Amersorb: a new high-performance polymeric separator for lead–acid batteries

Valérie Toniazzo[*]

Amer-Sil S.A., Zone Industrielle, L-8287 Kehlen, Luxembourg

Abstract

Given the recent improvements in valve-regulated batteries, lead–acid technology is nowadays considered to be well-suited for stationary power applications. Gel and absorptive glass mat (AGM) valve-regulated lead–acid (VRLA) batteries are complementary technologies and provide reliability and efficiency due to progressive optimization of the design and components. Special attention has been paid to the separation system, as its influence on the battery performance has been fully demonstrated. Polymeric calendered ribbed separators are traditionally used in gel VRLA batteries. For this technology, the separator is required to have high pore volume, optimized pore size, low acid displacement and low electrical resistance. It must also support efficient and controlled oxygen transfer. Glass–microfibre separators are presently the preferred material for AGM batteries. In addition to the properties listed for the polymeric type, glass–microfibre separators must not allow any drainage or stratification of the liquid electrolyte, and be able to retain their initial thickness after filling and during the battery life in order to sustain the initial compression in each cell. The Amersorb separator is well adapted to both technologies, for example: (i) the ribbed and corrugated patterns provide improved porosity (pore volume and pore-size distribution); (ii) the flat membrane is not only able to wick and retain the acid, but has also optimal compression properties (low compressibility and excellent springiness).
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1. Introduction

Despite the superior performance of alternative battery chemistries in terms of specific energy, lead–acid technology remains the most important secondary power source for stationary applications. This is because it represents the best compromise of specific energy, life, reliability, availability of raw materials, recycling of materials, and cost. Since their introduction on the market in the early 1970s, valve-regulated lead–acid (VRLA) batteries have been studied extensively [1–5] and have nowadays almost totally replaced the earlier flooded version in stationary power systems.

The basis of the functioning of VRLA batteries is now well understood. It contrast to flooded systems, where oxygen and hydrogen evolved, respectively, from positive and negative plates during overcharge are vented out so that water is lost, VRLA batteries are fitted with one-way valves that cause the gases to be retained inside the container. Because the system is closed, the oxygen generated at the positive plate during charging transfers to the negative plate where it is reduced ('recombined') back to water and therefore removes the need for water maintenance.

As the rate of oxygen transfer through liquid electrolyte is far too low, the sulfuric acid has to be immobilized in structures that have a certain percentage of free volume for gas transport. Two techniques are available for electrolyte immobilization: (i) absorption of acid in a glass–microfibre felt (absorptive glass material or AGM) at a saturation below 100% to allow free space for oxygen transfer; (ii) formation

* Tel.: +352 30 92 82 1; fax: +352 30 83 75.
E-mail address: amer-sil@amer-sil.com.
URL: http://www.amer-sil.com.

of a gel by dispersing 5–8 wt.% of silica with a high specific surface area in the acid to form a network that is susceptible to cracking under partial dry out to create gas paths. In both configurations, the separator is a critical component of the system. Therefore, besides the basic properties required from any separator in flooded systems (electronic insulation and protection against dendrites, promotion of ionic transfer, fixing of inter-plate spacing), the separators in AGM and gel VRLA batteries should have additional and very specific characteristics. Despite numerous studies of separators [6–11], the ideal separator has not been found for either AGM or gel batteries.

In order to compensate for some of the present weaknesses of separators, a microporous polymeric material, called Amersorb, has been recently developed by Amer-Sil S.A.: the flat Amersorb separator (no rib) is well suited for AGM batteries due to its improved compression properties, while the ribbed and corrugated patterns can be used in gel batteries and represent a step forward in the optimization of separators for stationary batteries.

2. Flat Amersorb separator for AGM batteries

More than in any other electrochemical cell, the separator in AGM versions of VRLA batteries is an important key of the battery success. Besides the basic insulation properties required from any separator system, the AGM separator, traditionally made of a mixture of coarse and fine glass–microfibres, has additional functions, as follows.

