

## EVOLUTION OF MECHANICAL PROPERTIES OF Pb–Sb–Sn–As–Se GRID ALLOYS FOR LEAD-ACID BATTERIES DURING NATURAL AGING

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Received October 1, 2023; revised November 1, 2023; accepted November 10, 2023

This study is devoted to the investigation of mechanical properties of a series of low-antimony Pb–Sb–Sn–As–Se grid alloys for lead-acid batteries in as-cast condition and after natural aging during storage. Mechanical properties were characterized by ultimate tensile strength, yield strength, elongation, and Young's modulus determined at room temperature using TIRAtest 2300 and P-0.5 universal testing machines. For most investigated as-cast alloys, an increase in ultimate tensile strength is accompanied by an increase in elongation. Within the temperature range between 70 °C and 150 °C, higher heating temperature of a casing mold does not markedly affect average elongation but causes the slight decrease (by ~4 %) in average ultimate tensile strength. When aged during storage for 30–33 days, the Pb–Sb–Sn–As–Se grid alloys, attain higher values of ultimate tensile strength, yield strength, and Young's modulus but lower values of elongation. This is due to precipitation of second-phase particles from lead-based solid solution oversaturated by antimony, arsenic, and selenium. The most noticeable effect of strengthening is observed during first five days of natural aging.

**Keywords:** *Lead-acid batteries, Low-antimony Pb–Sb–Sn–As–Se grid alloys, Natural aging during storage, Tensile tests, Ultimate tensile strength, Yield strength, Elongation, Young's modulus*

**PACS:** 61.82.Bg, 61.66.Dk, 62.20.-x, 62.20.Fe, 62.20.Mk, 64.70.Dv, 81.70.Bt, 81.40.Cd, 81.40.Lm

For many years, the most common battery chemistries used for lead-acid batteries were the high-antimony lead alloy compositions. Antimony gave to the pure lead strength, good castability and high performance on charge-discharge characteristics [1-4]. The concentrations of antimony to lead were initially in the 4–12 wt.% range [5-9]. But most battery manufacturers tried to minimize the antimony addition especially in batteries for stationary applications. High-antimony grids have higher hydrogen evolution (which also accelerates as the battery ages), and thus greater outgassing and higher maintenance costs. These problems are caused by the dissolution of antimony from positive electrode and its deposition or plating on negative electrode. This leads to lower charge voltage, a high discharge rate, increased water loss of the battery and therefore a short lifetime [10-14].

Using alloying elements, the antimony content of grid alloys can be reduced to the level that the drawbacks of low antimony are almost eliminated, but the positive effects are mainly retained [15-20]. Nowadays the grids for lead-acid batteries are made from lead antimony alloys together with minor additions of elements such as Sn, As, and Se [21-27]. These are added to confer properties such as grain refinement, castability and performance characteristics to the grids.

Adding antimony to produce hypoeutectic lead antimony alloys also ensures hardening of lead. The alloys containing 6 wt.% Sb appears to undergo optimum hardening, but the alloys with 2 wt.% Sb and less harden comparatively slow [28,29]. Antimony in low-antimony lead alloys has difficulty in precipitating and therefore substantially remains in solution through the casting, working process and aging period. Therefore, small amounts of arsenic and selenium retain beneficial characteristics of high-antimony alloys, such as the precipitation hardening effect, increasing supersaturation of antimony in lead when its concentration is relatively low (<2 wt.%). Additional alloying elements typically produce a fine and homogenize distribution of precipitation [27]. But only when the alloys are heat treated the alloys strengthen on aging due to formation of metastable arsenic- or selenium-bearing nuclei which facilitate the antimony precipitation process [30-32].

Heat treatment of the low-antimony lead alloys is performed under time and temperature conditions which do not result in a conventional solution treatment effect [33-40]. Solution treatment requires diffusion-controlled dissolution of the already precipitated antimony-rich phase. Such processes are slow since they depend on the solid-state movement of individual atoms from one crystal site to the next [41-45]. Strengthening occurs after quenching when the super-saturated solution precipitates in a form which strains the alloy crystal lattice and inhibits dislocation motion [46-51].

