

## COMPARISON OF Sn AND As EFFECT ON TENSILE PROPERTIES OF Pb–3.5%Sb GRID ALLOY FOR LEAD-ACID BATTERIES

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In this work, the effects of 0.5 wt.% Sn and 0.16–0.23 wt.% As on tensile properties of Pb–3.5%Sb grid alloy for lead-acid batteries were compared in the as-cast condition. The alloys were melted under different cooling-rate conditions in a casting mold preheated between 50°C and 170°C, with cooling rates ranging from 100 °C/s to 50 °C/s. Mechanical properties, such as ultimate tensile strength and percentage elongation, were measured at room temperature using the TIRAtest 2300 universal testing machine at a constant crosshead speed of 10 mm/min. It was established that as mold preheating temperatures rise, the elongation and ultimate tensile strength of the Pb–3.5%Sb–0.23%As alloy decrease by 13.9% and 11.8%, respectively. Addition of tin in place of some arsenic causes a decrease in ultimate tensile strength of the Pb–3.5%Sb–0.5%Sn–0.16%As alloy, but only by 2.8 %, whereas elongation increases by 2.4 %. It was concluded that additions of tin compensate for the negative effect of arsenic on the tensile properties of the Pb–3.5%Sb grid alloy, which relates to the formation of brittle arsenic-containing phases at the grain boundaries. Tin addition to the Pb–3.5 %Sb alloy produces higher tensile properties at room temperature than those obtained by the addition of arsenic.

**Keywords:** *Lead-acid batteries; Pb–Sb based grid alloys; Casting mold preheating temperature; Cooling rate; Tensile tests; Ultimate tensile strength; Elongation*

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Lead-acid batteries are the most widely used rechargeable batteries in the automotive and industrial sectors, powering many appliances and grid-scale power systems for decades [1-6]. Irrespective of the environmental hazards they pose, lead-acid batteries remain ahead of their peers because of their lower cost compared with lithium-ion or nickel-cadmium batteries. Industry will still have to deal with millions of lead-acid batteries, already existing and those planned to be produced in the next few years. The increasing demand for motor vehicles as countries undergo economic development and growth in the use of renewable energy sources, with the need for storage batteries, is directly proportional to the increasing demand for lead-acid batteries.

Lead-acid batteries contain cast lead alloy grids, packed with varying amounts of Pb, PbO<sub>2</sub>, or other active pastes to form the positive and negative grids. The grid is the most critical non-active component in a lead-acid battery. This is used to support the positive and negative active materials and to provide a conductive path for current to and from the plates during charging and discharging.

Lead grids alloyed with antimony are commonly used in batteries today. Lead-antimony grids have been well-researched and are known for their use in the positive electrode grids of lead-acid batteries, as antimony enables high charge-discharge performance and good castability [7-10]. However, high-antimony lead alloys are prone to sulfation and should not be left at low states of charge for extended periods of time [11-13]. These alloys increase battery gassing during charging, leading to high water loss. Because water must be added to these batteries, they have higher maintenance costs. Furthermore, high-antimony lead batteries have a high discharge rate and a short lifetime. These problems are caused by the dissolution of antimony from one electrode and its deposition on the other electrode [14]. Therefore, lead-antimony alloys for battery grids in automotive applications are produced only up to 3.5 wt.% Sb [15].

Medium-antimony lead alloys (containing 3.0–4.0 wt.% Sb) reduce the transfer of antimony to the negative plate and thus reduce water loss of the battery [16], especially when alloyed with additions of such elements as arsenic and tin [17-21]. The combination of these alloying elements with antimony enables the manufacture of battery grids with lower antimony content that meet the necessary mechanical strength and quality requirements.