(i) It is the immobilizing agent for the electrolyte solution and the only acid reservoir between the plates. Therefore, pore volume and acid absorbency are important characteristics and the porous structure has to be optimized to provide a sufficient supply of acid to the plates at the desired rate, while avoiding any acid stratification and drainage or any dendrite growth.

(ii) As oxygen recombination is the basic function of sealed VRLA batteries, the oxygen transfer efficiency has to be optimized in order to avoid water loss, but its rate has to be controlled so that complete recharge of the negative plate can occur before oxygen reduction takes place.

(iii) Maintenance of the plate-group compression is also critical, as most of AGM separators have insufficient 'springiness' and have the tendency to lose contact with the plate. This results in capacity decay and accelerated end of battery life. In order to maintain electrical contact and adequately support the active material, the separator must have minimal shrinkage on wetting, be resistant enough to compression and, most importantly, have good resiliency.

(iv) For processing, the separator must have adequate tensile strength and elongation, i.e., 0.875–1.75 kN m^{-1} mm^{-1} and 0.5–10%, respectively, as reported by McGregor et al. [12].

(v) Finally, the separator has to be delivered at an acceptable price.

Many studies have been performed in recent years to try to reach this complex combination of properties and functions. Significant improvements have been obtained on 100% glass AGM separators by increasing the ratio of fine fibres [13], or by performing an optimal segregation of the fine and coarse fibres in dual-layer materials [14]. Other attempts to introduce non-glass components have also been initiated, like the addition of synthetic fibres [15], the incorporation of silica [16], or the development of novel separator materials (polymeric [17], ceramic [18]). Despite all these efforts, however, not one of the present-day separators meets the requirements perfectly, when both performance and cost are considered. In particular, most of the separators appear to allow expansion and softening of the positive active-material, unless they are assembled with compression forces that are impracticable in production with conventional equipment and battery containers. Moreover, due to a non-optimal pore structure, the internal oxygen cycle is most of the time overworked when batteries are ageing. This results in charging and heating problems. The new Amersorb separator has been developed mainly to overcome these compression and oxygen-transfer issues.

2.1. Basic properties

The Amersorb microporous membrane is a mixture of polymer and mineral fillers, which promote very specific compression properties and pore-size distribution. In addition, the basic physical parameters first requested from any separation system in AGM batteries have been checked and fulfilled.

First, the new membrane is readily wet by sulfuric acid. The results listed in Table 1 show that the new separator has excellent wettability, as an acid drop requires less than 1 s to be fully absorbed by the material. Also, the pore volume (around 80%) appears to be sufficiently high to enable the polymeric sheet to absorb adequate electrolyte. Vertical wicking tests have demonstrated that the amount of acid absorbed per unit volume of separator is in the range of 0.9–1.0 g cm^{-3}, which is comparable with the amount of acid retained in the fibrous structure of conventional AGMs. The electrical resistance of the sheet is between 50 and 90 mΩ cm^2, depending

Table 1
Physical properties of flat Amersorb separators (typical ranges)

Pore volume (%)	79–81
Pore size (μm)	
First peak	0.02–0.10
Second peak	2–15
Electrical resistance (mΩ cm^2)	50–90
Acid (1.28 relative density) absorption (g cm^{-3})	0.9–1.1
Puncture resistance (N mm^{-1})	3.5–7.5
Wettability (s)	<1
Tensile strength (kN m^{-1} mm^{-1})	0.90–1.40
Elongation (%)	10–15

on the sheet thickness, which is fully compatible with its use in AGM batteries. The mechanical properties, i.e., puncture resistance between 3.5 and 7.5 N mm^{-1} tensile strength between 0.9 and 1.4 kN m^{-1} mm^{-1}, and elongation from 10 to 15%, are more than acceptable for conventional manufacturing processes [12].