In alloys of the lead-antimony system, the initial hardening produced by alloying is quickly followed by softening as the coarse structure is formed during storage. The effects of tin, arsenic, and selenium on low-antimony Pb–Sb alloys were investigated by some authors [29,32], but there is not complete research about the influence of these additives on the evolution of the tensile properties of Pb–Sb–Sn–As–Se alloys from as-cast condition and under natural aging during storage. The purpose of this paper is to achieve the mechanical strength within aging times suitable for storage using the combination of alloying elements such as antimony, tin, arsenic, and selenium with lead to manufacture lead-acid battery grids with low antimony contents (<1.77 wt.%), which show the necessary grid quality.

## MATERIALS AND METHODS

Commercial Pb–Sb–Sn–As–Se grid alloys for lead-acid batteries received from *Westa* ISI Corp. (City of Dnipro, Ukraine) were used in this research. The chemical composition of the studied castings is presented in Table 1. The alloys were composed of Sb (1.55–1.77 wt.%); Sn (0.11–0.17 wt.%); As (0.097–0.13 wt.%); Se (0.018–0.025 wt.%); impurities, such as Cu, Bi, Ag, S, Fe, Ni, Cd, and Zn (in descending order), (0.0217–0.0533 wt.% altogether); Pb – the remainder. The chemical analysis of elements present in the castings was performed using an ARL 3460 optical emission spectrometer. The Pb–Sb–Sn–As grid alloys were prepared in the melting pot of *Wirtz* manufacturing book mold grids casting machine (the USA). The melts at temperatures of 440–460 °C were poured into a casting mold preheated in the temperature range between 70 °C and 150 °C. The temperatures of the mold were controlled using chromel-alumel thermocouple.

Mechanical properties of the as-cast Pb–Sb–Sn–As–Se grid alloys used in this study were assessed in terms of ultimate tensile strength ( $\sigma_U$ ) and elongation ( $\gamma$ ). The tensile tests were performed at room temperature in P-0.5 tensile testing machine providing controlled uniformly increasing tension force. The specimens flat in the cross section which had a thickness of 0.3 cm, a total length of 6.0 cm, and a gage length of 4.5 cm (prepared according to DSTU 11701-84) were stretched up to failure. Six tests were conducted for each alloy composition and the average values were reported. The measurements were presented with their standard error of 2–8 %. In the results section, the mean curves determined from tensile tests are shown.

The as-cast Pb–Sb–Sn–As–Se alloys were put into storage where they naturally aged at room temperature for 30-33 days. Determination of the tensile properties was made 1 day after casting and then at intervals of 3–4 days. After each interval of aging time, six specimens in a row were exerted for tensile tests carried out on TIRAtest 2300 universal testing machine. Altogether were tested sixty specimens broken into 10 sets of 6 specimens each. Tensile experiments were carried out on the flat specimens at room temperature with a crosshead speed of 10 mm/min. Tensile force was applied stepwise with a step of 100 N until the stress part of stress-strain diagrams started falling back down slowly. As the load was applied, the stress and strain experienced by the test specimen were captured digitally and graphed. Values of ultimate tensile strength ( $\sigma_U$ ), yield strength ( $\sigma_Y$ ), and Young's modulus ( $Y$ ) were determined from plotted stress-strain diagrams. The data obtained were each calculated from an average of six specimens with error 3–5 %.

