

# New low-antimony alloy for straps and cycling service in lead–acid batteries

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## Abstract

Lead–antimony alloys used for the positive grids in lead–acid batteries for cycling service have generally used antimony contents of 4.5 wt.% and above. Tubular batteries for cycling service that impart high compression of the active material to the grid surface via gauntlet use alloys with antimony contents as low as 1.5 wt.%. These batteries are generally employed in less-severe cycling service. Value-regulated lead–acid (VRLA) batteries can give good cycling service without lead–antimony in the positive grid, but require a high tin content and high compression.

The change in automotive battery positive grid alloys to lead–calcium–tin and the tin contents of VRLA positive grids and straps have dramatically increased the tin content of the recycled grid and strap lead in the USA, Europe, and Australia. The higher tin contents can contaminate the lead used for lead–antimony battery grids and generally must be removed to low levels to meet the specifications.

This study describes a low-antimony alloy that contains a substantial amount of tin. The high tin content reduces the rate of corrosion of low-antimony positive grid alloys, improves conductivity, increases the bond between the grid and the active material, and cycles as well as the traditional 5–6 wt.% antimony alloys employed in conventional flat-plate batteries. The alloy is also used as a corrosion-resistant cast-on strap alloy for automotive batteries for high temperature service, as well as for posts, bushings, and connectors for all wet batteries.

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## 1. Introduction

During the past 10 years, there has been a major change in the composition of the lead alloys used for grids in automotive batteries. A modification to the manufacturing processes has accelerated this change. Grids for automotive batteries have changed from low-antimony alloys to lead–calcium and lead–calcium–tin alloys. These alloys have very low freezing ranges and have found extensive use in new continuous grid production that involves continuous cast-expanded, continuous cast-rolled expanded, continuous cast, continuous cast-rolled, and continuous cast-rolled punched processes.

Lead–calcium–tin–silver alloys have been developed to cope with the higher under-hood temperatures encountered by automotive batteries in modern aerodynamic automobiles. Increased loads on the batteries have resulted in greater demands

for improved recharge performance from greater depths-of-discharge. There has also been an increased utilization of value-regulated lead–acid (VRLA) batteries for automotive service as the loads have increased due to additional power electronics and electrical equipment on modern vehicles. These changes have significantly increased the tin content of present-day batteries.

## 2. Tin content of battery alloys

The composition of lead–antimony, lead–calcium, and lead–tin alloys used for grids, straps, posts, and connectors in lead–acid batteries is shown in [Tables 1 and 2](#).

The tin content of lead–antimony alloys used for grid alloys for automotive and cycling service is as low as 0.05 wt.% and can be as high as 0.4 wt.%. Lead–antimony strap alloys generally utilize slightly higher tin contents of between 0.05 and 0.5 wt.%. In general, lead–calcium alloys contain substantially more tin than lead–antimony alloys. Lead–antimony alloys are strengthened by a matrix of second phase antimony and arsenic particles. Tin plays a minor part in the mechanical properties of

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Table 1  
Tin content of antimony batteries

Lead–antimony automotive alloys		
Grid alloys	1.5–2.8 wt.% Sb	0.05–0.3 wt.% Sn
Strap alloys	2.7–4.5 wt.% Sb	0.05–0.4 wt.% Sn
Lead–antimony cycling battery alloys		
Grid alloys	4.5–11 wt.% Sb	0.05–0.3 wt.% Sn
Strap alloys	2.7–12 wt.% Sb	0.05–0.3 wt.% Sn

these alloys. It is used primarily for mould fill, recovery from deep discharge of lower antimony alloys, and bonding of grids to straps.

Lead–calcium alloys in general have much higher tin contents than lead–antimony alloys used for the same product. It has been shown that lead–calcium alloys reach higher mechanical properties when strengthened by the formation of  $\text{Sn}_3\text{Ca}$  rather than  $\text{Pb}_3\text{Ca}$  intermetallic compound particles [1,2]. This improvement is particularly high when the alloys are rolled [3]. Tin added to lead–calcium alloys has been shown [4,5] to reduce the rate of corrosion of the positive grids. Other studies [6,7] have demonstrated that tin improves the ability of the positive plate to be recharged after the battery has been deeply discharged. Lead–tin alloys are used as grid materials and strap alloys for VRLA batteries where the negative effects of antimony on battery performance are most severe. The high tin content of lead-, calcium-, tin-, and silver-based alloys reduces the rate of corrosion at elevated temperatures [8].