2.2. Pore-size distribution and influence on stratification, drainage, dendrite growth, and oxygen cycle

Standard AGMs made of a mixture of fine and coarse fibres generally have a pore size between 1 and 30 μm, depending on the ratio of fine fibres. Even with very high amounts of fine fibres, the specific surface-area of all-glass or hybrid (glass and organic fibres) separators does not exceed 2.5 m^2 g^{-1}, but has a strong impact on material cost. Most recent studies have demonstrated that the capillary forces developed inside these separators, even with increased content of fine fibres, are not high enough to avoid electrolyte drainage and acid stratification. Even the addition of silica has not solved efficiently the problem unless by the addition of significant amount of silica, which has an adverse influence on the mechanical properties of the separator. Drainage is harmful for electrical performance, as the heterogeneous distribution of electrolyte between the plates has not only a direct impact on the internal oxygen cycle, but also prevents the complete utilization of the active material since regions starved from electrolyte will be unable to participate fully in the discharge reaction. Moreover, this phenomenon has a tendency to increase as more water is lost during battery life. In the case of stratification, if the acid concentration gradient remains uncorrected, the situation will also lead to inefficient utilization of the active mass and give rise to a decreased capacity and aggravated local corrosion. Both drainage and stratification are attributed to a non-optimum pore-size distribution; more precisely, to a too large average pore size, even in the case of silica-loaded AGMs. In addition, dendritic growth can occur for the same reason and cause electrical shorts quite early in battery life.

In contrast to AGM separators, the Amersorb absorptive membrane has only half the pore volume made of pores of size 1–20 μm. As illustrated in Fig. 1, the second half of the porosity is due to small pores of between 0.02 and 0.10 μm. As a consequence, the capillary effect is so strong that any liquid is very well maintained inside the small pores, which limits electrolyte drainage. For the same reason, changes in acid density are expected to be much slower. In addition, due to the specific manufacturing process and resulting internal structure, the pores in the membrane are highly tortuous, which slows down or prevents dendritic growth even through the largest pores. The impact of the separator internal microstructure on the oxygen cycle is not only indirect, via a reduced electrolyte drainage, but is also direct on account of the unique pore-size distribution. Due to the occurrence of some larger pores against the positive plate, the acid will be
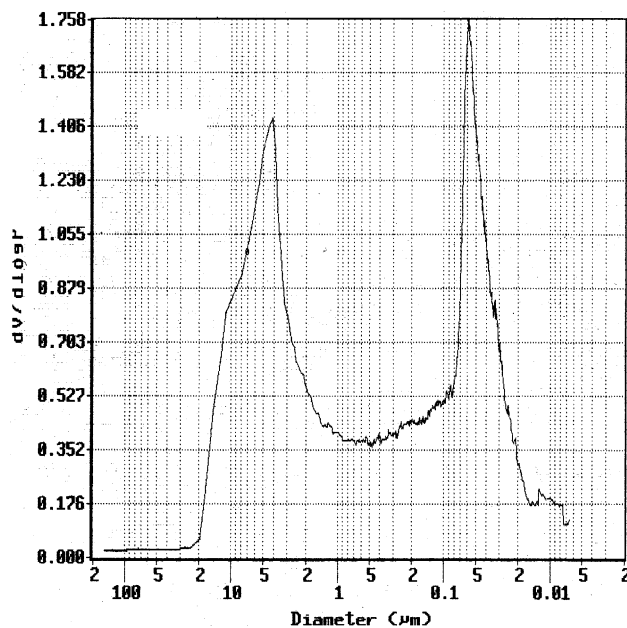

Fig. 1. Pore-size distribution of flat Amersorb separator measured by Hg intrusion porosimetry.

easily displaced by the evolved oxygen and gas transfer can be readily initiated. As a result, less oxygen will move along the positive plate and be vented out with concomitant water loss. Due to the high tortuosity of the pores, however, the oxygen transfer rate will be reduced. Moreover, the oxygen will experience more difficulty in displacing acid from the smaller pores, in which the capillary forces are higher. This will further reduce the transfer rate and therefore allow full recharge of the negative plate before oxygen-recombination takes place.