## RESULTS AND DISCUSSION

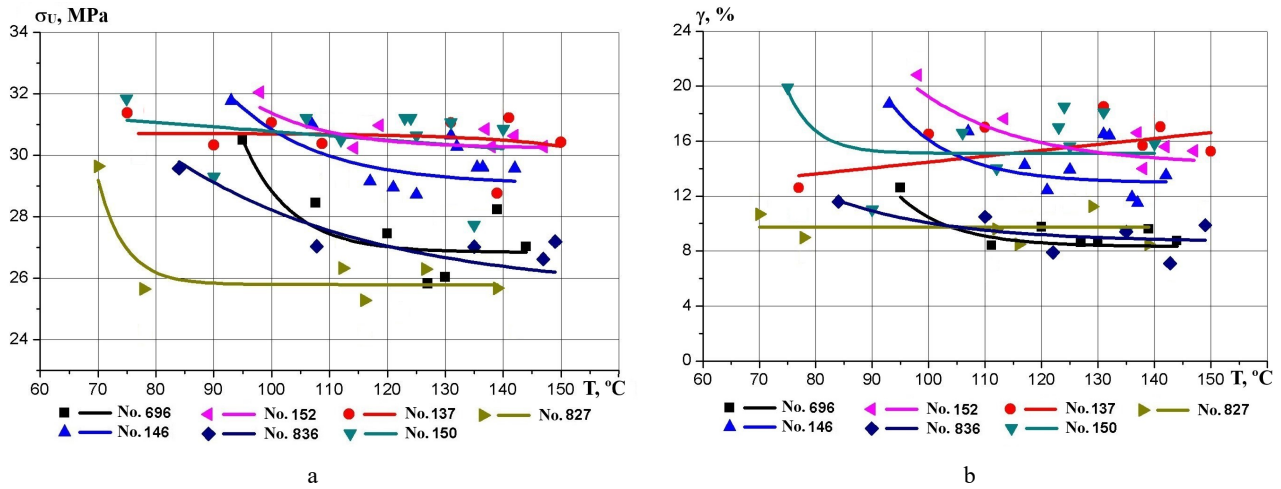
The studied as-cast Pb–Sb–Sn–As–Se grid alloys consist of two phases, namely primary dendrites of  $\alpha$ -Pb solid solution and  $\beta$ -Sb phase formed at the boundaries of  $\alpha$ -phase [1-3]. In the investigated compositional range, the influence of major alloying elements on ultimate tensile strength and elongation is quite irregular (Table 1). These irregularities are believed to be more experimental errors than significant variations in the properties of the alloys. But in general, ultimate tensile strength increases with increasing arsenic content. Most alloys become more ductile when become stronger. The combination of high ultimate tensile strength and high elongation ensures high toughness of the Pb–Sb–Sn–As–Se grid alloys. Meanwhile, increasing content of impurities in the investigated Pb-based alloys leads to a decrease in their elongation. This is because impurities segregate at grain boundaries of  $\alpha$ -Pb phase forming brittle secondary phases. Also, there are more interfaces between the phases which hinder the dislocation motion [35].

**Table 1.** The mechanical properties of the as-cast Pb–Sb–Sn–As–Se grid alloys for lead-acid batteries

Casting No.	Chemical composition, wt.%					$\sigma_U$ , MPa	$\gamma$ , %
	Sb	Sn	As	Se	Impurities		
952	1.62	0.14	0.10	0.021	0,0549	22.3	6.7
433	1.55	0.12	0.10	0.020	0.0533	25.5	8.7
980	1.58	0.13	0.11	0.018	0.0480	25.8	9.5
827	1.64	0.14	0.11	0.023	0.0452	26.3	9.7
696	1.77	0.13	0.10	0.024	0.0318	26.3	9.7
870	1.67	0.15	0.11	0.022	0.0314	26.8	9.7
836	1.61	0.13	0.11	0.025	0.0527	27.0	9.5
146	1.65	0.11	0.11	0.023	0.0270	29.4	13.9
202	1.66	0.13	0.12	0.022	0.0258	29.9	12.6
137	1.56	0.14	0.11	0.023	0.0212	30.4	15.4
150	1.70	0.11	0.12	0.023	0.0269	30.4	15.6
152	1.63	0.17	0.11	0.022	0.0217	30.4	16.0
0005	1.68	0.15	0.13	0.024	0.0291	31.7	12.4
<b>Average</b>	<b>1.64±0.04</b>	<b>0.13±0.01</b>	<b>0.11±0.01</b>	<b>0.022±0.001</b>	<b>0.0361±0.0113</b>	<b>27.9±2.3</b>	<b>11.4±2.7</b>