### 3. Recycling lead–acid batteries

When lead–acid batteries are recycled by conventional rotary furnace technology, the major alloying element to be recovered is antimony. These conventional smelters lose the tin to the slag. This has resulted in the development of low tin contents for lead–antimony alloy specifications. In these alloys, all the tin must be added as pure metal.

As seen above, the tin content of scrap batteries is almost equal to the antimony content. If the tin is recovered in the bullion along with the antimony, the alloy cannot be utilized. The normal specifications for lead–antimony alloys require the tin to be removed to the normal specified low levels that are found in most lead–antimony alloys. When the tin is removed from the bullion, the dross is contaminated with antimony and other

Table 2  
Tin content of calcium batteries

Battery alloys		
- Cast positive	0.08–0.10 wt.% Ca	0.2–0.8 wt.% Sn
- Cast/expanded negative	0.07–0.01 wt.% Ca	0.0–0.5 wt.% Sn
- Cast calcium–silver	0.03–0.07 wt.% Ca	0.5–1.2 wt.% Sn
- Cast stationary	0.05–0.08 wt.% Ca	0.8–1.2 wt.% Sn
- Rolled (expand)	0.06–0.08 wt.% Ca	1.2–1.6 wt.% Sn
- Rolled (punch)	0.06–0.08 wt.% Ca	1.3–1.5 wt.% Sn
- VRLA spiral		0.6–0.8 wt.% Sn
- VRLA positive	0.04–0.08 wt.% Ca	0.8–1.2 wt.% Sn
- VRLA strap	0.00–0.03 wt.% Se	1.2–3.0 wt.% Sn

impurities to such an extent that the tin cannot be recovered for re-use in lead–calcium alloys.

Higher tin contents can be very beneficial for the properties and behaviour of lead–antimony alloys. A method has been developed to recover a large portion of the tin back into the lead–antimony bullion. A new antimony alloy has been developed to take advantage of the advantageous aspects of the higher tin content for straps, posts, bushings, connectors, as well as for positive grids for cycling service.

### 4. Battery straps and terminals

Lead–antimony alloys for battery straps in automotive and wet cycling batteries generally contain about 2.5–3.5 wt.% antimony. The metallurgical basis alloys for the cast-on strap process have been well characterized [9]. The alloys generally contain various amounts of arsenic, tin, copper, sulfur, and selenium as additional alloying elements to the antimony. In general, the alloys contain very low amounts of tin with the normal level being about 0.05–0.12 wt.% [10] to give a soft and easily welded material.

Bantz and Gryczkowski [11] have developed a complex method to evaluate the proper composition for battery strap alloys and conclude that the tin should be kept as low as possible and should not exceed 0.5 wt.% to prevent deleterious corrosion behaviour. The high corrosion of alloys with higher tin levels can be offset by the use of higher arsenic contents, but should not exceed 0.25 wt.%.

Klebanov et al. [12] have indicated that the copper should be restricted to low levels in die-cast terminals. The result of these authors indicated that higher tin contents up to 0.6 wt.% increase the resistance of the alloy to corrosion. The higher tin content also provides anti-oxidant protection to the polypropylene case or cover material adjacent to the lead alloy part.

#### 4.1. Higher tin contents in battery parts

Virtually all the alloys used for strap, post, terminal, bushings, and connectors in wet cycling batteries restrict the tin content to levels less than 0.4 wt.% due, in part, to economic reasons. The higher tin contents increase the cost of the alloy, but much of the caution over the upper limit of tin may be due to investigations of alloys with tin contents at too low a level. Alloys with 2 wt.% tin are used for straps in VRLA batteries and are extremely corrosion-resistant and suffer none of the problems experienced with higher tin alloys as cited in the literature regarding battery straps and parts.