2.3. Improved compression properties

It has been well established that the plate volume changes during charge–discharge cycling. If the active mass is not restrained, the positive plate undergoes structural changes and this leads to shedding and premature capacity loss due to progressive loss of contact between adjacent particles and breakdown of the porous structure conductivity. The beneficial effect of compression of the positive active mass by the separator has been demonstrated by Hollenkamp [19] and confirmed for AGM batteries in a subsequent ALABC project [20]. If the separator is able to constrain the positive active-mass, then expansion and shedding are controlled and battery life is extended. Unfortunately, most conventional AGM separators are unable to fulfil this role.

In a first step, during the cell assembly when the plate group, which has been subjected to a defined initial dry compression, is further compressed and released in order to be inserted in the container, the AGM separators fail to come back to their initial compressed thickness, in order to maintain the desired initial dry compression. This has also been

Table 2
Compression loss, from 20 to 50 kPa and back to 20 kPa, of flat Amersorb separators compared with silica-loaded and hybrid AGM (values from ALABC project S3.2)

	Specific surface-area (SSA) ($m^2 g^{-1}$)	Compression (%) (20–50–20 kPa)
Amersorb	–	0
AGM (2 wt.% silica–8 wt.% organic fibres)	4.2	2.7
AGM (6 wt.% silica–8 wt.% organic fibres)	9.1	3.7
AGM (medium SSA–8 wt.% organic fibres)	1.4	4.2
AGM (high SSA–8 wt.% organic fibres)	1.8	4.1
AGM (very high SSA–8 wt.% organic fibres)	2.2	2.6

demonstrated for a series of hybrid AGM materials that contain 8 wt.% synthetic fibres and various amounts of fumed silica and fine fibres. In the compression test performed, the separator thickness loss has been measured at 20 kPa after a pressure load at 50 kPa (i.e., close to the insertion pressure in the assembly line). The results reported in Table 2 show that none of the tested materials was able to recover fully its 20 kPa thickness after a compression at 50 kPa. The thickness loss of a single separator sheet varied from 2.6% for the material with $2.2 \, m^2 g^{-1}$ specific surface-area to 4.2% for the material with $1.4 \, m^2 g^{-1}$ specific surface-area, while the silica-loaded samples lost more than 2.7% thickness. This is critical as these values have to be multiplied by the number of separator layers per cell, which leads to a significant loss in compression. Contrary to the AGM samples tested, the data in Table 2 indicate that the Amersorb membrane is perfectly able to resist a pressure load of 50 kPa with no variation in its thickness at 20 kPa (0% measured). This excellent springiness at medium compression forces assures a good initial dry compression in each cell.

In a second step, the separator should not shrink when acid is filled inside the container. Any kind of AGM material, even with synthetic fibres or mineral filler, does, however, have a tendency to contract on wetting. This gives rise to a further decay in compression during the battery-manufacturing process. As a consequence, very insecure calculations have to

be performed based on typical compression curves for each type of material used, in order to try to reach the desired wet compression. The thickness loss on wetting measured at 10 kPa for a standard AGM with $1.1 \, m^2 g^{-1}$ is shown in Fig. 2. More than 10% loss in thickness has been measured for this conventional 100% glass separator material. By contrast, no thickness variation could be measured for the Amersorb absorptive membrane under the same conditions. Other tests also have confirmed that the membrane does not shrink under wetting.

