

## Morphology, Phase Contents, and Chemical Composition of Nanopowders Produced by the Electrical Explosion of Tin-Lead Alloy Wires

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**Abstract** Phase contents and elemental composition of ultradispersed powders obtained by the electrical explosion of tin-lead alloy powders are investigated. It is demonstrated that during the explosion and subsequent cooling, surface layers of powder particles are enriched in lead compared to the initial alloy. The thermal stability of powders oxidizing in air is also investigated.

**Keywords** : Electrical explosion of wire, Tin-Lead alloy, Ultra fine powder, Nano particle

### 1. Introduction

The method of electrical explosion of a conductor (EEC) is promising for obtaining a diversity of materials with exotic physicochemical properties<sup>1)</sup>. The EEC process is characterized by high current densities ( $j > 10^{10}$  A/m<sup>2</sup>), fast metal heating (with a rate  $> 10^7$  K/s) to high temperatures ( $T > 10^4$  K)<sup>2)</sup>, and a very high power density ( $\sim 10^{14}$  W/m<sup>3</sup>)<sup>3)</sup>. The refractoriness of initial metals and large difference in their melting points present no problems for the EEC technology. The advantage of this technology of electrical explosion of conductors is that it allows alloys and intermetallic compounds in the ultradispersed state to be produced<sup>4)</sup>.

The morphology, phase contents, and dynamics of oxidation of powders produced by the electrical explosion of brass wires in an inert gas atmosphere were previously studied in<sup>5)</sup>. The content of the more refractory component (brass) increased from the periphery to the center of the conductor, and the increase of the energy deposited into the conductor was accompanied by the formation of finer powders and the increase in the relative fraction of the initial  $\alpha$ -phase depleted of zinc. Brass and zinc differ considerably by their physical properties; for example, their melting points are 1083 and 419°C, that is, they differ by a factor of  $\sim 2.5$ .

The present paper studies the morphology, phase,

and chemical composition of ultradispersed powders (UDP) produced by the electrical explosion of tin-lead alloy conductors.

### 2. Materials and Experimental Procedure

The UDP samples were produced using an experimental setup equipped with an automatic mechanism of conductor feeding. The frequency of electrical explosion repetition was in the range 10.6 Hz<sup>4)</sup>. Argon at a pressure of  $3 \cdot 10^5$  Pa was used as a working gas. Conductors were exploded in the fast explosion regime (according to the Bennet classification<sup>6)</sup>); electrical explosions were with an arc stage and an infinite current pause. The specific deposited energy and the energy in the arc stage were adjusted by changing the charging voltage, the charging capacitance, or the length of the explosive conductor. The tin-lead alloy wire of 0.53 mm in diameter was used as an explosive conductor. The length of the explosive conductor changed from 50 to 80 mm. The powder passivated in the argon atmosphere with the controllable air intake was then analyzed.

A DRON-3.0 x-ray diffractometer was used to evaluate semi-quantitative phase contents of powders. The chemical composition of the UDP surface was analyzed by the method of x-ray photoelectron spectroscopy (XPS) using a JSM-840 (Jeol) scanning electron microscope. The degree of dispersion of powders was

characterized by the average bulk-surface particle diameter determined by the thermal argon desorption method (BET). Particle shapes were observed with a scanning electron microscope. The content of residual metal, the melting point, and the initial temperature of UDP oxidation were determined by the method of differential thermal analysis with allowance for the phase contents of samples in the initial and fully oxidized states (on heating in air with a rate of 7.6 deg/s to a temperature of 920°C).

### 3. Results and Discussion

According to the data of transmission electron microscopy, powders were polydispersed, and the particles had spherical shapes or spherical shapes with slight faceting (Fig. 1). Particles of micron sizes (1–10  $\mu\text{m}$ ) with smooth surfaces and the submicron fraction of the powder enveloping these particles are observed in the photograph shown in Fig. 1(a). The shapes of the submicron particle fraction are close to

spherical ones (Fig. 1(b)).