In early work, Lander [13] found that tin additions to lead and lead alloys reduce the rate of bare metal corrosion to a significant degree. Pavlov [14] demonstrated that tin oxide doping of the corrosion layer produced thin layers that were more conductive than those formed on low-tin alloys. Nelson and Wisdom [15] showed that the addition of 0.7 wt.% or more tin could eliminate the passivation and provide exceptional corrosion resistance to VRLA positive grids. Simon et al. [16] and Bui et al. [17] have carried out definitive work on tin additions to lead and lead alloys

with regard to passivation, formation of PbO layers at the corrosion interface, and corrosion rate. It was concluded that tin additions of about 0.8–2.0 wt.% tin could yield the greatest benefit in reducing the corrosion rate and inhibiting the formation of passivating PbSO<sub>4</sub> layers by doping the corrosion film with tin dioxide.

From the above research, it can be concluded that a tin content of about 0.8 wt.% or above is required to decrease significantly the rate of corrosion of lead alloys. Previous investigations regarding strap, terminal, and bushing alloys had not increased the tin content sufficiently to take advantage of the reduction in the corrosion rate. Lower tin contents actually increase the rate of corrosion, but once the threshold is overcome, there is sufficient tin in the material to dope the corrosion product with tin oxides. Once such a layer is formed, it is much more resistant to the formation of sulfate layers on the surface of the battery part.

#### 4.2. Problems in strap welds

##### 4.2.1. Weld cracking

A new mode of failure of inter-cell welds is becoming more common with the increase in under-hood temperatures caused by the aerodynamic styling of modern automobiles. The failure results from cracks that propagate from the edges of the weld into the cross-section, as shown in Fig. 1. The formation of these cracks is due to the build-up of the corrosion layer on the flag or tombstone surface of the strap adjacent to the cell partition wall. The layer, which is composed mainly of lead sulfate, acts as a wedge to force the face of the tombstone away from the polypropylene cell wall. As its thickness increases with time, the corrosion layer exerts sufficient force to initiate and propagate cracks in the weld metal. Corrosion in the cracks compounds propagation and accelerates the mode of failure. At higher temperatures, the rate of corrosion of all components of the battery is increased. Positive grids have generally failed prior to the weld failures. Thus, with the introduction of more corrosion-resistant positive grid alloys, the

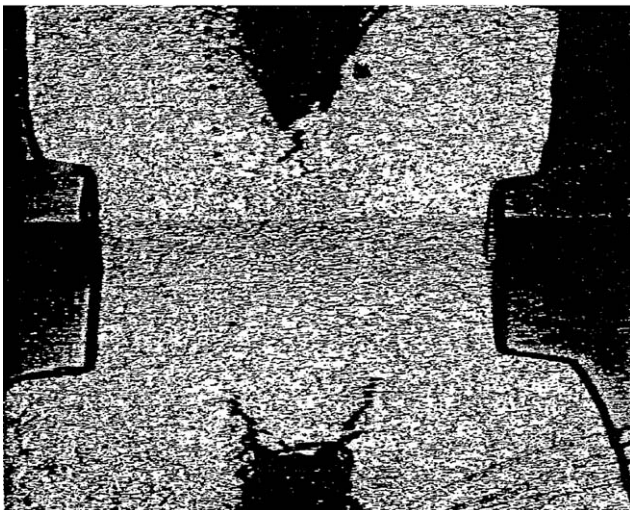


Fig. 1. Crack of weld due to build-up of sulfate.

weld corrosion has begun to play a significant role in battery failure.

##### 4.2.2. Build-up of corrosion product

In the area adjacent to the cell partition wall, the lead strap reacts with the available sulfuric acid to produce a layer of lead sulfate on the surface of the tombstone. Reaction with the acid depletes the area of acid and the product is water. As the localized concentration of water increases, the pH of the liquid rises and the lead becomes more soluble—particularly at elevated temperatures. Since this area is isolated, even more so because of the corrosion product, acid cannot diffuse readily into the region to raise the acid concentration of the remaining electrolyte. The high solubility of lead promotes rapid corrosion of the battery part to produce a mixed lead oxide–lead sulfate layer on the surface of the tombstone. As the thickness of the corrosion product increases, the layer exerts higher and higher stresses on the tombstone. Eventually, the tombstone will deform and cracks may propagate into the weld in a manner shown in Fig. 1.