Finally, the general behaviour under pressure and release has to be optimized in order to accommodate variations in the volume of the active mass during battery life. Compared with standard AGM, the compressibility of the Amersorb separator is tremendously reduced. The thickness loss at 100 kPa is less than 7% whereas that for the standard AGM material with a specific surface-area of $1.1 \, m^2 g^{-1}$ is 40%. This feature enables the positive active-mass to be much better constrained. On the other hand and in contrast with other new polymeric separators [19], the Amersorb membrane is not completely incompressible, which would lead to manufacturing problems for the insertion of the plate group into the container. As illustrated in Fig. 2, the Amersorb can be slightly compressed even at moderate compression loads (4% thickness loss at 40 kPa, for example), which would facilitate the insertion of plate groups.
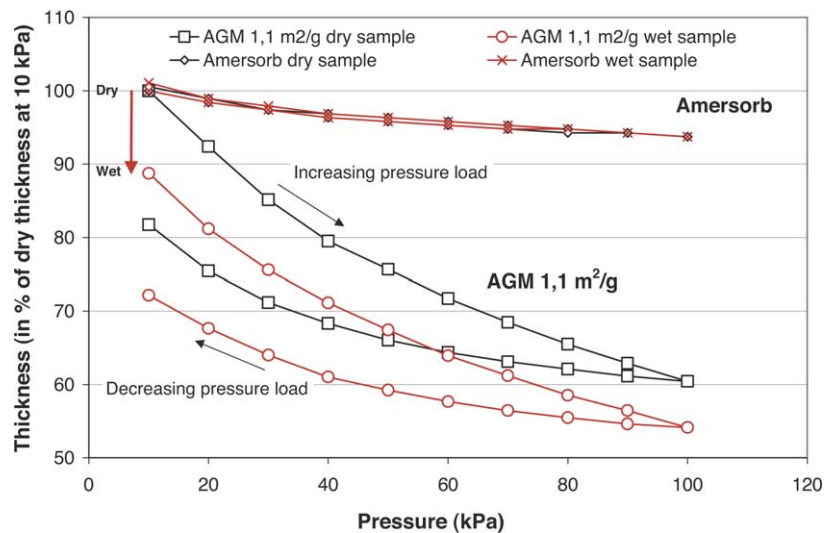

Fig. 2. Typical behaviour of Amersorb flat separator compared with conventional 100% glass AGM under increasing pressure from 10 to 100 kPa and back.

The decreasing pressure load curve for Amersorb (Fig. 2) also demonstrates the excellent resiliency of the membrane, as no thickness difference is observed at any compression level after a pressure load at 100 kPa. That is, the thickness measured at any compression when the pressure is decreased is comparable with the thickness measured at the same compression during increasing pressure. This is not the case with the AGM material tested, as illustrated by the large hysteresis of the compression curves, in both the dry and the wet stages.

In summary, the flat absorptive Amersorb membrane has improved compression properties, as it does not shrink on wetting and can avoid the positive active-mass expansion and shedding on account of its excellent springiness. In addition, the optimized pore-size distribution overcomes the drawbacks of other separators in that it regulates oxygen transfer and reduces electrolyte drainage, stratification and dendrite growth. As a result, the Amersorb membrane promises to be the candidate of choice for most AGM batteries.

3. Ribbed or corrugated Amersorb separator for gel batteries

Compared with AGM batteries, gel versions are less prone to electrolyte drainage and acid stratification. This is because of the efficient immobilization of acid in the silica gel network. The separator required to maintain the plate distance and prevent electronic shorts has historically been a rigid non-compressible material, which thus limits the occurrence of premature capacity loss associated with expansion of the positive active-material (so-called PCL2). The internal resistance of gel batteries is, however naturally increased compared AGM systems because the diffusion rate of ions through the viscous gelled electrolyte is generally one third of that in liquid electrolyte. Consequently, it is very important that the separator has an electrical resistance as low as possible in order to avoid an excessive internal resistance of the system. Additionally, the separator should have a low acid-displacement, since the fumed silica and the cracks in the gel structure already reduce the volume available for electrolyte. Due to these two major requirements, separators for gel batteries should have especially good wettability and high porosity. Also, the pore-size distribution has to be optimized in order to enable regulation of the oxygen transfer.

The corrugated version of the Amer-Sil ribbed separator, called 'Amergel' and developed a few years ago has already represented an important improvement for gel batteries compared with conventional micro-porous ribbed separators [21]. Nevertheless, a step forward has been made with the new Amersorb material. Initially developed in a flat pattern for AGM batteries, this product can also be produced with a ribbed or corrugated pattern. The next sections detail how the properties of this material can be beneficial to the performance of gel VRLA batteries.

