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## Multi-dimensional identification and analysis of nanoparticles in polydisperse aerosols in the workplace air by scanning electron microscopy with energy dispersive X-ray spectroscopy

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### ABSTRACT

**Introduction.** This study examines the impact of unintentionally generated nanoparticles on workers' health in the metallurgical industry. Given the high toxicity and unique penetrating abilities of nano-sized objects, their reliable identification within background polydisperse aerosols is critical. The study substantiates an integrated approach based on scanning electron microscopy and energy-dispersive X-ray spectroscopy. Their combination enables the precise identification of the morphology and elemental composition of individual particles. The described approach is of crucial importance for assessing occupational risks and creating effective prevention systems at work.

**Materials and Methods.** Aerosols from crude lead production were sampled using nylon membrane filters with a 0.2  $\mu\text{m}$  pore size. The morphology and elemental composition of the particles were established by scanning electron microscopy using a Hitachi SU8220 ultra-high-resolution microscope equipped with an Ultim Extreme EDXS detector.

**Results.** Measurements revealed high concentrations of aerosols (0.01–25  $\mu\text{m}$ ) dominated by nanoparticles generated by melt casting. The nanoparticle count reached 100.08 billion/ $\text{m}^3$ , although their mass fraction was low (up to 47.47  $\mu\text{g}/\text{m}^3$ ). Twenty-two chemical elements were identified in the composition of nanoparticles, the main ones being Pb, Na, Zn, As, Sn, and S. Most nanoparticles were multicomponent, usually consisting of three to five and up to eight elements; the aerosol was found to persist in the workplace air for a long time and spread throughout the workshop despite local exhaust ventilation.

**Limitations.** The study of workplace air aerosols was conducted locally in the area of core crude copper smelting operations.

**Conclusions.** Electron microscopy combined with X-ray fluorescence enabled detailed identification of nanoscale components within complex polydisperse aerosols. Owing to their high spatial resolution and opportunities of elemental microanalysis, these techniques helped to obtain reliable information on not only the morphology and dispersion of particles but also on their unique multicomponent composition, which allowed establishing patterns in the formation of multicomponent particles and determining their distribution by technological process phases.

**Keywords:** crude lead production; polydisperse aerosol; complex aerosol; nanoparticles; electron microscopy; nanoparticle identification technique; elemental composition of nanoparticles

**Compliance with ethical standards.** This study does not require the submission of a biomedical ethics committee opinion or other documents.

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## Многомерная идентификация и анализ наночастиц в составе полидисперсных аэрозолей воздуха рабочей зоны на основе сканирующей электронной микроскопии в сочетании с энергодисперсионной рентгеновской спектроскопией

ФБУН «Екатеринбургский медицинский научный центр профилактики и охраны здоровья рабочих промышленных предприятий» Федеральной службы по надзору в сфере защиты прав потребителей и благополучия человека, Екатеринбург, 620014, Россия

### РЕЗЮМЕ

**Введение.** Исследование посвящено проблеме воздействия непреднамеренно образующихся наночастиц на здоровье работников металлургической отрасли. С учётом высокой токсичности и уникальной проникающей способности нанообъектов критически важны такие задачи, как их достоверная селекция, идентификация в составе фоновых полидисперсных аэрозолей. В работе обоснован интегративный подход, сочетающий сканирующую электронную микроскопию и энергодисперсионную рентгеновскую спектроскопию. Такая комбинация позволяет точно идентифицировать морфологию и элементный состав индивидуальных частиц. Разработанный комплексный подход имеет определяющее значение для оценки профессиональных рисков и создания эффективных систем профилактики на производстве.

**Материалы и методы.** Исследование аэрозолей производства черного свинца проводили с использованием мембранных нейлонных фильтров (поры 0,2 мкм). Морфологию и элементный состав частиц изучали методом сканирующей электронной микроскопии на микроскопе сверхвысокого разрешения Hitachi SU8220, оснащённом ЭДРС-детектором Ultim Extreme.

**Результаты.** Изучение производства черного свинца выявило высокую концентрацию аэрозолей (0,01–25 мкм) с преобладанием наночастиц, образующихся преимущественно при розливе расплавов. Счётная концентрация наночастиц достигала 100,08 млрд/м<sup>3</sup>, хотя их массовая доля была невелика (до 47,47 мкг/м<sup>3</sup>). В составе наночастиц идентифицированы 22 элемента, основные из которых Pb, Na, Zn, As, Sn, S. Установлено, что большинство наночастиц были многокомпонентными (чаще всего 3–5 элементов, реже до 8), а аэрозоль сохранялся в воздухе длительное время, распространяясь по всему цеху, несмотря на местную вентиляцию.