The x-ray phase-content analysis demonstrates that the examined UDP samples have similar reflection patterns corresponding to those of the raw material. Table 1 presents the results of x-ray photoelectron spectroscopy of the raw material (sample 1), resultant UDP (samples 26), and submicron fraction of sample 2 (sample 7) separated by sedimentation. According to the data of integral probing of UDP samples (with the sample probing area  $S \approx 200 \mu\text{m}^2$ ), the content of tin atoms in the surface and subsurface layers was in the range 51.17–63.21 at%, and the content of lead atoms was in the range 35.47–46.37 at%. Thus, the surface and subsurface layers of powder particles are enriched in lead compared to the initial alloy (sample 1 in Table 1). This enrichment can be explained by the higher diffusion coefficient of tin atoms compared to that of lead atoms in the vicinity of the crystallization point of particles cooled from the surface in the process of separation of EEC products. The data of integral probing (samples 26 in Table 1) demonstrate that the percentage of lead increases when the specific energy deposited into the conductor  $W/W_c$  decreases. This can be explained by the fact that when the specific energy deposited into the conductor  $W/W_c$  decreases, the velocities of separation of the explosion products also decrease and hence their cooling rates also decrease. As a result, the diffusion processes decelerate, the diffusion period increases, and the atoms with higher diffusivity have time to diffuse into hotter internal layers of particles. Of definite interest is the fact that surface and subsurface layers of particles of the submicron fraction contain 8 at% of brass (sample 7 in Table 1). Such a high content of brass in the surface and subsurface layers of particles (with the layer thickness  $>5 \cdot 10^8 \text{ m}$ ) of the submicron fraction can be explained by higher volatility of brass compared to lead and tin. As a result, during electrical explosion the atoms are transformed predominantly into the vapor (cluster) phase from which the submicron fraction of the powder is formed by coagulation. Higher rate of cooling of the vapor products of the electrical explosion limits the period of the diffusion process, and copper atoms have no time to diffuse into hotter internal regions of particles. Probing of individual particles demonstrates that the contents of tin and lead atoms may increase or decrease compared to their contents in the raw material (samples 14 in Table 1).

The average bulk-surface diameter of particles

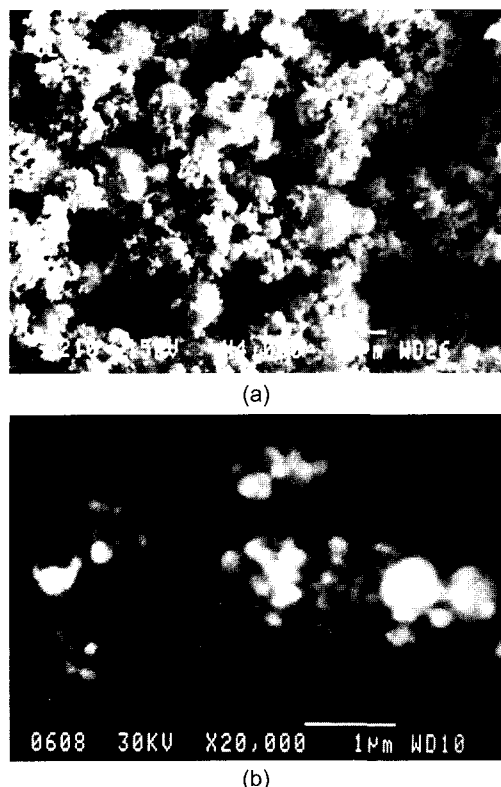


Fig. 1. SEM photos of Sn-Pb particles produced by EEC : (a)  $\times 4000$  and (b)  $\times 20,000$ .