3.1. Wettability, pore volume and ionic transfer

It is well understood that the pore volume of separators for gel batteries has to be maximized in order to insert as small an amount of insulating material as possible between the plates, and thus reduce the contribution to internal cell resistance. If this pore volume is not properly filled by electrolyte, i.e., if air bubbles remain trapped inside the pores of the separator, the ionic exchange will be reduced because ions are not able to travel through a gas phase, but only via more or less viscous ionic media (liquid or gelled sulfuric acid). Therefore, another key property for the separator, in addition to high pore volume, is to have a good wettability to enable the electrolyte to enter perfectly the whole pore volume, whatever the gel consistency.

It has already been mentioned above that the new material, unlike most conventional polymeric separators, has the ability to absorb around 1 g of acid per cubic centimetre of material, and even to wick the electrolyte by capillarity. The last row of data in Table 1 shows that the Amersorb material has an excellent wettability, as it has been measured that a drop of acid needs less than 1 s to be completely absorbed.

The pore volume measured for a series of ribbed and corrugated Amersorb separators with thickness ranging from 2.10 to 3.70 mm (typical for traction gel and stationary gel cells), is given in Table 3. All separators have a pore volume above 75%, and the corrugated versions of the Amersorb even reach porosities above 80% (for example 82.4% for the 2.10 mm separator). The electrical resistance measured for the different patterns is also extremely reduced compared with that of conventional separators of the same thickness, i.e., it does not exceed 120 mΩ cm^2 for a ribbed separator with 3.4 mm total thickness, and 62 mΩ cm^2 for a corrugated Amersorb separator with 3.70 mm thickness. For comparison, conventional ribbed separators with 3.70 mm thickness have an electrical resistance of the order of 200 mΩ cm^2.

The acid displacement is also tremendously reduced, as illustrated in Table 3 (only 219 ml m^{-2} for a 3.70 mm Amersorb separator with a 0.73 mm backweb).

The minimal values of electrical resistance and acid displacement measured for both Amersorb ribbed and corrugated separators illustrate perfectly the explanation given earlier that both wettability and high pore volume are required in order to minimize as far as possible the contribution of the separator to the internal cell resistance.

Given the above features, the electrical performance of cells assembled with the Amersorb material are expected to be very much improved, as supported by the findings of a previous study [9]. In that work, the authors studied the impact of the volume porosity on the electrical performance of 130-Ah gel VRLA cells. Laboratory tests conducted with separators of different porosity showed that cells assembled with low-porosity separators (50% volume porosity) delivered only 75 cycles, as opposed to 150 cycles for medium-porosity materials (70% volume porosity), and 500 cycles for high-porosity polymeric membrane (85% volume porosity). It was further

Table 3
Physical properties of Amersorb separators in ribbed and corrugated patterns (typical values)

	Ribbed Amersorb			Corrugated Amersorb		
Thickness (mm)	2.10	2.80	3.40	2.10	2.80	3.70
Backweb (mm)	0.57	0.66	0.60	0.42	0.66	0.73
Basis weight (g m^{-2})	371	452	490	246	358	390
Pore volume (%)	76.9	76.7	75.8	82.4	81.1	82.1
Pore size (μm)						
First peak	0.05	0.05	0.05	0.06	0.05	0.05
Second peak	10	7.5	4.5	5	4.5	5
Electrical resistance (mΩ cm^2)	89	101	120	35	65	62
Acid displacement (ml m^{-2})	207	252	273	140	200	219
Wettability (s)	1	1	1	<1	<1	<1

reported that separators with pore sizes between 1 and 10 μm gave much superior performance than conventional separators with a pore size below 1 μm (gel cells with separators of pore size between 1 and 5 μm achieved 675 cycles, and separators with pore sizes between 5 and 10 μm lasted for more than 1000 cycles, while only 425 cycles were obtained with standard separators of pore size below 1 μm).

As for the flat pattern, the pore-size distribution analysis of the ribbed and corrugated Amersorb separators shows a bimodal distribution with half of the volume porosity coming from small pores between 0.02 and 0.10 μm, and the second half from larger pores between 1 and 10 μm (Figs. 3 and 4). Based on the former study, the larger pores should act to increase the electrical output and life of stationary gel batteries. The proportion of bigger pores is higher for the corrugated Amersorb separators than for the ribbed version as shown in Figs. 3 and 4. It is therefore expected that the corrugated ver-

sion of the Amersorb separator leads to even better electrical performance.