**Ограничения исследования.** Исследование аэрозоля воздуха рабочей зоны выполнено локально в области проведения основных работ при плавке черного свинца.

**Заключение.** Применение комплекса методов электронной микроскопии в сочетании с рентгенофлуоресцентным анализом позволило провести детальную идентификацию наноразмерных компонентов в составе сложных полидисперсных аэрозолей. Благодаря высокому пространственному разрешению и возможности элементного микроанализа эти методы обеспечили получение достоверных сведений о морфологии и дисперсности частиц и, что очень важно, их уникальном многокомпонентном составе. Это позволило установить закономерности формирования многокомпонентных частиц и определить их распределение по фазам технологического процесса.

**Ключевые слова:** производство черного свинца; полидисперсный аэрозоль; аэрозоль сложного состава; наночастицы; электронная микроскопия; методика идентификации наночастиц; элементный состав наночастиц

**Соблюдение этических стандартов.** Исследование не требует представления заключения комитета по биомедицинской этике или иных документов.

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## Introduction

In modern occupational health and medicine, the issue of the impact of nanoparticles (NPs, < 100 nm) on workers in metallurgical and metalworking industries is becoming particularly pressing. Technological processes characterized by high temperatures and significant energy consumption (smelting, welding, cutting, and electrical discharge machining) are accompanied by the incidental generation of large volumes of nanoparticles. According to the results of air quality monitoring, concentrations of ultradisperse aerosols in industrial settings can reach peak values of 7.8 million particles per cm<sup>3</sup> [1–11]. Experimental and epidemiological data indicate that inhalation exposure to fine particulate matter triggers the development of systemic respiratory, cardiovascular, immune, and neurological disorders [1, 12–19].

Despite the proven risks specific to nanoparticles, current regulations are largely based on mass-based indicators of total aerosol, which leads to an underestimation of the actual threat posed by the nanofraction. This regulatory and methodological gap represents a key barrier to ensuring safety of workers at metallurgical plants, where complex mixtures of metal and metal oxide NPs are regularly generated [2, 6, 20, 21]. Therefore, the development of a reliable methodology for the identification and quantification of nanoparticles in the workplace air is a priority. Scanning electron microscopy (SEM), combined with energy-dispersive X-ray spectroscopy (EDX), enables the analysis of individual nanoscale aerosol particles, including the determination of the multi-element composition of samples [22–26]. The development of a comprehensive approach to identifying the nanofraction of aerosols based on these methods is crucial for risk assessment and the implementation of effective preventive measures in industrial settings.

*The aim of this study is to identify nanoparticles within polydisperse aerosols of complex composition in the workplace air of a crude lead production facility using electron microscopy and X-ray fluorescence analysis.*

## Materials and Methods

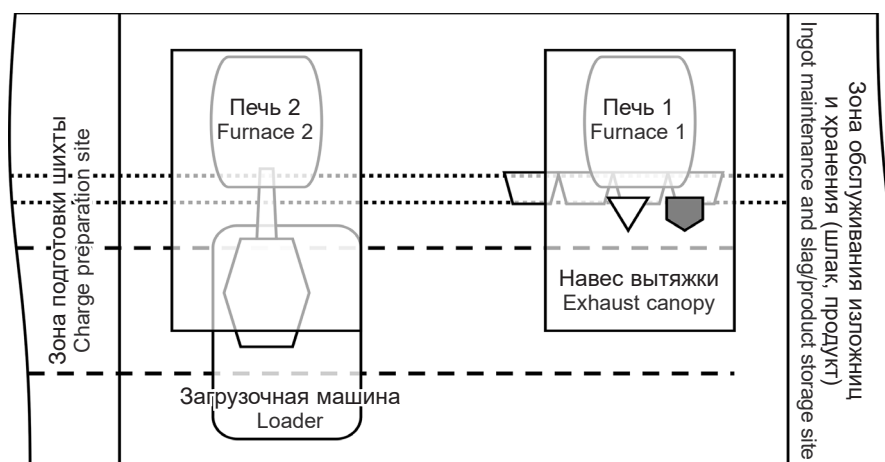
**Description of the production process.** The study was conducted

at a plant producing crude lead from secondary raw materials (scrap batteries and waste from copper smelting). The site houses two short drum furnaces equipped with extraction systems. The furnaces are loaded with prepared charge materials using a charging machine. Smelting is a reduction process at temperatures of 1,100–1,200 °C; crude lead and slag-dust product are periodically poured into moulds and transported further along the production line. A diagram of the site and sampling points is shown in Fig. 1.