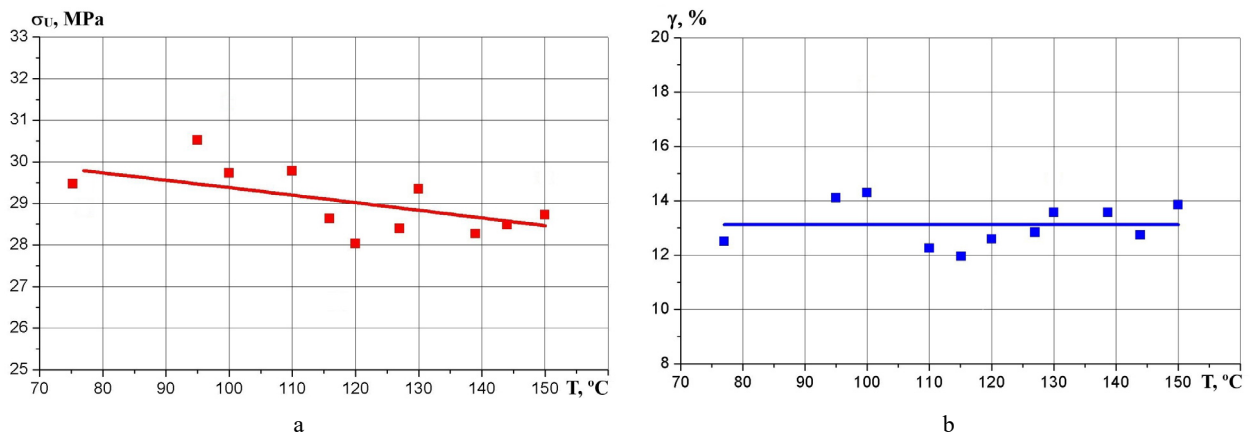
The ultimate tensile strength data show a decrease with increasing heating temperature of casting mold in the range between 70 °C and 110 °C, folowed by practically constant or slightly decreasing values at temperatures upwards of 110 °C (Fig. 1a). Tensile strength results on as-cast specimens tend to be influenced by imperfections, such as microscopic cracks or cavities which weaken castings produced at the mold temperatures above 110 °C due to slower cooling rate. These defects create a weak point where a crack can initiate. The experimental data indicate some

irregularities in elongation behavior although this characteristic is usually affected by casting imperfections to a greater degree than the ultimate tensile strength (Fig. 1b).



**Figure 1.** Effect of heating temperature of casting mold on tensile properties of castings: a – ultimate tensile strength; b – elongation