##### 4.2.3. Strap corrosion at reduced potential

On the positive side of the weld, the part should be coated with lead dioxide. Because the acid is restricted in the area of the tombstone adjacent to the cell wall, the corrosion product can be a mixed lead sulfate–lead dioxide layer. At the strap/weld interface, the potential of the positive side can be reduced to a level below that required to keep lead dioxide both stable and protective. At this potential, the lead can rapidly be corroded to form the mixed corrosion product. This form of corrosion due to the reduced potential of the lead metal was described by Lander [18] and is shown in Fig. 2. At potentials slightly negative to the normal positive potential, the lead can be corroded rapidly. The corrosion product on the surface of the positive tombstone is seen under polarized light in Fig. 3. It consists of both lead dioxide and a large amount of lead sulfate.

In a similar manner, the potential of the negative part of the weld can become more negative than the protective value and therefore a corrosion film of lead sulfate will develop. The sulfate layer can become quite thick and can exert as much, or

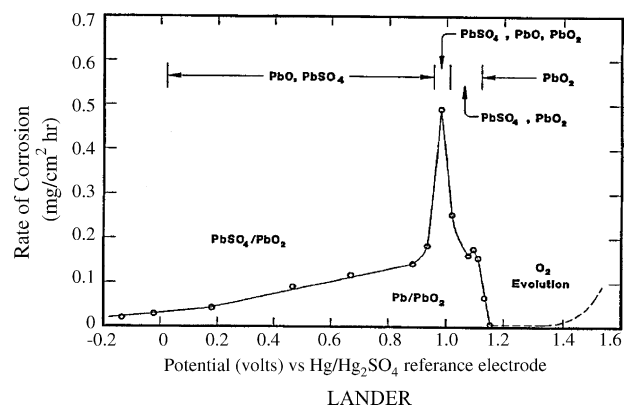


Fig. 2. Rate of corrosion of lead as function of electrode potential [18].

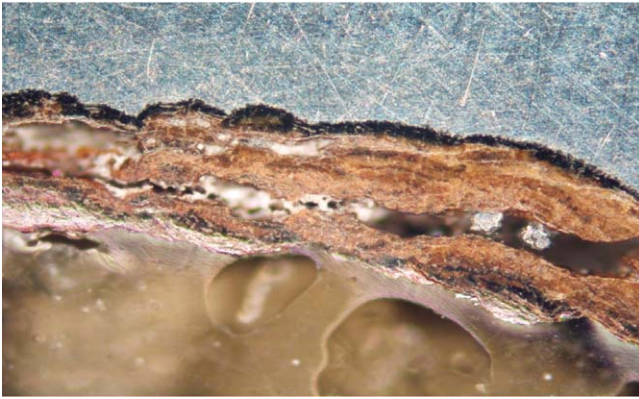


Fig. 3. Corrosion product of positive strap under polarized light. Magnification 70 $\times$ .

more, stress as the mixed lead sulfate–lead dioxide layer on the positive side of the weld. A very thick corrosion layer of lead sulfate on the surface of a negative tombstone is shown in Fig. 4. The surfaces of the positive and negative portions of the strap in the region near the weld are coated with a layer of lead sulfate, as seen in Fig. 5. Thus, the weld is beginning to crack.



Fig. 4.  $\text{PbSO}_4$  corrosion product on negative strap. Magnification 70 $\times$ .



Fig. 5. Growth of corrosion product on both sides of plastic cell wall. Magnification 35 $\times$ .