**Particle density analysis.** Samples of settled dust were collected around the perimeter of the site near the sampling point at furnace 1 from horizontal surfaces free of overhanging elements. Collection was carried out using a natural-bristle brush into polypropylene zip-lock bags. A total of 10 dust samples were collected. Prior to analysis, the dust was filtered through a 100 µm mesh sieve and dried in an oven. The particle density of the settled dust was determined using the pycnometric method with a neutral liquid in accordance with GOST 5180–2015\*\*\*\*\*. It should be noted that this standard was developed for soils; however, within the scope of this study, it was used to obtain estimated values for the density of settled dust as an accessible integrated indicator. PMS-0.65 silicone oil (SiliconSnab LLC, Russia) was used as the neutral liquid.

**Aerosol analysis.** For the study of the aerosol by scanning electron microscopy, 47-mm nylon (NY) disc membrane filters with a nominal pore size of 0.2 µm by Sartorius (Sartorius AG, Germany) were used; these had previously been evaluated for their ability to capture the nanofraction of the aerosol [27]. A custom-designed filter holder with a cone (intake orifice of 1.5 cm<sup>3</sup>) was used [28]. Samples were collected using an AirChek TOUCH Pump personal aspirator (SKC Inc., USA). During the smelting of lead-containing recycled materials, 16 aerosol samples were collected; sampling times ranged from 10 to 60 minutes depending on the stage of the process, at an air flow rate of 1.5 L/min (Table 1).

\* ГОСТ 5180–2015 Грунты. Методы лабораторного определения физических характеристик. М.: Стандартинформ; 2016.



**Рис. 1.** Схема участка короткобарабанных печей: треугольник – рабочее место плавильщика (в момент выпуска шлака и продукта); трапеция – изложницы; пятиугольник – точка пробоотбора аэрозоля.

**Fig. 1.** Schematic diagram of a short-drum furnace section: triangle – smelter's workplace (at the moment of slag and product tapping); trapezoid – molds; pentagon – aerosol sampling point.

Таблица 1 / Table 1

### Stages of crude lead smelting with the designation of the collected aerosol samples and description of concomitant processes in the workshop

Этапы плавки черного свинца с обозначением отобранных проб аэрозоля и описанием работ, проходящих в это время в цехе

| Этап<br>Stage                                                                                                       | Обозначение<br>пробы<br>Sample designation | Временные<br>промежутки, мин<br>Time intervals, min | Комментарии и описание работ, проходящих параллельно<br>Comments and description of concomitant processes                                                                                                                                                                                                                   |
|---------------------------------------------------------------------------------------------------------------------|--------------------------------------------|-----------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Загрузка печи<br>Furnace loading (FL)                                                                               | ЗП<br>FL                                   | 0–15                                                | Загрузка началась через 15 мин после завершения слива предыдущей партии<br>Loading started 15 minutes after the previous batch was poured out                                                                                                                                                                               |
| Плавка<br>Melting (M)                                                                                               | П-1<br>M-1                                 | 15–45                                               | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | П-2<br>M-2                                 | 45–75                                               | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | М-3<br>P-3                                 | 75–105                                              | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ДЗП<br>AFL                                 | 105–120                                             | —                                                                                                                                                                                                                                                                                                                           |
| Дозагрузка печи<br>Additional furnace loading (AFL)                                                                 |                                            |                                                     |                                                                                                                                                                                                                                                                                                                             |
| Плавка<br>Melting                                                                                                   | П-4<br>M-4                                 | 120–180                                             | Разделка лётки и ВШП на печи 2. Кантовка изложниц в зоне обслуживания<br>Cutting the taphole and pouring of slag, matte, and middlings from furnace No. 2. Turning molds in the service area                                                                                                                                |
|                                                                                                                     | П-5<br>M-5                                 | 180–210                                             | ВШП и ВС на печи 2. Транспортировка заполненных изложниц от печи 2 в зону обслуживания и хранения через печь 1<br>Pouring of slag, matte, and middlings (PSMM) and pouring of molten lead (PML) from furnace No. 2. Transportation of filled molds from furnace No. 2 to the service and storage area through furnace No. 1 |
|                                                                                                                     | П-6<br>M-6                                 | 210–240                                             | Частичный излив расплава на печи 1. Загрузка печи 2<br>Partial pouring of melt from furnace No. 1. Loading of furnace No. 2                                                                                                                                                                                                 |
|                                                                                                                     | П-7<br>M-7                                 | 240–270                                             | Частичный излив расплава на печи 1<br>Partial pouring of melt from furnace No. 1                                                                                                                                                                                                                                            |
|                                                                                                                     | П-8<br>M-8                                 | 270–300                                             | Частичный излив расплава на печи 1. Дозагрузка печи 2<br>Partial pouring of melt from furnace No. 1. Loading of furnace No. 2                                                                                                                                                                                               |
|                                                                                                                     | П-9<br>M-9                                 | 300–330                                             | Разделка лётки. Кантовка изложниц в зоне обслуживания. Извлечение остывшего ШП из изложниц<br>Taphole cutting. Ingot mold tilting in the service area. Removing cooled slag, matte, and middlings from the molds                                                                                                            |
|                                                                                                                     | ВШП-1<br>PSMM-1                            | 330–340                                             | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ВШП-2<br>PSMM-2                            | 340–350                                             | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ВШП-3<br>PSMM-3                            | 351–360                                             | —                                                                                                                                                                                                                                                                                                                           |
| Выпуск<br>шлакоштейнового<br>промпродукта<br>Pouring of slag,<br>matte, and middlings<br>(PSMM) from the<br>furnace | ВШП-1<br>PSMM-1                            | 330–340                                             | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ВШП-2<br>PSMM-2                            | 340–350                                             | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ВШП-3<br>PSMM-3                            | 351–360                                             | —                                                                                                                                                                                                                                                                                                                           |
| Выпуск свинца<br>Pouring the molten<br>lead from the furnace<br>(PML)                                               | ВС-1<br>PML-1                              | 360–370                                             | —                                                                                                                                                                                                                                                                                                                           |
|                                                                                                                     | ВС-2<br>PML-2                              | 370–380                                             | Загрузка началась через 15 мин после завершения слива предыдущей партии<br>Loading started 15 minutes after the previous batch was poured out                                                                                                                                                                               |
|                                                                                                                     | За весь период<br>For the entire period    | Сред.<br>Mean                                       | 0–380                                                                                                                                                                                                                                                                                                                       |

