

THE PROPERTIES OF HARDENED LEAD

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ABSTRACT

Lead hardened through oxide scattering is investigated as raw materials for the fabrication of the support gratings of lead accumulators in order to increase their life service. For the preparation of lead powder hardened through oxide scattering we used air jet pulverisation, a technique specific to powder metallurgy. Two research approaches are presented in the paper: one in which the oxide was introduced through oxidised lead powder, and the other in which additional lead oxide was introduced. Oxide dispersion into metallic matrix was achieved by pronounced deformation through extrusion of the powder. During extrusion, the particles are compelled to pass through the mould calibration zone and consequently they are considerably deformed. As an effect of this deformation, the particles of fragile oxide which cover the lead powder particles are crashed and carried away to the material flowing through the matrix longitudinally on the extruded semi-product. The content of oxide introduced in matrix by the oxidised lead powder depends on the powder particle size. This paper presents the effect of the powder particle size and oxide phase dispersion in the metallic matrix on the mechanical, chemical and electrical properties of the extruded semi-products. The semi-products made from lead powder belonging to the grain size class of $< 40 \mu\text{m}$ and oxide particles allowance have the highest mechanical resistance and the best corrosion behaviour. Using controlled oxidised lead powder better extruded semi-products are obtained in terms of mechanical resistance, corrosion behaviour and electrical conductivity as compared with lead and Pb-Sb alloy. A higher electrical conductivity represents an important advantage especially for the starting accumulators where the voltage fall should be as low as possible.

KEYWORDS: mechanical resistance, lead accumulators, oxide dispersion

1. Introduction

Lead hardened through oxide scattering is investigated as raw materials for the fabrication of the support gratings of lead accumulators in order to increase their life service [1-2].

Two research approaches in order to obtain hardened materials by oxide phase dispersed in Pb metallic products were discussed previously [3-5]. In the first variant partially (superficial) oxidised Pb metallic powder has been used. In the second approach fine lead oxide particles $\text{PbO}\cdot\text{Pb}_2\text{O}_3$ were added to the partially oxidised powder mixture belonging to the finest grain size class ($< 40 \mu\text{m}$). Oxide scattering into the metallic matrix was achieved by deforming the compressed product through extrusion. During extrusion, the particles are compelled to pass through the matrix calibration zone

and consequently they are considerably deformed. As an effect of this deformation, the films of fragile oxide which cover the lead powder particles are crashed and carried away by the material that flows through the mould, thus being obtained a fine and uniform dispersion along the length of the extruded samples. Due to the material flowing under the three-dimensional compression, oxide-free surfaces are created, which facilitate the lead atom diffusion among particles, thus achieving sintering and therefore material consolidation. As a blank assay, were used lead and Pb – 8 % Sb alloy, which were extruded under the same conditions as the powder compressed samples.

This paper presents the effect of the oxide phases dispersion in the Pb metallic matrix on the mechanical resistance, creep strength and electrical resistivity of the lead hardened by oxide dispersion as compared to the properties of lead and Pb-Sb alloy.

1. Experimental conditions

In parallel, eight series of materials have been prepared and investigated in terms of mechanical and electrical properties:

- series I: cast and extruded lead;
- series II: cast and extruded Pb - 8% Sb alloy;

- series III: extruded superficially oxidised lead powder of (56 – 71) μm grain size;
- series IV: extruded superficially oxidised lead powder of (40 – 56) μm grain size;
- series V: extruded superficially oxidised lead powder of < 40 μm grain size.

