



Effect of boric acid on the corrosion behavior of Pb-Sb alloy grids in lead-acid battery electrolyte solution

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ABSTRACT

Positive grid corrosion in lead-acid batteries (LABs) remains one of the principal factors limiting battery service life. This study investigated the influence of boric acid on the corrosion behavior of Pb-Sb 2 wt% alloy grids in a sulfuric acid electrolyte ($d = 1.27 \text{ g.cm}^{-3}$) by cyclic voltammetry (CV), linear sweep voltammetry (LSV), and Raman spectroscopy. Boric acid markedly attenuated the electrochemical activity of the alloy, as evidenced by lower CV peak currents and reduced corrosion kinetics. Raman spectroscopy confirmed the formation of PbSO_4 and mixed sulfate/oxide corrosion products after cycling. Notably, the blank and boric-acid-containing samples exhibited identical peak positions but substantially different intensities, indicating that H_3BO_3 suppresses corrosion-film growth without altering the underlying corrosion chemistry. After 24 h, the corrosion-inhibiting effect of the boric acid additive becomes clearly evident compared to the additive-free sample: the corrosion current density $J_{\text{corr}} = 6.40 \times 10^{-8} \text{ A cm}^{-2}$ is reduced by a factor of two, while the polarization resistance $R_p = 428540 \Omega$ increases by 2.6 times relative to the sample without an additive. Collectively, these results demonstrate that H_3BO_3 acts as an effective electrolyte additive, forming a thinner and more stable protective layer on Pb-Sb grids and thereby enhancing their corrosion resistance in a lead-acid battery.

Introduction

Lead-acid batteries remain one of the most important commercial energy storage systems owing to their low cost, high safety, ability to deliver high current, and long-established manufacturing technology [1-4]. However, their operational durability is still limited by several concurrent degradation processes in concentrated H_2SO_4 electrolyte, among which positive grid corrosion, deterioration of the PbSO_4 layer, and water loss due to hydrogen and oxygen evolution are the primary factors responsible for cell failure [5-8]. In practical battery systems, Pb-Sb alloy grids are widely used because of their good mechanical strength and castability, and they improve battery charging efficiency

[9,10]. Over the years, the new requirement for maintenance-free technology in transportation or stationary applications was met with the advent of VRLA technology and an improvements in the grid alloy: the reduction of the Sb content (<2.5 wt%) in the alloy used as the grid [5].

Corrosion of Pb-Sb grids in H_2SO_4 electrolyte proceeds through the oxidation of lead to PbO/PbO_2 and the formation of intermediate product layers containing PbSO_4 , PbO_x , and phases associated with the metal grid substrate [1,3,9]. The growth of this corrosion layer can significantly alter charge-transfer resistance, the transport of H^+ and SO_4^{2-} ions, and the protective or destructive nature of the surface film toward the underlying metal substrate [11]. Therefore, an effective

electrolyte additive should not only reduce corrosion rate, but also maintain or improve electrode reaction kinetics, suppress water decomposition, and avoid excessive loss of capacity or charge acceptance [12].

In recent years, inorganic additives in electrolyte solutions have been regarded as an effective and economical approach to improving the electrochemical behavior of lead-acid batteries [3]. Numerous studies have shown that inorganic acids and salts, such as H_3PO_4 , H_3BO_3 , SnSO_4 , Na_2SO_4 , and complexing agents such as Na_2EDTA , can modify ionic activity in the electrolyte, regulate PbSO_4 crystal morphology, increase hydrogen and oxygen evolution overpotentials, or form protective surface layers that slow down corrosion. In particular, H_3BO_3 has been reported to increase HER and OER overpotentials and to promote the formation of dense borate-containing products, thereby improving the corrosion resistance of lead grids. Romero et al [13] investigated the effects of different boric acids on the electrochemical properties and confirmed a decrease in the oxide reduction charge (23 %) and an increase in the resistance of the corrosion process were found by the addition of 0.50 wt% H_3BO_3 , while subsequent research also considered the boric acid as a promising additive after studying different mixed additives [14,15]. This concentration was therefore adopted here, as it balances effective borate-based protection against the solubility limits and ion-transport issues that arise at higher H_3BO_3 levels.