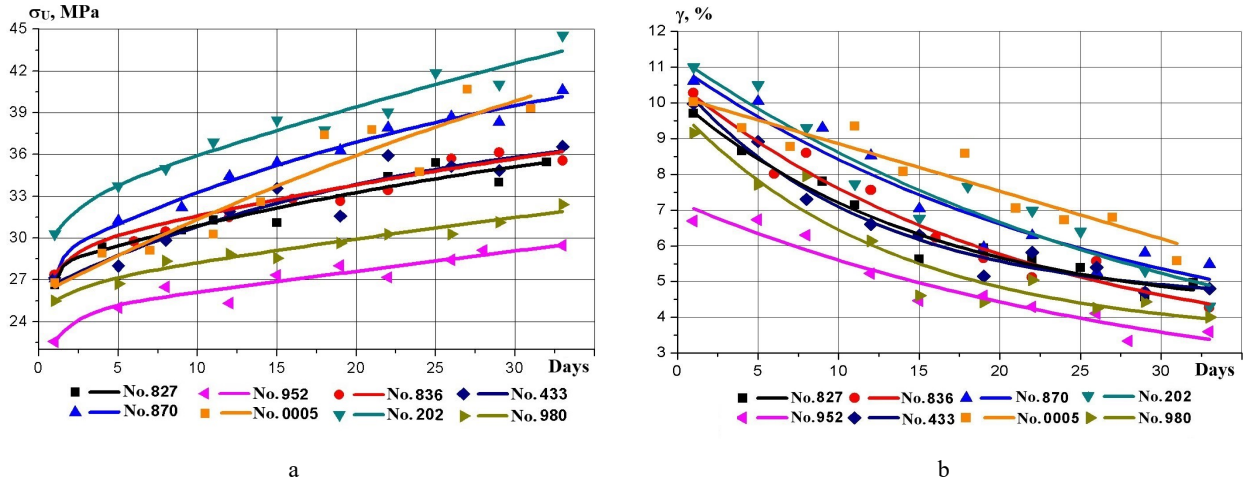
Based on the tensile test results, average curves for a set of specimens in the as-cast condition are calculated and a linear trend line is given for the subsequent analysis of the influence of heating temperature of casting mold on tensile properties, as shown in Fig. 2. The data confirm that the tensile strength is lowered (Fig. 2a), although the elongation of the alloys is not appreciably affected by heating temperature (Fig. 2b). With mold temperature increasing from 75 °C to 150 °C, average tensile strength decreases from 29.8 MPa to 28.5 MPa (by ~4 %). The temperature dependence of ultimate tensile strength is approximately 0.017 MPa/°C. It is possible that changes in ultimate tensile strength behavior may be attributed to the fact that cooling rate of the as-cast specimens decreases as a heating temperature of casting mold increases. Aside from deleterious effect of casting flaws, the slower cooling rate also produces coarser microstructure of the alloys, weakening them in ultimate tensile strength, while not appreciably affecting their elongation to failure. Besides, in slow cooling the lead grains tend to push the major alloying elements and impurities to the grain boundaries which is the lowest energy condition. These elements at the grain boundaries still provide dislocation blocking, but not so well as uniformly dispersed ones. The residual internal stresses cause tensile strength tests to give lower values. Meanwhile, considering instability of tensile properties at heating temperatures below 110 °C, a mold during casting should be preheated over the temperature range of 110 °C to 150 °C despite the slight deterioration in average values of ultimate tensile strength.



**Figure 2.** Average values of tensile properties vs. heating temperature of casting mold: a – ultimate tensile strength; b – elongation

The mechanical properties of the Pb–Sb–Sn–As–Se alloys are dependent not only on the heating temperature of casting mold but also on the time of natural aging during storage after cooling to room temperature. The casting conditions assure oversaturation of lead-rich  $\alpha$ -phase in the structure of the alloys which is sufficient for age-strengthening. Fig. 3 shows the effects of room temperature storage on the aging characteristics of the alloys identified by changes with time in the ultimate tensile strength and the elongation determined using P-0.5 tensile testing machine. Some alloys experienced up to a 40-% increase in ultimate tensile strength (from 22–32 MPa to 30–44 MPa) during 33 days of storage after casting. All aging curves have the same characteristic shape since strengthening occurs in two stages: at first rapid strengthening during the first 5 days, followed by a much longer period of gradual strengthening

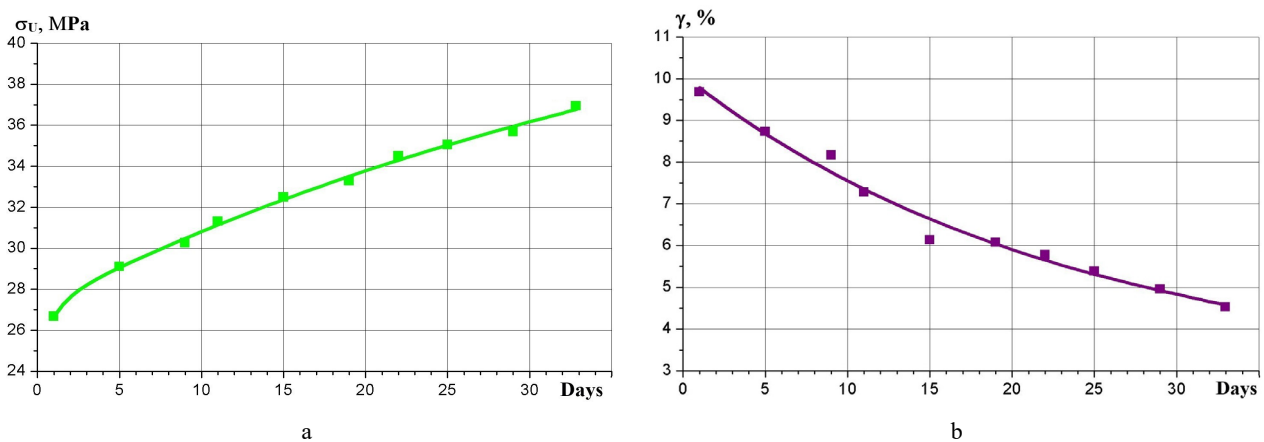
(Fig. 3a). This process is mainly attributed to the precipitation of the finely dispersed antimony in the supersaturated lead-rich matrix [29,30]. What is more, the best strengthening results are obtained with alloys of higher selenium and arsenic contents. As known, these additional alloying elements produce a fine and homogenize distribution of precipitates [27]. The smaller grains have the larger area of grain boundaries that inhibit dislocation motion. Therefore, reducing the grain size increases the available nucleation sites for precipitated antimony.