The main difficulty with casting lead-antimony alloys is that the decrease in antimony content is accompanied by the appearance of hot cracks. Solidification occurs in a coarse dendritic structure with cracks along grain boundaries [9]. Tin additions have been shown to improve castability, mechanical properties, deep cycling and to reduce the tendency towards passivation and corrosion [22-25]. Another element, arsenic, has been added to lead-acid batteries and is currently used mostly with lead-antimony grids because it allows for lower antimony content which minimizes the self-discharging characteristics of higher antimony composition batteries [26-31]. In addition to improving lead-antimony grids, alloying lead grids with arsenic at low percentages (<0.1%) increases hardness, yields finer grains, and improves casting properties in lead-arsenic grids.

Suitable grid alloys must possess acceptable tensile properties for processability, enabling the alloys to be cast into plates of the desired configuration [32]. In other words, the alloys can be processed under desired battery assembling and manufacturing conditions [33-35]. Ultimate tensile strength and elongation are among the mechanical properties important to grid performance. Among the factors that can affect these properties is the cooling rate of the alloys during casting, which is regulated by the mold preheating temperature [35-38]. Changes in this parameter has been commonly used to control grain size and second phase dispersion that are responsible for achieving the required mechanical properties [39-42]. Although much attention has focused on lead-antimony grid alloys for lead-acid batteries, research on the factors influencing tensile properties remains scarce. Currently, the microstructure and hardening mechanisms are mostly determined using metallography, hardness measurements, and/or electrical resistivity measurements [43-45]. But studying the tensile properties of these grid alloys is indispensable for ensuring their safe and reliable application and has important research value and practical significance. Therefore, this work focuses on comparative analysis of the effect of tin and arsenic additions to the Pb-3.5%Sb grid alloy on tensile properties as a function of mold preheating temperature during conventional casting.

### MATERIALS AND METHODS

The alloys for this study were cast in the melt pot of the cast-on-strap machine on the production line of BM Company (Austria) for lead-acid battery grids at Westa Corp. (City of Dnipro, Ukraine). The molten alloys maintained at the temperature of  $450 \pm 10$  °C were poured in different cooling rate conditions into a preheated mold, which temperature was changed in the range between 50 °C and 170 °C with an interval of 5 °C. Then the casting mold was left to cool to room temperature at different rates, ranging from 100 °C/s to 50 °C/s. The temperature was measured using a K-type chromel-alumel thermocouple with a standard error of  $\pm 5$  °C. The diameters of the thermocouple electrode wires and their junction were 1 mm and 2 mm, respectively. For each mold preheating temperature, six specimens were collected from each casting for further investigation.

The weight values of alloy chemical composition determined by atomic absorption spectroscopy using ARL 3460 instrument were 3.5 wt.% Sb, 0.5 wt.% Sn, 0.16–0.23 wt.% As, and Pb – the balance. Trace amounts of impurities, such as Al and Se, amounted to less than 0.02 wt.%. The additional amount of trace elements was a factor that largely fluctuated depending on the blending ratio of scrap. Samples were obtained from a same casting batch to guarantee an identical chemical composition. Three samples for chemical analysis were taken at the time of the casting to ascertain the exact chemical composition of each alloy.

The tensile tests were conducted at room temperature on a TIRAtest 2300 PC-controlled testing system at a constant crosshead speed of 10 mm/min. The ultimate tensile strength ( $\sigma_U$ ) and percentage elongation ( $\sigma$ ) were determined one day after casting. Standard flat samples with a total length of 60 mm, a gage length of 45 mm, and a thickness of 3 mm were prepared for tensile tests. For this purpose, casting mold made of structural carbon steel that had 2.5 mm thick thermal coating of cork suspension inside was designed (Fig. 1). Each reported value of tensile properties corresponded to the mean of six measurements. The average uncertainty of measurements was estimated at approximately  $\pm 5$  %.