In this research, a systematic investigation of the effects of the inorganic additive H_3BO_3 on the corrosion behavior of a Pb-Sb 2 wt% alloy clarified the interaction mechanism between the electrolyte and the alloy surface. It provided a scientific basis for selecting suitable additive formulations to improve the service life and reliability of lead-acid batteries.

Experimental

Materials and chemicals

The commercial Pb-Sb alloy (2 wt%) was obtained from the PINACO battery factory in Dong Nai, Vietnam. The molten metal was cast at 450°C into an iron mold maintained at 220°C under ambient air to produce cylindrical specimens with a diameter of 10 mm and a height of 5 mm. Ethanol (EtOH), Boric acid (H_3BO_3), >99%, Macklin, China; H_2SO_4 , > 99.5%, Merck, Germany.

Methods

Inductively coupled plasma optical emission spectrometry

The metallic components in the lead alloys under investigation were analyzed using ICP-OES Spectrolab emission spectroscopy, model LAB M12.

Electrochemical measurements

Electrochemical tests were carried out using a conventional three-electrode cell connected to an Autolab PGSTAT204 potentiostat. The Pb-Sb alloy embedded in epoxy resin, with an exposed geometric area corresponding to $r = 5 \text{ mm}$ (area surface 0.785 cm^2), was used as the working electrode; a mercury sulfate electrode (MSE) served as the reference electrode; and a $1.0 \text{ cm} \times 1.0 \text{ cm}$ platinum sheet was used as the counter electrode. The electrolyte was H_2SO_4 with a density of 1.27 g cm^{-3} , prepared either without an additive or with 0.5 wt% H_3BO_3 .

Before each measurement, the electrode surface was mechanically polished using 800-, 2000-, and 3000-grit SiC papers, rinsed with anhydrous ethanol and double-distilled water, and then subjected to cathodic pre-reduction at -1.3 V for 10 min to remove surface oxides. All electrochemical measurements were performed at room temperature. Cyclic voltammetry (CV) was first conducted in the potential range of -1.7 to 1.7 V at a scan rate of 5 mV s^{-1} , and additional CV tests in the anodic working region of 0.7 to 1.7 V were performed at 10 mV s^{-1} for 50 cycles to simulate the operating condition of the positive grid.

Linear sweep voltammetry (LSV) was performed in accordance with ISO 17475:2005 at a scan rate of 1 mV s^{-1} . Open-Circuit Potential (OCP) stabilization time is 30 min, over a potential window of $\pm 250 \text{ mV}$ relative to the OCP. The corrosion potential (E_{corr}), corrosion current density (J_{corr}), and polarization resistance (R_p) were determined by Tafel extrapolation using NOVA 2.1.9 software after immersion periods ranging from 30 min to 48 h.

Raman spectroscopy

Raman spectra of the Pb-Sb alloy before CV cycling and after CV cycling were collected using a Thermo Scientific DXR3 Raman Microscope, a 532 nm excitation laser, a $50 \mu\text{m}$ slit aperture, a laser power of 4 mW, and a $50\times$ objective lens. Measurements were performed on the pristine alloy surface and on the electrodes after electrochemical cycling in H_2SO_4 with and without H_3BO_3 . The Raman microscope provides high-sensitivity confocal Raman analysis suitable for surface characterization of corrosion products and electrochemically formed layers.

Results and discussion

Analysis of metal content in PbSb alloy

The ICP-OES results (Table 1) confirmed that the alloy composition is consistent with the declared Pb-Sb 2 wt% commercial grade commonly used for lead-acid battery grids.

Table 1: Percentage content of major metals in lead alloys in the study.