#### 4.3. Grain structures of straps

Many companies restrict the use of nucleants for straps. For many years and in many parts of the world, copper and sulfur have been used to reduce the size of the primary dendritic grains that are formed in the solidification process. When the grains are large, the cast straps are prone to cracking and the welds are brittle because they, too, contain large grains. Straps with large grains often exhibit porosity due to trapped gases that segregate at the grain boundaries. Large grains also tend to form depressions at the intersection of two grains. Electrolyte can be isolated in a depression at the surface of the tombstone and promote a corrosion cell reaction on either the positive or negative side. The varying grain size can produce non-uniform mechanical properties throughout the strap. If the tin content of the alloys is increased above about 0.4 wt.%, the grain-refining effects of copper and sulfur are dramatically reduced, as demonstrated in Fig. 6. The photograph shows large, directional grains in a strap that contains 0.9 wt.% tin with 0.01 wt.% sulfur, and 0.09 wt.% copper.

By contrast, lead–antimony alloys modified by selenium are unaffected by the addition of tin and produce fine grains, even at very high tin contents. The use of selenium for both battery grids and straps has been examined extensively [19–23]. The structure of a lead, 3 wt.% antimony, 1 wt.% tin, 0.1 wt.% arsenic alloy modified with 0.02 wt.% selenium alloy (the TASC alloy) is extremely uniform for cast-on straps. The uniform grain structure not only prevents cracking, but also imparts ductility to the part. The latter is beneficial for welding and resists stresses from corrosion of the tombstones, as seen in Fig. 7.

#### 4.4. Benefits of high tin to reduce cracked straps

Tin additions to lead inhibit the oxidation of lead to lead oxide. At high pH values, lead can first be oxidized to lead hydroxide under either positive or negative conditions. The lead oxide or hydroxide can react with sulfuric acid to form lead sulfate. The lead–water–sulfate pH versus potential diagram given by Pourbaix [24], reproduced in Fig. 8, shows the

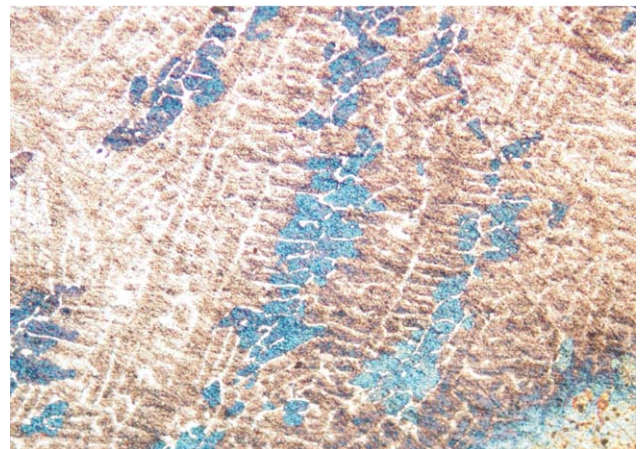


Fig. 6. Large grain size in a lead–3 wt.% antimony alloy with 0.9 wt.% tin, copper and sulfur nucleants. Magnification 140 $\times$ .

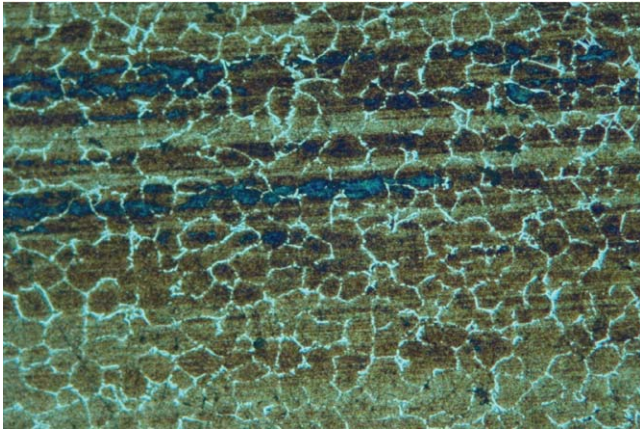


Fig. 7. Fine uniform grain structure of TASC alloy strap with selenium. Magnification 140×.

wide range for the formation of lead oxide and subsequent lead sulfate.