**Figure 3.** Effect of aging time on tensile properties of castings: a – ultimate tensile strength; b – elongation

The aging effect of the Pb–Sb–Sn–As–Se alloys during storage also results in a decrease in the elongation (Fig. 3b). Depending on castings composition, elongation reduces by 50–57 % (from 7–11 % to 3.5–5.1 %) after 33 days of storage. Such behavior can be explained by precipitation of supersaturated lead-based solid solution which makes the alloys lattice distortion and hinders dislocation motion. The casting No. 0005 combining a sufficient tensile strength with the highest elongation of any contains simultaneously the highest amounts of arsenic (0.13 wt.%) and selenium (0.024 wt.%) (Table 1). This alloy undergoes precipitation strengthening due to antimony as well as other minor alloying elements, particularly arsenic and selenium, forming particles within the lattice of the casting. The grids produced contain a higher volume of second-phase particles which inhibit dislocation and grain boundary movement and thus make the grids more stable.

Average values of the ultimate tensile strength and elongation for the investigated castings are plotted in the graphs of Fig. 4. As expected, ultimate tensile strength raises from 26.7 MPa to 37.0 MPa (by ~40 %), but elongation reduces from 9.7 % to 4.5 % (by ~2 times) after 33 days of aging. Aging time dependence of the ultimate tensile strength is 0.39 MPa/day, whereas the elongation's dependence is 0.54 %/day. Thus, as the ultimate tensile strength increases, the alloys tend to become more brittle and less prone to tension, as shown by the decreased elongation to failure. But for grid alloys, the increase in ultimate tensile strength might be of greater importance than the decrease in elongation which accompanies the aging processes.



**Figure 4.** Average values of tensile properties vs. aging time: a – ultimate tensile strength; b – elongation

Fig. 5 illustrates typical stress-strain diagrams that use data from tensile tests performed in TIRAtest 2300 universal testing machine (exemplified by six specimens aged for 14 days). All curves display a linear elastic range where the stress and strain are proportional to each other and a gradually rising part (without pronounced yield plateau) where specimens undergo plastic deformation.



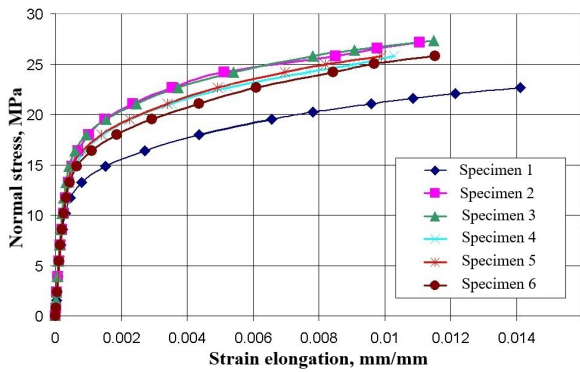


Figure 5. Typical stress-strain curves for tensile tests on 6 specimens aged for 14 days