Meas.	Pb Conc.	Sn	Sb	Ag
1	97.859	0.087	1.831	0.024
2	97.837	0.097	1.847	0.024
3	97.859	0.089	1.848	0.024
4	97.845	0.087	1.835	0.023
W. Min	97.850	0.100	1.835	0.023
<x>	97.850	0.090	1.838	0.023
W. Max	100	0.150	2.250	0.025

Meas.	Bi	As	Se	Fe
1	0.024	0.123	0.030	0.003
2	0.026	0.116	>0.031	0.002
3	0.024	0.116	0.030	0.002
4	0.024	0.134	0.031	0.002
W. Min	0.024	0.14	0.031	0.002
<x>	0.024	0.122	0.031	0.002
W. Max	0.030	0.180	0.025	0.005

The Pb content is approximately 97.86 wt%, while the Sb content averages 1.84 wt%, both falling within the specified manufacturing range for 2 wt% Sb grid alloys. The Sn level is very low (~0.09 wt%), and minor impurity elements such as Ag, Bi, As, Se, and Fe remain below their respective maximum specification limits. This composition indicates that the corrosion and electrochemical behaviors observed in the present work can be attributed primarily to the Pb–Sb matrix, without significant interference from abnormal impurity concentrations.

Cyclic voltammogram

The cyclic voltammetry (CV) results of the Pb–Sb alloy containing 2 wt% Sb, recorded in H_2SO_4 electrolyte with a density of $1.27 \text{ g}\cdot\text{cm}^{-3}$, were obtained for both the additive-free electrolyte (Blank) and the electrolyte containing 0.5% boric acid (BA). After 5 cycles in the potential window from -1.7 to 1.7 V, scan rate of 5 mV/s (Error! Reference source not found.), and after 50 cycles in the range from 0.7 to 1.7 V, scan rate 10 mV/s (Fig 1b) corresponding to the operating conditions of the positive grid in lead-acid batteries.

The cyclic voltammetry results exhibited the typical electrochemical characteristics of the lead-acid system, with multiple oxidation and reduction processes corresponding to the phase transformations among Pb, PbSO_4 , PbO, and PbO_2 . Two additional processes, hydrogen evolution (H) and oxygen evolution (O), were also observed at the extremes of the potential window. The cathodic branch exhibited four characteristic peaks:

- c1 ($\text{PbSO}_4 \rightarrow \text{Pb}$, -1.04 V),
- c2 ($\text{PbO} \rightarrow \text{Pb}$, -0.8 V),
- c3 ($\text{Sb}^{x+} \rightarrow \text{Sb}$, -0.64 V),

- c4 ($\text{PbO}_2 \rightarrow \text{PbSO}_4$, 1.06 V).

The anodic branch exhibited four corresponding peaks:

- a4 ($\text{Pb} \rightarrow \text{PbSO}_4$, -0.96 V),
- a3 (dissolution of Sb, -0.37 V),
- a2 ($\text{Pb} \rightarrow \text{PbO/PbO}_2$, 1.02 V), and
- a1 ($\text{PbSO}_4 \rightarrow \beta\text{-PbO}_2$, 1.07 V).