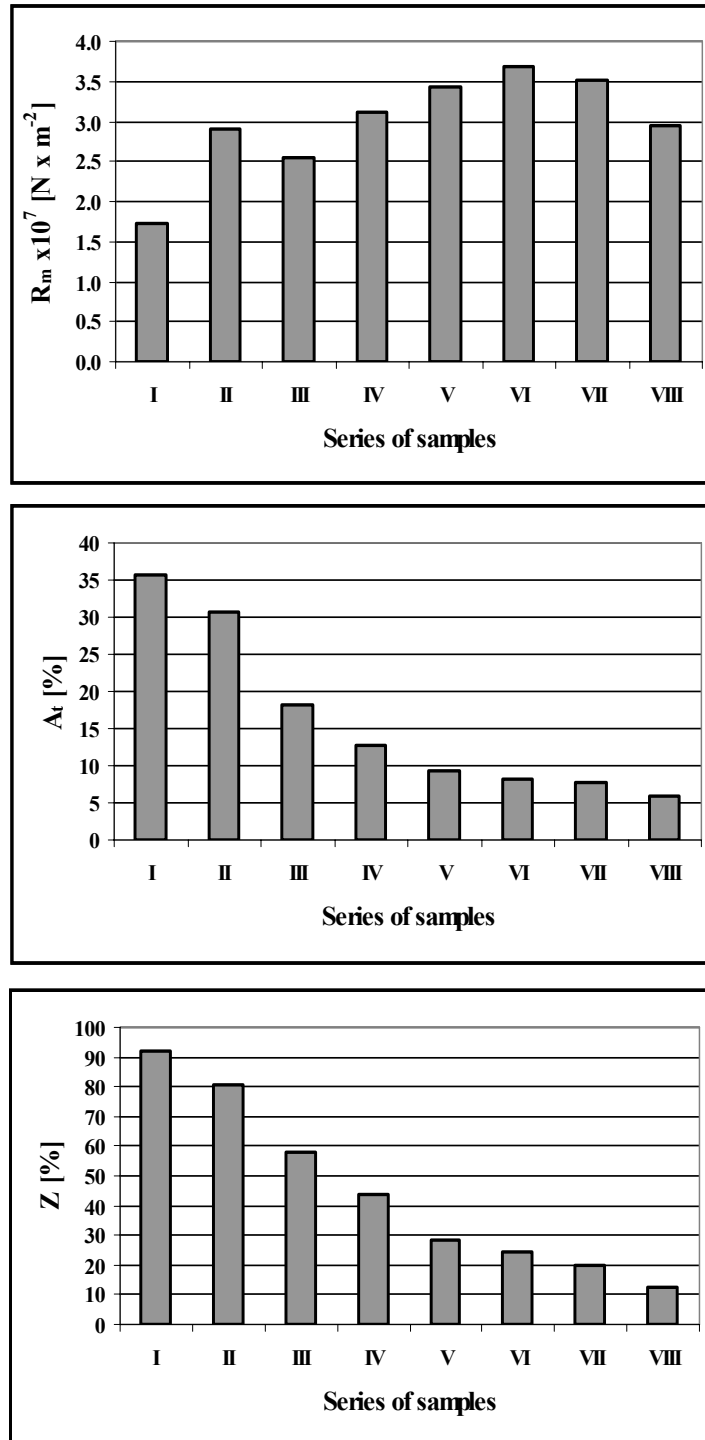


Figure 1. The mechanical characteristics determined at tensile test for the 8 studied series of samples.

– series VI: extruded superficially oxidised lead powder of grain size $< 40 \mu\text{m}$, adding 0.5 % Pb_3O_4

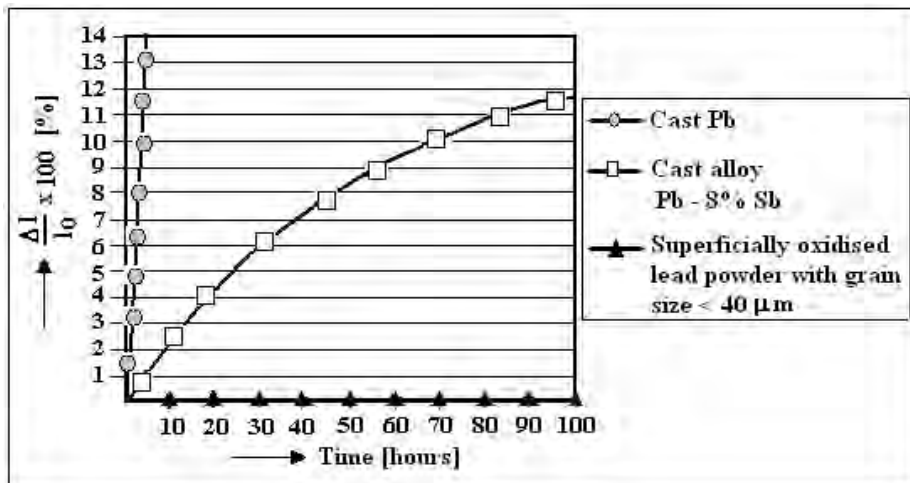
– series VII: extruded superficially oxidised lead powder of grain size $< 40 \mu\text{m}$, adding 1 % Pb_3O_4

– Series VIII: extruded superficially oxidised lead powder of grain size $< 40 \mu\text{m}$, adding 2 % Pb_3O_4 .