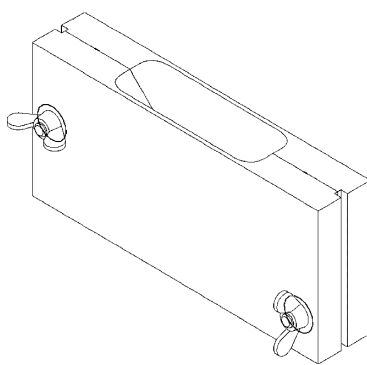


Figure 1. General view of casting mold

### RESULTS AND DISCUSSION

Variation of the preheating temperatures of a casting mold has the potential for influencing the tensile properties of the studied grid alloys to a noticeable extent. As the mold preheating temperature increases from 50 °C to 170 °C, the values of ultimate tensile strength for Pb-3.5%Sb-0.23%As alloy decrease from 38.9 MPa to 34.8 MPa (by 11.8 %) with an average decrement equaling to 0.03 MPa per 1 °C (Fig. 2a). The percentage elongation of the alloy exhibits the same trend with respect to mold preheating temperature. The elongation decreases from 8.2 % to 7.2 % (by 13.9 %) with an average decrement of about 0.008 % per 1 °C (Fig. 2b). So, the alloy tends to become more brittle, less prone to deformation, as shown by the decreased elongation [46]. The tensile properties of the Pb-3.5%Sb-0.23%As alloy are less influenced by cooling conditions between 50 °C and 110 °C.

Considering that elongation and ultimate tensile strength of the alloy are nearly the same at mold temperatures lower 110 °C, a mold during casting should be preheated over the temperature range between 50 °C and 110 °C. Besides, this ensures 1.5 times higher cooling rates of the alloy and enhances the supersaturation of the lead-based solid solution [47]. As a result, less brittle arsenic precipitates can form at the grain boundaries in the as-cast structure [27].

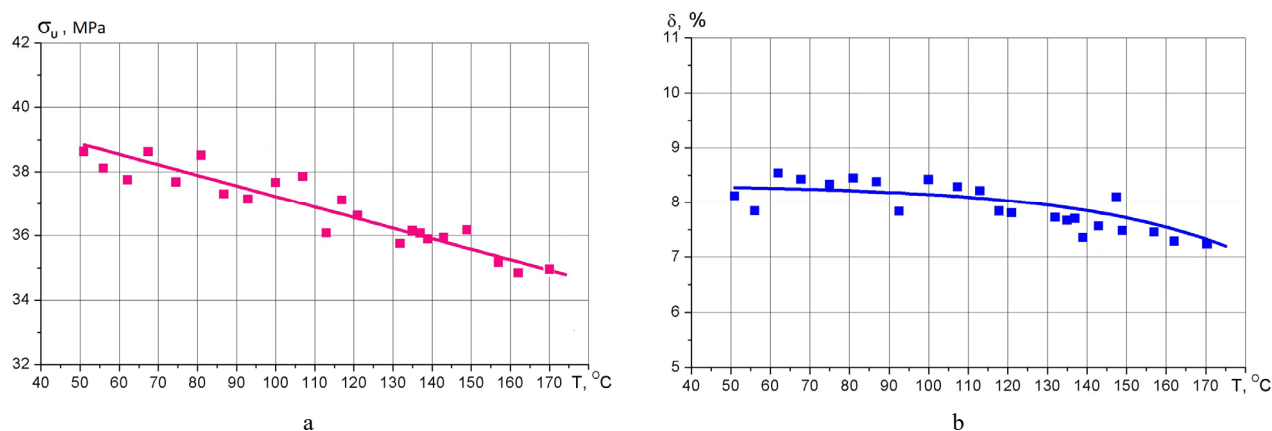


Figure 2. Effect of preheating temperature of casting mold on tensile properties of Pb–3.5%Sb–0.23%As alloy: a – ultimate tensile strength; b – elongation