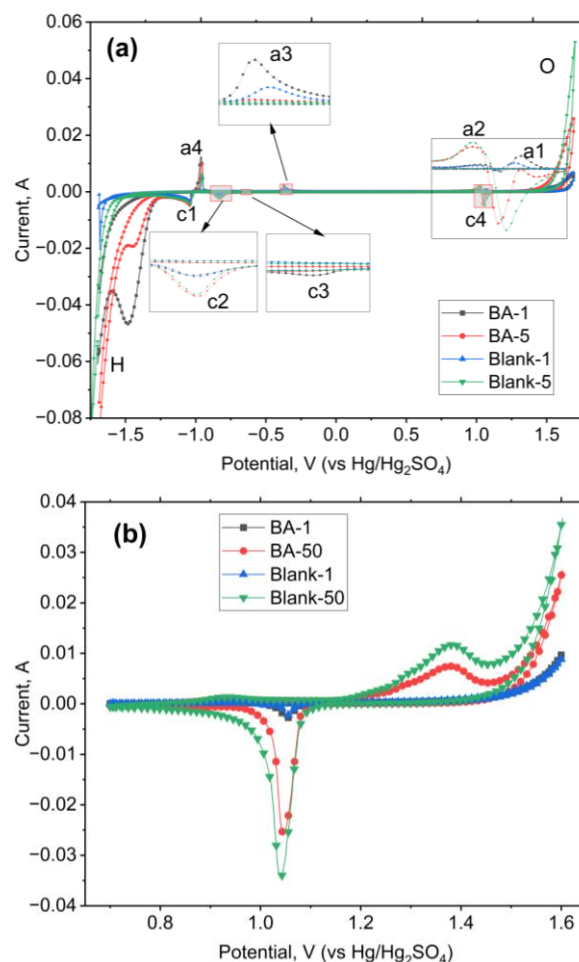


Fig 1: Cyclic voltammograms of the Pb–Sb electrode in blank and BA (0.5 wt% H_3BO_3) electrolytes: (a) full potential window (-1.7 to 1.7 V, 5 mV s⁻¹, 5 cycles); (b) positive-grid operating range (0.7 to 1.7 V, 10 mV s⁻¹, 50 cycles).

The characteristic cathodic peaks correspond to the reduction processes $\text{PbSO}_4 \rightarrow \text{Pb}$ (c1) and $\text{PbO} \rightarrow \text{Pb}$ (c2), $\text{PbO}_2 \rightarrow \text{PbSO}_4$ (c4), while the anodic peaks correspond to the oxidation processes of $\text{PbSO}_4 \rightarrow \beta\text{-PbO}_2$ (a1), $\text{Pb} \rightarrow \text{PbO/PbO}_2$ (a2), $\text{Pb} \rightarrow \text{PbSO}_4$ (a4), and the dissolution of Sb and the reduction of Sb ions in the electrolyte solution. These characteristic peaks are consistent with the electrochemical behavior of the Pb–Sb alloy used as the electrode backbone, in which Sb both influences the matrix microstructure and participates in surface redox processes consistent with previous studies [9,12,13,16]. The oxidation and reduction

peaks increased as the number of scans increased due to enhanced WE surface [13]. Focusing on the grid corrosion process, which primarily occurs at the positive grid [3,17], CV scans were performed over the positive electrode potential range of 0.7-1.7 V at a scan rate of 10 mV.s⁻¹ (Error! Reference source not found.)

The blank sample exhibits higher peak currents, indicating greater surface conversion and more pronounced growth of electrochemically active corrosion products. In contrast, the lower peak currents observed in the H₃BO₃-containing electrolyte suggest that boric acid suppresses the reaction kinetics and limits the development of PbSO₄/PbO_x films. This behavior is consistent with the inhibitory role of boric acid, which is known to increase the evolution overpotentials and reduce grid corrosion in lead-acid systems [12,14,15].

The pristine Pb-Sb alloy exhibits only weak, broad Raman features, confirming that the metallic substrate is weakly Raman-active and that the surface state dominates the measured signal. After CV cycling in H₂SO₄, both the blank and H₃BO₃-containing samples exhibit the same main Raman bands (Fig 2), indicating that the corrosion products are chemically similar in both electrolytes, but their intensities differ substantially.

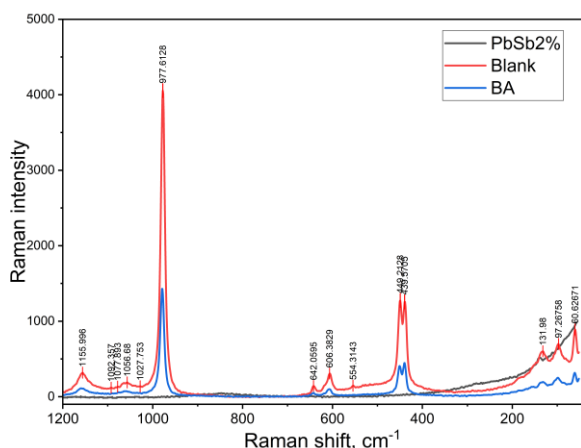


Fig 2: Raman spectra of PbSb alloy samples before CV scanning and after 50 CV cycles in additive-free (blank) and boric acid 0.5 wt% additive (BA) electrolyte solutions.