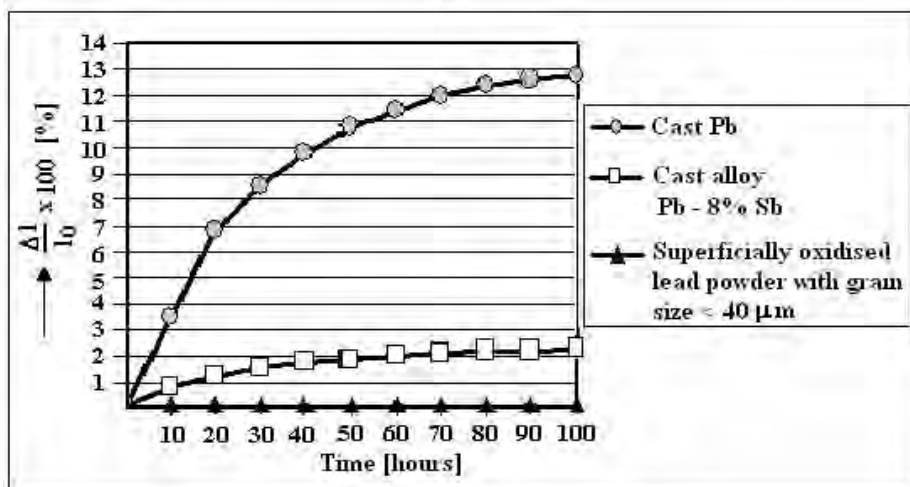
The superficially oxidised powder was obtained by the authors by air-atomization, in an installation with horizontal jet. Compressed samples were achieved in a steel die pressing with the diameter of 20 mm, using the oxidised powder and the mixture of oxidised powder with powder of $\text{PbO}\cdot\text{Pb}_2\text{O}_3$. In order to achieve the oxide-dispersion in the lead matrix, the compressed samples were extruded, thus obtaining the final samples, semi-products (wires), with diameters of 8, 6 and 4 mm and a length greater than 300 mm.

For comparison, lead samples of technical cleanliness and of Pb - 8% Sb alloy, used for acid lead accumulators, having a diameter of 20 mm, were cast. The cast samples were obtained through extrusion under the same conditions as the compressed ones from powders.

In each series under investigation, samples taken from the extruded semi-products having the deformation degree of $\varepsilon_1 = 6.25$, $\varepsilon_2 = 11$, $\varepsilon_3 = 25$ were used. The degree of deformation "ε" was calculated with respect to the initial section before deformation and the final section of the extruded semi-products ($\varepsilon = S_0 / S_f$). The paper presents only the results concerning the samples having the highest deformation degree through extrusion: the assays with the diameter of 4 mm, where the ratio between the initial section and the extruded section equals to 25.



a)



b)

Figure 2. The creep behaviour of the materials made by extrusion and strained at: $\sigma_1 = 0.5 \times 10^7 \text{ Pa}$ (a) and $\sigma_2 = 1 \times 10^7 \text{ Pa}$ (b)

2. Experimental Results

3.1 Mechanical Properties of Hardened Samples

The effect of the following parameters on the mechanical properties was investigated:

- powder particle size (1st variant of research - series III, IV, V);
- amount of particles pre-introduced into the lead oxide (2nd variant - series VI, VII, VIII).

The following determinations were made: tensile strength (R_m), percentage total elongation at fracture (A_t), percentage reduction of area (Z). The results are presented in the figure 1.