3.2. Pore-size distribution and oxygen transfer

The prevailing theory for oxygen transfer from the positive to the negative plate in gel batteries is via micro-channels in gel structure. Initially, when the gel is formed by mixing silica and sulfuric acid, there is total liquid saturation. The gel structure is completely filled with the electrolyte, as is the active material, the gel|electrode interfaces and the separator (given that the wettability is good enough, as mentioned above).

Therefore, in the early stages of operation, the gel cell functions more like a flooded cell, as no path is initially available for gas transfer. Then, the gas evolved at the end of the charging period is forced to escape through the valve and
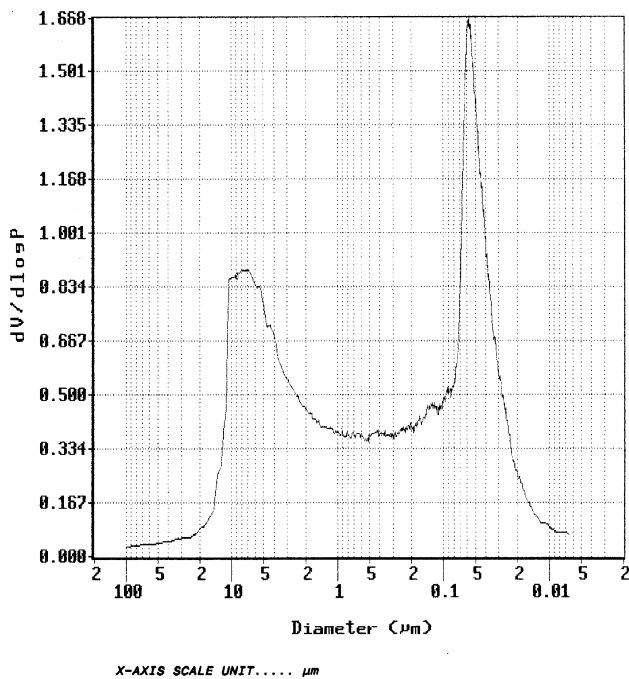

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Fig. 3. Pore-size distribution of Amersorb ribbed separator measured by mercury intrusion porosimetry.
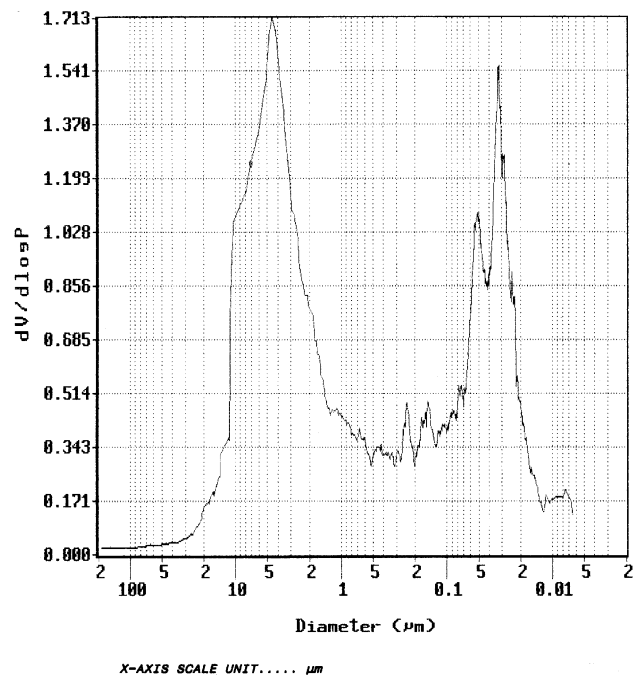