The intense band at approximately 977 cm⁻¹ is assigned to the symmetric stretching vibration ν_1 of the sulfate group in PbSO₄, and is widely recognized as the most characteristic Raman band for sulfate formation on lead-based electrodes. The bands in the 1060–1100 cm⁻¹ region are attributed to the ν_3 stretching modes of sulfate and are consistent with basic lead sulfate phases, particularly lanarkite (PbO·PbSO₄) and 4PbO·PbSO₄. In the 610–430 cm⁻¹ region, the observed bands are assigned to the ν_4 bending modes of sulfate coupled with Pb–O lattice vibrations, further supporting the

presence of mixed sulfate/oxide corrosion products. The low-wavenumber features around 132 cm⁻¹ and 97 cm⁻¹ are assigned to lattice vibrations of heavy lead-containing corrosion phases and indicate the development of a crystalline corrosion layer on the electrode surface rather than a response from the bulk Pb-Sb alloy [18,19]. Overall, these Raman features confirm the formation of a multilayer corrosion film dominated by sulfate-containing lead compounds.

Although the blank and BA samples exhibit the same set of peaks, the blank sample has much higher intensities, particularly at 977, 439, and 397 cm⁻¹, indicating a thicker, more crystalline corrosion film. By contrast, the H₃BO₃ sample shows weaker peaks, meaning that boric acid suppresses the growth, crystallization, and surface coverage of PbSO₄/PbO_x. Thus, H₃BO₃ does not change the type of corrosion products, but it clearly reduces their amount and slows the development of the corrosion film.

Influence of additives on electrochemical corrosion behavior.

The corrosion parameters derived from the Tafel plot analysis - namely the corrosion current density (J_{corr}) and the polarization resistance (R_p) of the Pb-Sb 2 wt% alloy immersed in H₂SO₄ electrolyte with and without 0.5 wt% H₃BO₃ at various immersion times are summarized in Table 2, Fig 3 a, b.

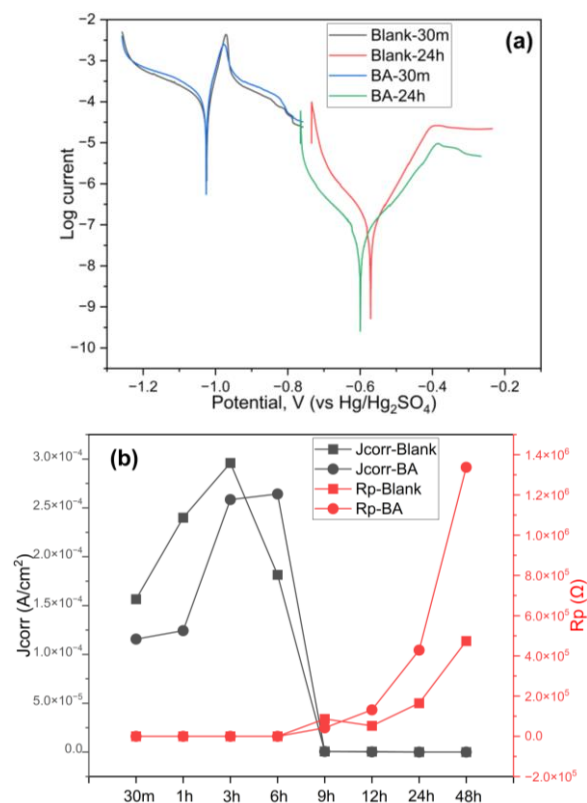


Fig 3: (a) Representative Tafel plots of Pb-Sb alloy in blank and BA electrolytes at 30 min and 24 h; (b) evolution of J_{corr} and R_p with immersion time (30 min–48 h) for blank

and BA samples.

The LSV results demonstrate that boric acid significantly affects the corrosion behavior of the Pb-Sb 2 wt% alloy in H_2SO_4 , as reflected in changes in both the corrosion current density (J_{corr}) and the polarization resistance (R_p). In most immersion periods, the H_3BO_3 -containing electrolyte yields lower J_{corr} and higher R_p values than the blank electrolyte.