In a similar manner, tin is first oxidized to tin oxide. This compound is not, however, stable and can be fully oxidized to tin dioxide. Lead oxide or lead hydroxide can be reduced back to metallic lead by the tin oxide, which is oxidized to tin dioxide via a simple redox process [20]. In this reaction, the lead oxide or hydroxide becomes progressively thinner and thinner and more enriched in tin dioxide. On increasing the tin content of the strap metal to levels of 0.8–2.0 wt.%, there is sufficient tin to reduce dramatically the formation of lead oxides and, subsequently, lead sulfates.

The tin dioxide corrosion product dopes the lead corrosion product at a sufficient concentration to cover the surface with a passive layer. This protects the metal over the range of pH and potentials predicted by the Pourbaix diagram [24] given in Fig. 9. Once the tin dioxide is formed, it is stable and no longer reacts in the corrosion layer. Tin dioxide also makes the corrosion layers more conductive than insulating. Insulating layers seal off corrosion cells and promote corrosion beneath the layer.

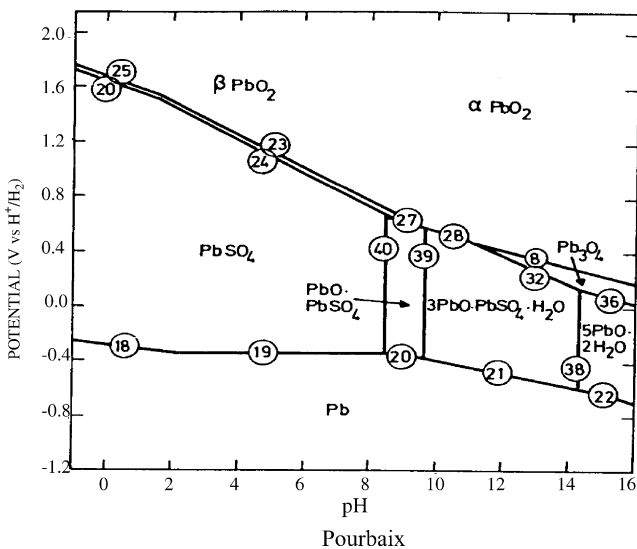


Fig. 8. Simplified potential-pH diagram of Pb-H<sub>2</sub>O-SO<sub>4</sub><sup>2-</sup> system at 25 °C.

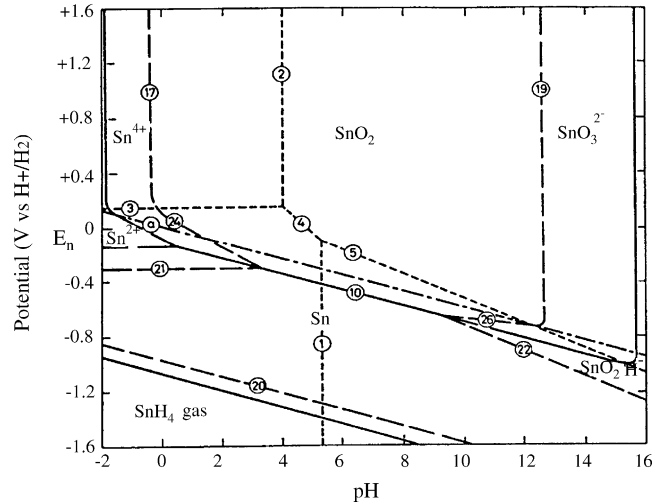


Fig. 9. Potential-pH diagram of Sn-H<sub>2</sub>O-oxide system at 25 °C.

If the tin is held at sufficiently high levels, virtually no lead sulfate is formed at the surface of the tombstone. Without corrosion, there is less stress on the weld area to initiate cracks. The surface of a TASC alloy weld after 4500 hot SAE J240 cycles is shown in Fig. 10. There is negligible corrosion product on either the positive or the negative surface of the part in the weld area.

A high tin content increases the amount of eutectic liquid present at solidification of the alloy. This, in turn, allows the strap to remain liquid for longer times at the eutectic temperature and thus produce an improved bond between the grid lug and the strap. The combination of greater amounts of eutectic material and selenium produces a very uniform strap for reduced corrosion and more uniform welding, as seen in Fig. 11.