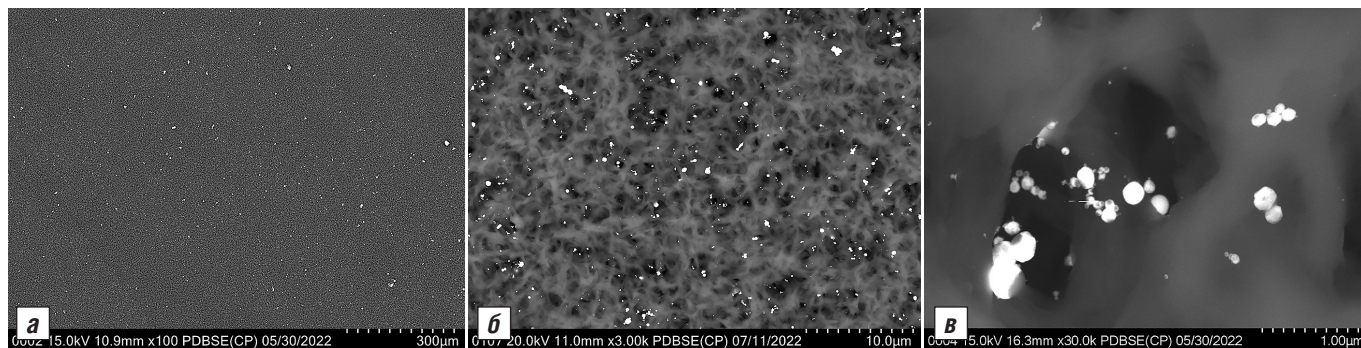


Рис. 2. Электронные микрофотографии частиц аэрозоля на поверхности фильтра при увеличении: а –  $\times 100$ ; б –  $\times 3000$ ; в –  $\times 30\,000$ .

Fig. 2. Electron micrographs of collected aerosol particles at а –  $\times 100$ ; б –  $\times 3,000$ ; and в –  $\times 30,000$  magnification.

The filters were affixed to workbenches using conductive carbon tape for analysis. The filter surfaces were cleaned in a ZONE II ozone cleaning system (Hitachi, Japan). The samples were then coated with a 10-nm-thick conductive carbon layer using a Leica ACE600 vacuum sputtering system (Leica, Austria). The filter surfaces were imaged using a Hitachi REGULUS SU8220 ultra-high-resolution scanning electron microscope equipped with a five-section backscattered electron detector (Hitachi, Japan). The filter surfaces were examined at magnifications of  $\times 600$ ,  $\times 1,500$ ,  $\times 6,000$  and  $\times 50,000$ . The number of fields of view ranged from 40 to 150 depending on the particle load on the filter. The lower threshold for the number of particles analyzed was 1,000 at each magnification.