**Table 1. Contents of metal elements in surface and subsurface layers from the data of XRPES**

Sample No.	$W/W_c$	$W_d/W_c$	Object of research	Pb (at.%)	Sn (at.%)	Cu (at.%)
1	–	–	Raw material, integral probing ( $S \approx 200 \mu\text{m}^2$ ).	30.98	67.16	1.86
2	1.12	0.03	UDP, integral probing ( $S \approx 200 \mu\text{m}^2$ )	37.98	62.02	
			Particle diameters: 1 $\mu\text{m}$	32.98	67.02	
			2 $\mu\text{m}$	42.51	57.49	
3	0.97	0.01	7 $\mu\text{m}$	19.09	80.91	
			UDP, integral probing ( $S \approx 200 \mu\text{m}^2$ )	39.42	60.58	
			Particle diameters: 1 $\mu\text{m}$	28.30	71.70	
4	0.67	0.00	3 $\mu\text{m}$	29.02	70.98	
			5 $\mu\text{m}$	5.19	94.81	
			UDP, integral probing ( $S \approx 200 \mu\text{m}^2$ )	46.27	53.73	
5	0.51	2.16	Particle diameters: 1 $\mu\text{m}$	45.98	54.02	
			4 $\mu\text{m}$	31.44	68.56	
			9 $\mu\text{m}$	6.52	93.48	
6	1.2	0.29	UDP, integral probing ( $S \approx 200 \mu\text{m}^2$ )	46.37	53.63	
7	1.12	0.03	UDP, integral probing ( $S \approx 200 \mu\text{m}^2$ )	35.47	63.21	
			UDP, integral probing of submicron fraction of sample 2 ( $S \approx 200 \mu\text{m}^2$ )	40.83	51.17	8.00

**Table 2. Thermal stability of powders oxidizing in air**

Sample No.	$W/W_c$	$W_d/W_c$	$\alpha_s$ ( $\mu\text{m}$ )	Initial melting temperature ( $^{\circ}\text{C}$ )	Initial oxidization temperature ( $^{\circ}\text{C}$ )	Increase in the sample mass during heating to $920^{\circ}\text{C}$ (wt%)
–	–	–	–	175	–	–
2	1.12	0.03	0.146	167	200	18.0
3	0.97	0.01	0.161	169	208	17.9
4	0.67	0.00	0.220	167	210	17.8
5	0.51	2.16	0.127	170	200	17.8
6	1.2	0.29	0.117	169	200	17.1

changes from 0.117 to 0.220  $\mu\text{m}$ . Higher degrees of dispersion of powders are observed not only when the specific energy deposited into the conductor  $W/W_c$  increases but also when the energy of the arc stage  $W_d/W_c$  increases (samples 3 and 4 in Table 2). The resultant powders have lower melting points (167–169 $^{\circ}\text{C}$ ) compared to the raw material (175 $^{\circ}\text{C}$ ). The initial oxidation temperature is independent of the average bulk-surface diameter of particles. The masses of the samples heated to 920 $^{\circ}\text{C}$  increased by 17.1–18.0 wt%, which is equivalent to 92–93 wt% of oxidized alloy.

#### 4. Conclusions

1. In the examined ranges of energy deposited into the conductor and energy of the arc stage ( $W/W_c = 0.51-1.2$  and  $W_d/W_c = 0-2.16$ ), initial alloy does not decompose. All UDP samples have similar reflection

patterns that correspond to the pattern of the raw material. At the same time, the homogeneous structure of particle surfaces is broken, namely, the surfaces of powder particles are enriched in lead compared to the lead content in initial alloy.

2. The percentage of lead on the particle surfaces increases as the specific energy deposited into the conductor decreases, whereas the submicron powder fraction is enriched in a more volatile contamination element - copper.

3. The resultant powders have lower melting points (167–169 $^{\circ}\text{C}$ ) compared to the raw material (175 $^{\circ}\text{C}$ ).

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### References

1. D. Morokhov, L. I. Trusov and V. N. Lapovok: Physical Phenomena in Ultradispersed Media. Energoatomizdat, Moscow (1984).
2. V. A. Burtsev, N. V. Kalinin and A. V. Luchinskii: Electrical Explosion of Conductors and Its Application to High-Power Electrical Equipment Systems, Energoatomizdat, Moscow (1990).
3. N. N. Stolovich: Electroexplosive Energy Converters, Naukai Tekhnika, Minsk (1983).
4. E. I. Azarkevich, A. P. Ilin, D. V. Tichonov and G. V. Yablunovskii: Electroexplosive Synthesis of Ultradispersed Powders of Alloys and Intermetallic Compounds, Fiz. Khim. Obrab. Mater., **4** (1997) 85.
5. A. P. Lyashchko, G. G. Savelev and D. V. Tichonov: Morphology, phase contents, and oxidation of powders produced by the electrical explosion of brass wires, Fiz. Khim. Obrab. Mater., **6** (1992) 127.
6. F. D. Bennet: High-Temperature Exploding Wires, Progress in High-Temperature Physics and Chemistry, Vol. 2, Pergamon Press, New York, (1968) 1.