At 30 min, the electrode surface had just been mechanically polished and cathodically reduced (-1.3 V, 10 min) to remove surface oxides, so the initial E_{corr} was close to the characteristic potential of the Pb/PbSO₄ couple (approximately -1.0 V vs. MSE). As the immersion time increased, the ongoing open-circuit corrosion process progressively oxidized metallic Pb to PbSO₄ and, in part, to PbO_x/PbO₂; these higher-valence oxide species possess equilibrium potentials considerably more positive than that of metallic Pb. As the oxide/sulfate film gradually became predominant and fully covered the underlying metal surface, the measured open-circuit potential shifted toward the value characteristic of the PbO_x/PbSO₄ system, accounting for the observed positive shift in E_{corr} .

Table 2: The results of Tafel plot analysis for PbSb2% alloy in the electrolyte with and without boric acid additive at various immersion times.

Time	Blank		
	$J_{\text{corr}}, \text{A/cm}^2$	$J_{\text{corr}}, \text{A}$	R_p, Ω
30m	1.57×10^{-4}	1.23×10^{-4}	85.295
60m	2.40×10^{-4}	1.88×10^{-4}	56.868
3h	2.96×10^{-4}	2.32×10^{-4}	44.213
6h	1.82×10^{-4}	1.43×10^{-4}	67.216
9h	7.02×10^{-7}	5.51×10^{-7}	86396
12h	5.56×10^{-7}	4.37×10^{-7}	52167
24h	1.30×10^{-7}	1.02×10^{-7}	164590
48h	5.89×10^{-8}	4.62×10^{-8}	473860

Time	Boric acid (BA)		
	$J_{\text{corr}}, \text{A/cm}^2$	$J_{\text{corr}}, \text{A}$	R_p, Ω
30m	1.16×10^{-4}	9.08×10^{-5}	101.94
60m	1.24×10^{-4}	9.75×10^{-5}	104.32
3h	2.59×10^{-4}	2.03×10^{-4}	59.79
6h	2.64×10^{-4}	2.07×10^{-4}	56.683
9h	6.36×10^{-7}	4.99×10^{-7}	42477
12h	1.97×10^{-7}	1.54×10^{-7}	131679
24h	6.40×10^{-8}	4.99×10^{-8}	428540
48h	2.08×10^{-8}	1.63×10^{-8}	1337312

At short immersion times of 30 and 60 min, the difference between the two systems is already evident. The blank sample exhibits higher J_{corr} , whereas the boric-acid-containing sample shows higher R_p , increasing from 85.295 to 101.94 Ω at 30 min and from 56.868 to

104.32 Ω at 60 min. These results suggest that boric acid is rapidly adsorbed on the alloy surface, thereby reducing the number of active sites and forming an initial protective layer that hinders charge transfer.

At intermediate immersion times of 3 and 6 h, fluctuations in both J_{corr} and R_p are observed in both systems. This behavior can be attributed to the dynamic growth and restructuring of the corrosion layer, where metal dissolution, formation of PbSO₄/PbO_x species, and rearrangement of the surface film occur simultaneously. Although the J_{corr} values do not always decrease monotonically in the presence of H_3BO_3 , the overall increase in R_p indicates that boric acid still promotes the development of a more protective interfacial layer [20,21].

At longer immersion times, particularly from 12 to 48 h, the beneficial effect of H_3BO_3 becomes more pronounced. The sample containing boric acid exhibits markedly higher R_p and lower J_{corr} , indicating the formation of a thicker and more resistive surface layer. At 48 h, the R_p value reaches 1,337,312 Ω in the presence of H_3BO_3 , which is approximately 2.8-fold higher than that of the blank sample (473,860 Ω). This result demonstrates that boric acid not only delays the initial corrosion process but also improves the long-term stability of the corrosion film [20,21].