#### 4.5. Strap welding

In the welding process, the tombstone-shaped parts of both the positive and negative straps are simultaneously pushed or extruded through a hole in the cell wall which divides one cell



Fig. 10. Weld area of TASC strap showing no corrosion. Magnification 35×.

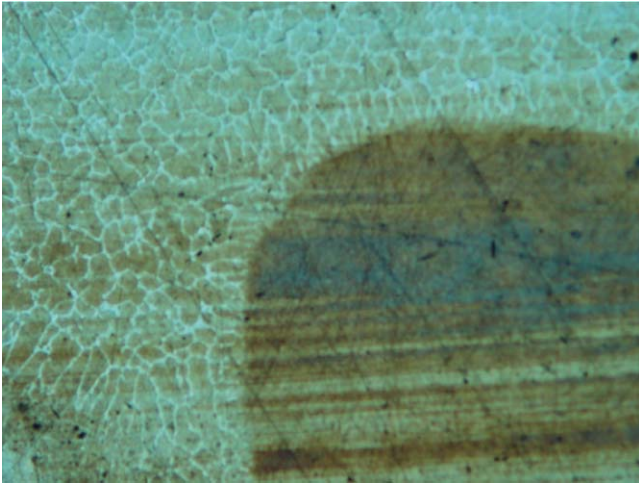


Fig. 11. Bond between Pb–Ca grid and TASC 3 wt.% antimony strap showing excellent grain structure. Magnification 70 $\times$ .

Table 3  
Composition of TASC alloy

Antimony	2.7–3.3 wt. %
Tin	0.8–1.2 wt. %
Arsenic	0.05–0.2 wt. %
Selenium	0.015–0.025 wt. %
Copper	0.020 wt. % max.

of the battery from another. Once through the hole, the extruded parts are melted and fused together by the application of an electrical current. The melted material forms an electrical path between cells and also seals the hole between the two cells to prevent leakage of acid and subsequent stray currents between cells. The integrity of the weld is controlled by the uniformity and ductility of the strap metal, the ease of melting, and the amount of eutectic liquid present. All are enhanced by the addition of tin and a selenium nucleant. The composition specification for the TASC alloy is given in Table 3.

Normally, tin additions to lead–antimony alloys increase the yield strength and make the alloy more brittle. With addition of selenium and lowering of the arsenic content, a high tin alloy can be modified to produce high ductility as well as sufficient mechanical strength. The mechanical properties of the 3 wt.% antimony TASC alloy with high tin modified with selenium are given in Table 4. The material maintains nearly the same mechanical properties for the first 24 h and this is beneficial for uniform welding after slight time delay. The alloy has a low ratio of yield strength to ultimate tensile strength, which is a

Table 4  
Mechanical properties of TASC alloy as a function of time

Aging time (h)	Tensile strength (N mm <sup>-1</sup> )	Yield strength (N mm <sup>-1</sup> )	Elongation (%)	Toughness YS/TS
1	37.9	24.0	30	0.63
2	37.9	24.5	30	0.64
8	37.9	24.8	29	0.65
24	39.3	26.2	28	0.67
120	41.7	28.8	23	0.70

measure of the toughness of the metal or the ability of the metal to deform without cracking. The toughness of the alloy significantly enhances the resistance of the weld to cracking and to corrosion (Table 4).

## 5. Bushings and connectors

The TASC alloy has sufficient tin to keep the surfaces of the cast or forged parts extremely bright and shiny. The alloy prevents the formation of black posts during storage and processing. A high tin content also improves the connection between the battery terminal and cable clamp. Any corrosion product formed on the post surface is doped with tin oxide, which serves to maintain a conductive path through to the clamp and prevent a high resistance layer that might retard recharge. In a similar manner, connections between cells in deep cycle traction cells are enhanced by the high tin content.