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Fig. 4. Pore-size distribution of Amersorb corrugated separator measured by mercury intrusion porosimetry.
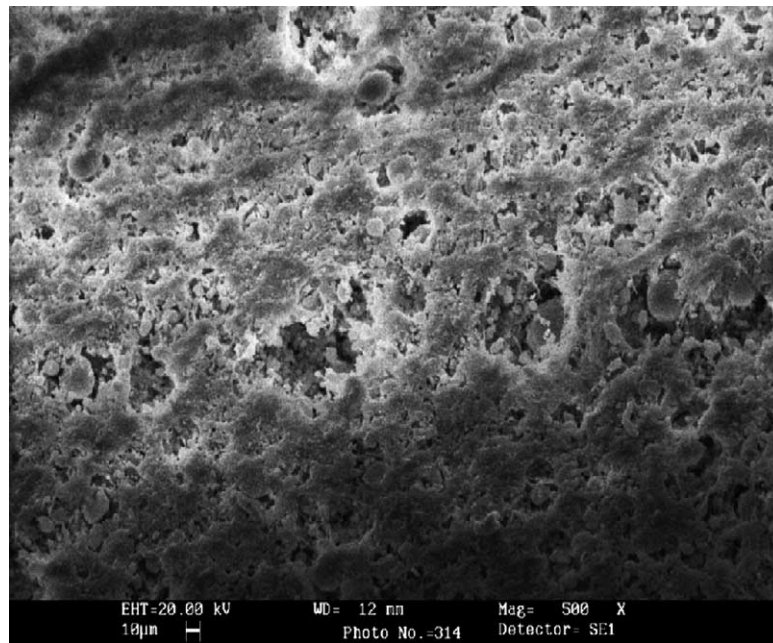
Fig. 5. Scanning electron micrograph of surface of corrugated Amersorb (magnitude 500×).

some water is lost. Depending on the gel consistency, leading to a weaker or a stiffer three-dimensional structure, the amount of cycles and water loss necessary to form micro-channels ('cracks') can vary. As soon as sufficient dry-out has occurred, the micro-channels have a chance to build up randomly through the gel. Since oxygen is liberated at the positive plate as a gas, it has a natural tendency to follow the path of least resistance and to travel preferentially upwards due to gravity. The active-material|gel interface is blocked, however, by the presence of an electrolyte film. Therefore, as soon as sufficient pressure is built up, oxygen gas has to make its way through the gel itself, via the micro-cracks.

Based on this model, it is obvious that the internal structure of the separator, especially its pore-size distribution, will be of first importance to optimize the oxygen transfer from the positive plate to the negative plate. The pore size and tortuosity, and the distribution of the pores at the separator surface, have to match as closely as possible the gel micro-channels to allow oxygen gas to continue its route from the gel micro-cracks on the positive side, to the gel micro-cracks on the negative side, via the separator interconnected pores. Since the process of gel cracking is totally random and the micro-channels probably vary from sub-micron to several microns size, it is very important for the separator surface to match this pattern.

On account of its bimodal pore-size distribution (Figs. 3 and 4), the Amersorb separator appears to be very well suited for an efficient, but well regulated, oxygen transfer, as the surface presents a wide range of opening sizes to match the gel micro-cracks (Fig. 5). In addition, the high tortuosity of the larger pores not only reduces the transfer rate of oxygen (thus allowing full recharge of the negative

plate), but also prevents the growth of dendrites through the separator.

4. Conclusions

The new Amersorb microporous material represents a step forward in the improvement of VRLA batteries. The flat version with enhanced fluid management and compression properties ameliorates the weaknesses in AGM batteries with respect to drainage and stratification, and to premature capacity loss due to the shedding of the positive active-mass. The ribbed or corrugated patterns have lower electrical resistance and acid displacement than any commercially available polymeric separators. These attributes raise the performance of gel batteries. It should also be noted that the unique manufacturing process for the Amersorb separator can allow the incorporation of antimony-scavenging additives [22] and thereby will permit a return to the use of Pb–Sb alloys for the positive grid for both gel and AGM types of battery. This would lead to easier grid casting and solve the strong intergranular corrosion problem encountered nowadays with Pb–Sn–Ca grids, but still with a reduced water loss because the antimony would be trapped inside the separator [23].

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