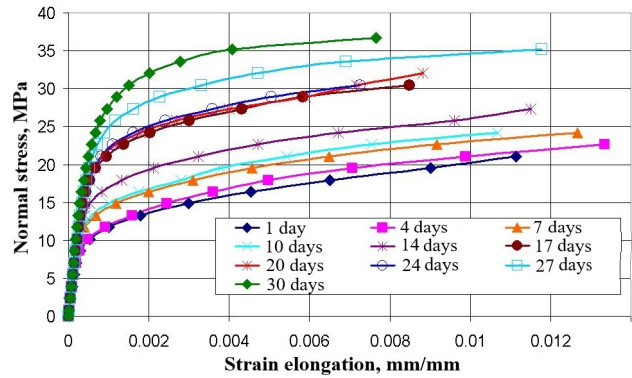
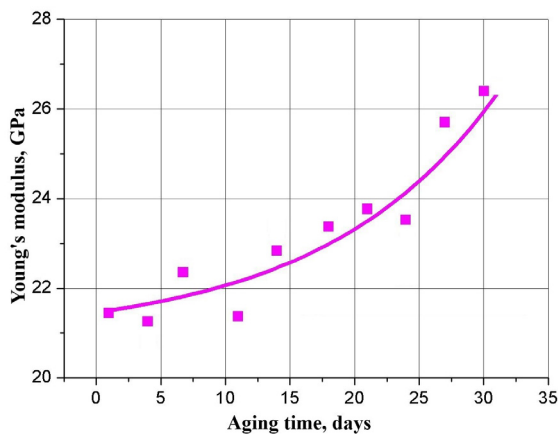
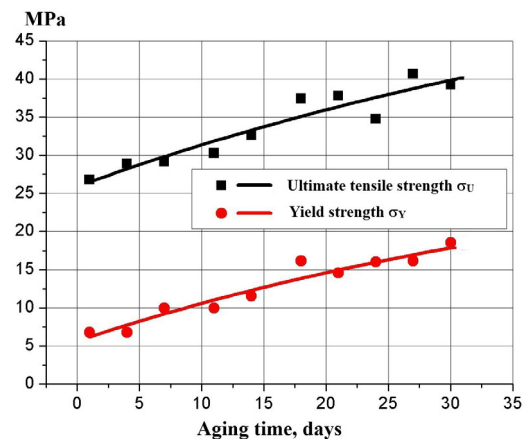


Figure 6. Stress-strain diagrams averaged over 10 sets of 60 specimens aged for 1–30 days

The slope of a linear region of the stress-strain graphs increases with prolonging aging time during storage, which evidences the improvement of elastic properties (Fig. 6). Besides, the curves have a larger elastic region where the stress-strain relationship is linear, so the stress at which specimens deform plastically increases. The lift height of the stress-strain graphs increases with aging time which means that the studied Pb–Sb–Sn–As–Se alloys become stronger. These observations are confirmed by calculated average values of Young's modulus, yield strength, and ultimate tensile strength as functions of aging time (Fig. 7). After 30 days of aging, the values of Young's modulus raise from 21.5 MPa to 26.4 MPa (by ~23 %) (Fig. 7a), yield strength and tensile strength increase correspondingly from 7 MPa to 18 MPa (by 2.4 times) and from 27 MPa to 39 MPa (by ~44 %) (Fig. 7b). Experimental data also show a distinct parallelism between the yield strength and ultimate tensile. Both curves rise steadily as the aging time is increased up to 30 days. Elongation decreases from 10 % to 5.3 % (by 47 %), which is in good agreement with results shown in Fig. 4b.



a



b

Figure 7. Average values of tensile properties vs. aging time: a – Young's modulus; b – yield strength and ultimate tensile

A higher Young's modulus indicates that the investigated Pb–Sb–Sn–As–Se alloys become stiffer after natural aging and require higher force to be deformed. The yield strength is most affected by the second-phase particles precipitated during aging because they inhibit dislocation and grain boundary movement and decrease permanent deformation of the alloys when stresses exceed the yield strength. As the number of second-phase particles is increased by longer aging time, the yield strength reaches a highest value. The ultimate tensile data confirm the results obtained in other mechanical tests on P-0.5 tensile testing machine (Fig. 4a). The yield strength is proportionally lower than the ultimate tensile strength during the studied period of natural aging. This is due to the grain structure and second-phase particle distribution in the structure of the alloys. Lower amounts of alloying elements are present in solution which precipitate as second-phase particles within the lead matrix. This location of the precipitates is less effective in increasing the yield strength by blocking the motion of dislocations within the grains.