## 6. High-tin, low-antimony alloys for cycling service

The 3 wt.% antimony TASC alloy can be used as the positive alloy for grids of wet batteries for cycling service applications such as golf carts, fork lift trucks, sweeper scrubbers, locomotives, and mining vehicles. In the past, positive grids have been composed of 5–6 wt.% antimony with small amounts of tin and arsenic as nucleants to prevent cracking and penetrating corrosion. In general the higher the antimony content, the higher is the rate of corrosion of the grid and thus the greater is the transfer of antimony to the negative plate during service. As more antimony is transferred, the rate of gassing increases and the rate of water consumption increases. Preferably, the grid would be produced from a lower antimony alloy to reduce: the corrosion of the positive grid, the transfer of antimony, the gassing rate, and water loss. For flat-plate batteries, however, the antimony content must be maintained above 4.5 wt.% to enable the positive grid to recover from deep discharge.

### 6.1. Battery construction

A 3 wt.% antimony alloy, which is commonly used for cast-on strap alloys, can be utilized for positive grids in cycling lead–acid batteries through the addition of a relatively high percentage of tin. Batteries with the high-tin, low-antimony TASC alloy for the positive grids cycle as well as those with a 5–6 wt.% antimony alloy. An alloy of 3 wt.% antimony, 1.06 wt.% tin, 0.01 wt.% arsenic, and 0.015 wt.% selenium was cast into normal positive grids for application in golf-carts

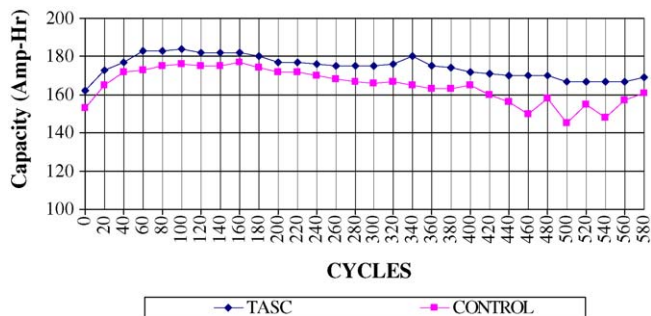


Fig. 12. TASC alloy evaluation, discharge: 25 A–10.5 V; charge: 25 A, 15.5 V, and 10 h.

batteries. The grids were processed in the same manner as normal grids of 5.6 wt.% antimony, 0.3 wt.% tin, 0.2 wt.% arsenic, and 0.022 wt.% selenium, which served as a control. The same batch of paste was used to paste both series of grids and the plates were cured and formed in the same manner.

### 6.2. Battery testing

The batteries were tested using the BCI deep-discharge regime, i.e., discharge at 25 A to 10.5 V, recharge at 25 A at 15.5 V for 10 h at 25 °C. The control batteries had an original capacity of 150 Ah. The capacity gradually increases to about 175 Ah after the first 50 cycles and remains at that level through 180 cycles when it begins a slow decline in capacity. Batteries containing low-antimony, high-tin TASC positive grids have a higher initial capacity and also an increasing capacity over the first 50 cycles to reach about 185 Ah. These batteries also maintain this capacity through about 160 cycles. At this point, the capacity begins to decline. It was expected that the decline would continue as cycling proceeded because the grids do not contain sufficient antimony. Instead, the capacity levelled out at about 175 Ah, remained at this level through 350 cycles, and maintained higher capacity than the control through 600 cycles. The test results are shown in Fig. 12.

The higher capacity is believed to be due to the higher conductivity of the low antimony, high tin, TASC grids. The high tin content dopes the positive active-material and thereby improve the recharge performance and results in the maintenance of capacity.

## 7. Conclusions

The addition of tin to the positive grids of lead–calcium and battery grids have dramatically increased the tin content of auto-

motive and VRLA batteries. The tin has been lost to the slag in most battery recycling processes. Where the tin can be recovered, it cannot be utilized back into lead–calcium alloys because of the high impurity content of the recovered bullion. When recovered in a lead–antimony alloy, the tin may be utilized to produce low-antimony–high-tin alloys with unique properties for straps, bushings, and terminals. When employed for positive grids in cycling batteries, the alloy can give higher capacity, lower gassing rates, less water usage, and faster recharge than normal high 5–6 wt.% antimony alloy grids with the same or better life.

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