Based on these results, the following remarks can be pointed out:

1. The tensile strength of the partially oxidised lead powder samples of different grain size (series III, IV, V) is higher than that of the blank assays made from cast lead (series I).
2. As the powder grain size is being refined (series III, IV, V) there is an increase in the number of fine particles of oxide dispersed in the lead matrix; consequently, there is a considerable increase in the mechanical resistance, while the rigidity gets much improved (this is necessary for the gratings used as support in the lead accumulators) as compared to the materials in the series I and II;

3. The addition of oxide particles to the finest powder in small quantities, below 2 % Pb_3O_4 (series VI, VII) leads to a higher mechanical resistance and a greater rigidity of the deformed materials;
4. Increasing the mass percentage of particles from 0.5 % to 1 % (series VI-VII), decreases slightly the tensile strength; this trend is more pronounced when the mass percentage is increased to 2% (series VIII);
5. Addition of oxide to the finest powder ($< 40 \mu m$) should be made in small amounts (mass percentage below 2%) to prevent the decrease in the mechanical characteristics (R_m , A_t , Z).

3.2 Creep Behaviour of Hardened Samples

The creep behaviour of the samples was evaluated for two constant tensile loads at the temperature of 20°C. The results are presented in figure 2.

One can notice a much better behaviour of the hardened samples by oxide dispersion as compared to the blank assays of Pb or Pb – 8 % Sb.

The high creep resistance is accounted for by the presence in the lead matrix of fine oxide particles crashed by deformation; these particles act as inhibitors and therefore cause a delay in the growth of the new recrystallised grains.

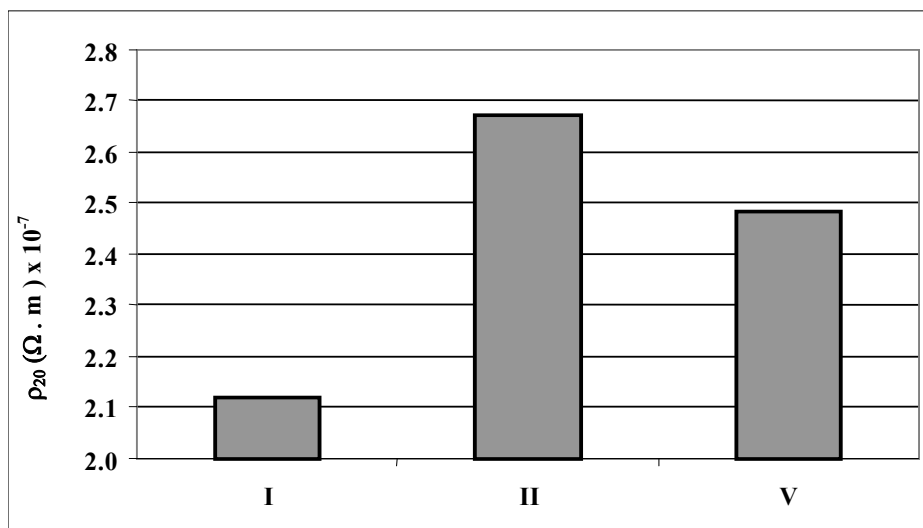


Figure 3. The electrical resistivity of the semi-products obtained by extrusion.

3.3 Electrical Resistivity of Hardened Samples

The measurements to determine the electrical resistivities were carried out by the fall voltage method on the samples being run by a calibrated current of 10 A at the temperature of 20°C. The figure 3 presents the electrical resistivity of the blank assays and for the samples from the V series, hardened by oxide dispersion. One can notice that both the Pb-Sb alloy and the hardened lead by oxide dispersion present a higher resistivity compared to the lead with technical cleanliness.

4. Conclusions

The results of the research show that the oxide dispersion in the lead matrix leads to a significant improvement of the creep strength and to an increase close to 20 % of the mechanical resistance, as compared to the Pb – 8 % Sb alloy, and close to 100 %, as compared to the lead with technical cleanliness. That recommends the lead hardened by oxide dispersion for producing the support gratings for acid accumulators with lead used for cars, as they are particularly subjected to creep, because of the high density of the lead combined with a low mechanical resistance.

By determining the electrical resistivity, it was shown that using the lead hardened through oxide dispersion, instead of the Pb – 8 % Sb alloy, does not lead to the increase of the internal electrical resistance of the accumulator and it keeps the conversion capacity of the high current strength needed for car-starters.

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