### CONCLUSIONS

In this study, the effects of alloying elements, casting conditions and aging time during storage on the tensile properties of the Pb–Sb–Sn–As–Se grid alloys were investigated. From the experimental results and their analysis, the following conclusions can be made. Ultimate tensile strength and elongation results for the alloys in as-cast condition are quite irregular, but in general do not change greatly with changing composition within the range of Sb (1.55–

1.77 wt.%); Sn (0.11–0.17 wt.%); As (0.097–0.13 wt.%); Se (0.018–0.025 wt.%); impurities (0.0217–0.0533 wt.%); Pb – the remainder. Most of the as-cast Pb–Sb–Sn–As–Se alloys simultaneously combine enhanced values of ultimate tensile strength and elongation, which made them valuable for structural grid components that experience high dynamic loads. Increase in a heating temperature of casting mold causes the slight decrease in the tensile strength but does not significantly affect the elongation. Pouring of the molten metal into a casting mold preheated in the temperature range between 110 °C and 150 °C was found to be effective in stabilizing the tensile properties of the studied grid alloys.

The storing of Pb–Sb–Sn–As–Se alloys for 30–33 days does not weaken them in any degree. Due to natural aging the structure of the alloys undergoes microscopic changes that increase both the yield strength and the ultimate tensile strength, simultaneously increasing Young's modulus and decreasing elongation. The strongest effect of strengthening is observed during the first 5 days of natural aging. Thus, the investigated alloys strengthen rather rapidly so that the grids can be utilized in a short period of time after production without excessively long aging time or artificial aging.

The work was performed within the framework of research projects of National Academy of Sciences of Ukraine No. 1.3.6.18 “Development of new methods and improvement of known ones to investigate mechanics of transport and energetic systems” (2017-2021) and No. 1.3.6.22 “Development of mathematical models and investigation of ground transport and energetic systems” (2022-2026).

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### ЗМІНА МЕХАНІЧНИХ ВЛАСТИВОСТЕЙ СПЛАВІВ Pb–Sb–Sn–As–Se ДЛЯ СТРУМОВІДВОДІВ СВИНЦЕВО-КИСЛОТНИХ БАТАРЕЙ ПІД ЧАС ПРИРОДНОГО СТАРІННЯ

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Роботу присвячено дослідженню механічних властивостей низькосурм'яних Pb–Sb–Sn–As–Se сплавів для струмовідводів свинцево-кислотних батарей у литому стані та після природного старіння під час зберігання на складі. З використанням універсальних розтяжних машин TIRAtest 2300 і P-0.5 визначено межі міцності на розтяг, межі плинності, подовження і модуль Юнга за кімнатної температури. Для більшості досліджених сплавів у литому стані збільшення межі міцності на розтяг супроводжується збільшенням подовження. Підвищення температури нагріву ливарної форми в температурному інтервалі 70–150 °C практично не впливає на середні значення подовження сплавів, але незначно знижує їх середню межю міцності на розтяг (на ~4%). Внаслідок старіння під час зберігання на складі впродовж 30–33 діб спостерігається підвищення значень межі міцності на розтяг, межі плинності і модуля Юнга, але зниження значень подовження сплавів Pb–Sb–Sn–As–Se для струмовідводів. Такі зміни пов'язані з виділенням частинок вторинних фаз з твердого розчину на основі свинцю, пересиченого сурмою, миш'яком і селеном. Найбільший зміцнювальний ефект спостерігається впродовж перших п'яти діб природного старіння.

**Ключові слова:** свинцево-кислотні батареї; низькосурм'яні Pb–Sb–Sn–As–Se сплави для струмовідводів; природне старіння під час зберігання; випробування на розтяг; межа міцності на розтяг; межа плинності; подовження; модуль Юнга