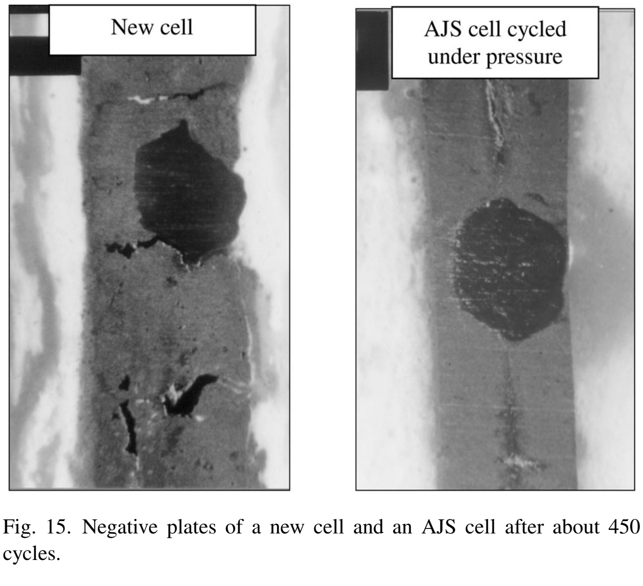


Fig. 15. Negative plates of a new cell and an AJS cell after about 450 cycles.

Thirdly, the thickness of the negative plates of the cycled AJS cell decreased compared to the new plate (see Table 3). The plate thickness was almost reduced to the thickness of the grid, i.e. 1.65 mm.

Finally, the application of a mechanical pressure influences the structure of the negative active mass. Fig. 16 shows pictures of the negative electrode of two cells after about 450 full cycles: a gel cell that was maintained at the initial nominal thickness during cycling and an AJS cell that was cycled under 60 kPa mechanical pressure applied with springs.

The negative plate of the cycled gel cell shows prints of the ribs of the separator (white circle). The active material shows three layers, a ‘‘sandwich structure’’ with the external

Table 3
Negative plate thickness and active mass porosity of the new and cycled cells

	Cell type		
	New	Cycled AJS	Cycled gel
Mechanical pressure		High	Low
Top thickness (mm)	1.9	1.7	1.9
Middle thickness (mm)	1.9	1.7	2.1
Bottom thickness (mm)	1.9	1.7	1.8
Porosity of the active mass (%)	55–57	40	46

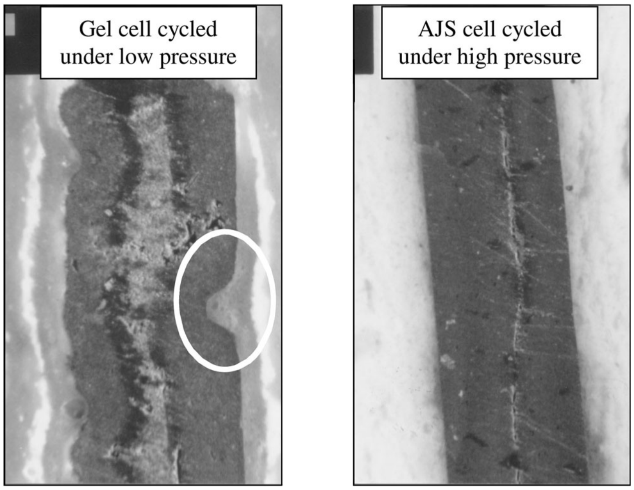


Fig. 16. Negative plates of cycled cells: gel under low pressure and AJS under high pressure.

layers consisting of a dense material and the inner layer being more porous. For some samples, the inner layer had formed some cracks. On the negative electrode of the AJS cell cycled under higher uniform mechanical pressure, the inner layer is much thinner, but also forms thin cracks at some places.

In short, the application of a high, uniform mechanical pressure seems to result in compaction of the ‘‘sponge structure’’ of the negative active material. This compaction can possibly reduce the capacity of the negative plate. In order to solve the problem that may appear on the negative electrodes in a ‘‘compression battery’’, either higher expander amounts in the negative paste and/or less over-pasting of the negative plate could be considered. In this way, the pressure would be transferred to the grid, thus allowing the negative active material to maintain its porosity.

In conclusion, the application of mechanical pressure on the walls of a lead-acid cell has the following distinct effects.

On the positive plate, the active mass is stabilised. The material is less porous than for the slightly compressed cell and the electrode has less expanded for the same number of cycles, additionally, shedding is hindered by the formation of a dense layer on the surface of the electrodes. The stabilisation of the positive active material is probably the main reason for the significant improvement of the cycle life obtained with AJS and high initial external mechanical pressure.

As a second positive effect, mechanical pressure possibly reduces the positive grid corrosion. The theoretical study of corrosion under mechanical pressure has to be verified by measurements of grid corrosion in real cycled cells. This will be part of our further work.

On the negative plate, the active material is compacted, possibly reducing the capacity of the negative electrode by decreasing its porosity. This drawback can possibly be prevented by the use of thicker grids and no over-pasting since the pressure would be transferred to the grids instead of to the active material.

4. General conclusions

The new AJS separator proves to give very good cycling results and allows the study of the effect of mechanical pressure on the cycling behaviour of cells, on the structure of the active material and on the corrosion of lead.

The application of mechanical pressure to a VRLA cell containing an incompressible separator leads to a significant increase in cycle life in comparison to a cell containing a compressible separator. The increase of the initial mechanical pressure from 30 to 80 kPa allows almost a doubling of the cycle life for cells with AJS material.

For cells kept at a constant distance, the changes in the molar volume of the active material increase the mechanical pressure during discharge and decrease it during charge. During the whole of cycle life, the mechanical pressure increases irreversibly.

The addition of phosphoric acid reduces the low rate capacity (C/5), while the higher rate capacity (C/2) is not affected. The presence of phosphoric acid decreases the expansion of the active material, but it seems to lose its effect as ‘‘positive active material expansion constraining agent’’ after a certain number of cycles (about 350 cycles).

The effect of mechanical pressure is the result of three factors:

- a stabilisation of the positive active material by restraining the expansion of the positive electrode;
- possibly a reduction of the anodic corrosion of lead;
- a compaction of the negative active material. The loss of porosity could be reduced when the electrodes are not over-pasted and the pressure is transferred to the grid instead of to the active mass.

5. Further work

Analysis of the active material should provide information on the layer structure of the electrodes after cycling under mechanical pressure. The nature and porosity of these layers will be studied.

Further work on the corrosion of pure lead under mechanical pressure will be carried out in order to understand the mechanism of corrosion reduction.

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