When Pb–Sb–As grid alloy contains up to 0.5 wt.% tin in place of some of the arsenic, provided that the arsenic level is at least 0.16 wt.%, the ultimate tensile strength slightly decreases from 40.0 MPa to 38.9 MPa (by 2.8 %) while elongation insignificantly increases from 8.1 % to 8.3 % (by 2.4 %) with higher mold temperature (Fig. 3). The temperature dependencies of tensile properties are approximately 0.012 MPa/°C and 0.002 %/°C, respectively. So, these characteristics remain virtually unchanged, which correlates with insignificant changes in the grain size of the Pb–3.5%Sb–0.5%Sn–0.16%As grid alloy as the mold temperature increases from 75 °C to 165 °C. As shown, the tensile properties are more stable between 140 °C and 165 °C. Considering that elongation of the alloy is nearly the same at mold temperatures above 110 °C, while ultimate tensile strength tends to slight decrease up to 140 °C, a mold during casting should be preheated over the temperature range between 140 °C and 165 °C.

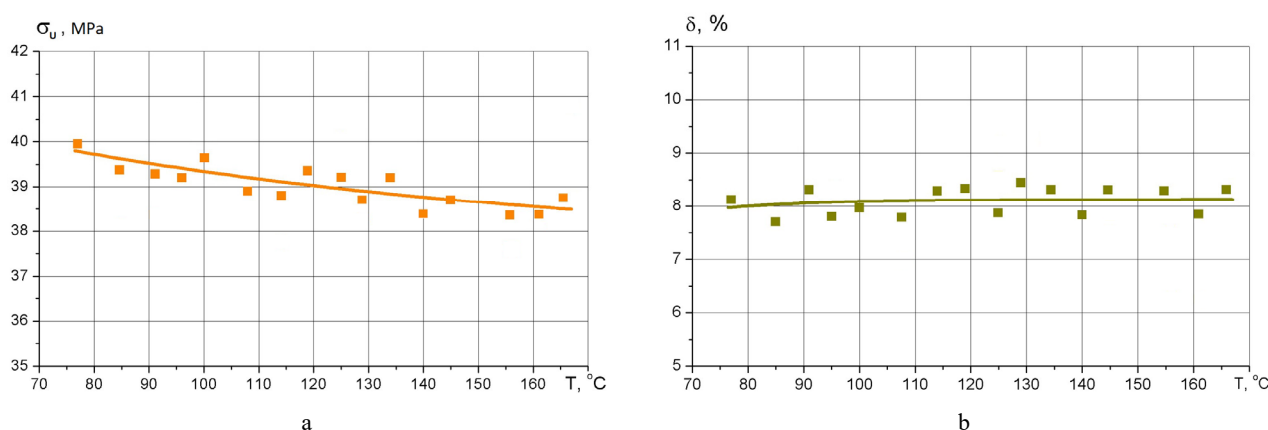


Figure 3. Effect of preheating temperature of casting mold on tensile properties of Pb–3.5%Sb–0.5%Sn–0.16%As alloy: a – ultimate tensile strength; b – elongation

Considering obtained results, it may be assumed that with preheating temperature of the mold increasing, the cooling conditions become closer to an equilibrium kinetic. The effect of the slower cooling rate results in the decreased distortion in the alloys' crystal structure, so the dislocation motion occurs easily, hence the tensile strength decreases. Since mold temperature is related to the cooling rate, the increase in the mold temperature enlarges grains in the structure of the alloys [48]. By increasing the grain size, the area of grain boundaries which can block dislocations and impede their movement decreases, which is disadvantageous for the strength as well. As a result, ultimate tensile strength of both alloys tends to reduce.