Mechanism of corrosion inhibition by H_3BO_3

Based on the combined CV, LSV, and Raman results, a schematic mechanism for the corrosion-inhibiting action of H_3BO_3 on the Pb-Sb 2 wt% alloy is proposed in Fig 4. In the additive-free electrolyte, anodic polarization of the Pb-Sb surface generates Pb^{2+} , which reacts with SO_4^{2-} to nucleate PbSO₄ and subsequently converts, in part, to PbO_x and basic lead sulfate phases ($\text{PbO} \cdot \text{PbSO}_4$, $4\text{PbO} \cdot \text{PbSO}_4$) during repeated cycling, consistent with the Raman bands at 977, 1060–1100, and 610–430 cm^{-1} assigned to the ν_1 , ν_3 , and ν_4 modes of sulfate, respectively. In the blank system, this corrosion layer grows into a thick, loosely packed, and defect-rich film, as indicated by the high CV peak currents (Fig 1b) and the strong Raman intensities at 977, 439, and 397 cm^{-1} (Fig 2). Such a porous structure allows continued diffusion of H^+ and SO_4^{2-} through the film to the metal/film interface, sustaining active dissolution of the alloy; this is reflected in the low R_p values obtained at all immersion times in Table 2 ($R_p = 473860 \Omega$ at 48 h).

In the presence of 0.5 wt% H_3BO_3 , boric acid is expected to dissociate partially into $\text{B}(\text{OH})_3/\text{B}(\text{OH})_4^-$ species in the H_2SO_4 electrolyte and to adsorb preferentially at high-energy sites on the growing PbSO₄/PbO_x surface, such

as grain boundaries, crystal defects, and nucleation centers. This adsorption is proposed to hinder the lateral growth and crystallization of PbSO_4 , promoting the incorporation of borate species into the sulfate/oxide network and yielding a corrosion layer that is thinner, less crystalline, and structurally more disordered than that formed in the blank electrolyte. This proposed pathway is fully consistent with the experimental observations: (i) the CV peak currents in the BA electrolyte are systematically lower than in the blank (Fig 1a, b), indicating restricted surface conversion; (ii) the Raman spectra of the BA sample show the same peak positions as the blank-confirming that the chemical identity of the corrosion products (PbSO_4 , basic lead sulfate) is unchanged but with substantially reduced intensities, indicating a thinner and less crystalline film (Fig 2); and (iii) the LSV results show that J_{corr} in the BA sample decreases by a factor of two (from 1.30×10^{-7} to $6.40 \times 10^{-8} \text{ A cm}^{-2}$) while R_p increases by 2.6 times (from 164590 to 428540 Ω) at 24 h, and this protective effect becomes even more pronounced at 48 h, where R_p reaches 1,337,312 Ω , approximately 2.8-fold higher than the blank (Table 2). Together, these results support a mechanism in which H_3BO_3 does not alter the fundamental corrosion chemistry of the Pb-Sb alloy but instead acts as a growth-limiting and pore-blocking agent, forming a denser borate-modified sulfate/oxide layer that impedes $\text{H}^+/\text{SO}_4^{2-}$ transport and thereby slows the rate of grid corrosion in sulfuric acid electrolyte [12,13].

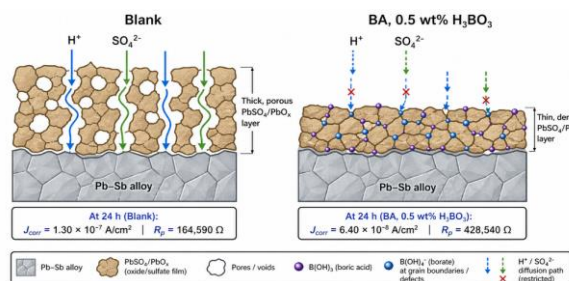


Fig 4: Proposed corrosion-inhibition mechanism of H_3BO_3 on Pb-Sb alloy

Conclusion

Electrochemical measurements show that adding 0.5 wt% H_3BO_3 to the sulfuric acid electrolyte decreases the corrosion current density (J_{corr}) and increases the polarization resistance (R_p) of Pb-Sb 2 wt% alloy grids, indicating a clear improvement in corrosion resistance. Raman spectroscopy confirms that the corrosion layer formed after cycling consists of PbSO_4 and basic lead sulfate phases, but with significantly lower band intensities in the presence of boric acid, indicating a thinner and less developed sulfate/oxide film. Overall, these findings demonstrate that H_3BO_3 is a promising electrolyte additive for Pb-Sb grids, capable of slowing

corrosion-film growth and stabilizing a more protective surface layer in lead-acid battery environments.

Acknowledgments

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