Besides the slower cooled alloys have a less uniform structure due to more severe segregation effects. The grain boundaries contain relatively higher arsenic and tin content [25–27]. Due to the segregation, grain boundaries become weaker than similar boundary areas (which contain fewer alloying elements) in rapidly cooled alloys. The segregated arsenic forms own phases which can make the Pb–3.5%Sb–0.23%As alloy more brittle as confirmed by its decreased elongation. When the preheating temperature of the casting mold is higher, the slight increase in elongation of the Pb–3.5%Sb–0.5%Sn–0.16%As alloy indicates that arsenic contributes more to the embrittlement of grain boundaries

than tin. Tin may also prevent the migration of arsenic towards the boundaries, thereby ensuring a more uniform structure of the Pb–3.5%Sb–0.5%Sn–0.16%As alloy. As a result, tensile properties of this alloy show less noticeable changes with variation of mold preheating temperatures as compared with those of the Pb–3.5%Sb–0.23%As alloy.

### CONCLUSIONS

The tensile properties of the Pb–3.5%Sb–0.23%As and Pb–3.5%Sb–0.5%Sn–0.16%As grid alloys are found to be sensitive to the temperature parameters of the casting procedure. Changing the preheating temperature of the casting mold in the range 50–170 °C reduces the ultimate tensile strength of both alloys by 11.8% and 2.8%, respectively. At the same time, elongation of the Pb–3.5%Sb–0.23%As decreases by 13.9 %, but that of the Pb–3.5%Sb–0.5%Sn–0.16%As increases by 2.4 %, exhibiting an inverse trend compared to the strength with respect to preheating mold temperature. Based on the results obtained, it is assumed that tin has a positive effect on the tensile properties of the Pb–Sb–As alloy. The improvement in tensile properties can be attributed to a more uniform structure and less brittle arsenic phases at the grain boundaries of the tin-containing alloy.

Within the range of 110 °C to 170 °C, the Pb–3.5%Sb–0.23%As alloy is more sensitive to cooling conditions in the mold. But when a mold is maintained at temperatures below 110°C, the ultimate tensile strength and elongation generally stabilize. For the same reason, the casting parameters for the Pb–3.5%Sb–0.5%Sn–0.16%As alloy, should be kept in a range 140 °C–165 °C.

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## ПОРІВНЯННЯ ВПЛИВУ Sn ТА As НА ВЛАСТИВОСТІ НА РОЗТЯГ СПЛАВУ Pb–3,5%Sb ДЛЯ СТРУМОВІДВОДІВ СВИНЦЕВО-КИСЛОТНИХ АКУМУЛЯТОРІВ

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В роботі порівнювали вплив 0,5 ваг.% Sn та 0,16–0,23 ваг.% As на властивості на розтяг литого сплаву Pb–3,5%Sb для струмовідводів свинцево-кислотних акумуляторів. Сплави виплавляли в умовах охолодження з різною швидкістю в ливарних формах, попередньо підігрітих в інтервалі температур від 50 °C до 170 °C, що відповідало швидкості охолодження в діапазоні від 100 °C/с до 50 °C/с. Механічні властивості, а саме межу міцності на розтяг і відносне подовження, вимірювали за кімнатної температури на універсальній випробувальній машині TIRAtest 2300 за сталої швидкості траверси 10 мм/хв. Встановили, що зі збільшенням температури підігріву ливарної форми подовження і межа міцності на розтяг сплаву Pb–3,5%Sb–0,23%As зменшуються на 13,9 % та 11,8 %, відповідно. Додавання олова замість миш'яку знижує межу міцності на розтяг лише на 2,8 %, тоді як подовження збільшується на 2,4 %. Зроблено висновок, що додавання олова компенсує негативний вплив миш'яку на властивості на розрив сплаву Pb–3,5%Sb для струмовідводів, пов'язаний з утворенням крихких фаз із вмістом миш'яку на границях зерен сплавів. Додавання олова до сплаву Pb–3,5%Sb забезпечує кращі властивості на розтяг за кімнатної температури, ніж додавання миш'яку.

**Ключові слова:** свинцево-кислотні акумулятори; сплави на основі системи Pb–Sb для струмовідводів; температура підігріву ливарної форми; швидкість охолодження; випробування на розрив; межа міцності на розрив; подовження