The elemental composition of the nanoparticles was identified using an Ultim<sup>®</sup> Extreme windowless 100 mm<sup>2</sup> EDAX detector at an accelerating voltage of 20 kV. Prior to the analysis of each sample, the detector was optimized using a standard cobalt sample for X-ray fluorescence analysis. Both individual nanoparticles and clusters of up to several dozens of nanoparticles were analyzed, provided there were no larger particles in the field of view. The least number of spectra obtained for a single sample was 100.

**Data analysis.** Electron micrographs were analyzed using the open-source ImageJ software offering automated object detection, measurement of their projected area ( $A$ ), the minimum diameter of the particle's projected area ( $d_m$ ), and the aspect ratio (AR). For each particle, the equivalent projected diameter ( $d_p$ ) and the particle volume ( $V$ ) were calculated.

The diameter  $d_p$  represents the diameter of a circle having the same area as the dust particle, as projected onto a two-dimensional image, and is calculated as follows:

$$d_p = \sqrt{(4 \cdot A) / \pi}. \quad (1)$$

The volume of the particles was determined following the approach of Anderson et al. [29], according to which the height of a particle is equal to its smallest dimension (the minimum diameter of the particle's projected area):

$$V = (\pi \cdot A \cdot d_m) / (6 \cdot \sqrt{AR}). \quad (2)$$

To calculate the number concentrations, the particles falling within the range were counted, and for volume concentrations, the sum of the volumes of these particles was determined. Measurement results obtained at different magnifications were combined by means of a proportional conversion from large particles to small ones.

When calculating the mass concentrations of nanoparticles, their elemental composition and the identification of unique nanoparticle compounds (NPCs) formed during the production of crude lead, the following assumptions were made:

- 1) the density of the particles in the resulting aerosol is uniform, regardless of the size and composition of these particles;
- 2) the proportions of unidentified elements in the composition of nanoparticles, including H, C, O and N, are negligible relative to the identified elements;
- 3) the elemental composition of all nanoparticles within a single analysis area is homogeneous; and
- 4) the contribution of nanoparticles smaller than 20 nm to the volume and, consequently, mass concentration of particles is negligible.

Thus, the total mass concentration of nanoparticles ( $C_m$ ,  $\mu\text{g}/\text{m}^3$ ) was calculated using the formula:

$$C_{m\text{ ич}} = C_{v\text{ ич}} \cdot \rho, \quad (3)$$

where  $C_v$  is the total volumetric concentration of NPs,  $\text{mm}^3/\text{m}^3$  and  $\rho$  is the density of particles,  $\text{g}/\text{cm}^3$ .

The mass concentration of identified chemical elements ( $X_m$ ,  $\mu\text{g}/\text{m}^3$ ) was calculated using the formula:

$$\bar{X}_{m\text{ ич}} = (x_i \cdot C_{m\text{ ич}}) / 100\%, \quad (4)$$

where  $x_i$  is the mean relative weight percentage of the element in the sample, %, and  $x_i$  is the relative weight percentage of the element in the  $i$ -spectrum, %.

## Results

Electron microscopy findings revealed that the samples of suspended particles collected at the smelter's workplace in a lead-containing scrap processing plant were characterized by size heterogeneity: the particle diameter varied over a wide range of 0.01–25  $\mu\text{m}$ . The particles were formed as a result of a condensation process and therefore were predominantly regular spherical or cubic in shape (Fig. 2).

Furthermore, heterogeneity in the morphology of the aerosol particles was observed (Fig. 3).

The examination of the overall size distribution of the aerosols showed that the total count of aerosol particles ranged from 0.13 to 138.58 billion/ $\text{m}^3$ , depending on the stage of the crude lead smelting (Table 2).

To calculate the mass concentrations of fine particles, the mean density of aerosol particles was determined by estimating the densities of the particulate matter. According to the study results, the density of dust particles in the furnace area was  $4.76 \pm 0.61 \text{ g}/\text{cm}^3$  (mean  $\pm$  standard deviation), with a minimum/maximum range of 3.84/6.05  $\text{g}/\text{cm}^3$ . When interpreting the results, it was taken into account that settled dust may differ in morphology, degree of agglomeration and phase composition from aerosol particles suspended in the workplace air. Consequently, the density values obtained were regarded as approximate and were used for a rough estimate of particle mass concentrations.

Analysis of the particle size distribution of the aerosols revealed the predominance of fine particles. However, the proportion of fine particles in the peak volume concentration did not exceed 2.1% (Fig. 3). The number, volume, and mass concentrations of fine particles ranged from 0.06 to 100.08 billion/m<sup>3</sup>, 0.006–9.958 million μm<sup>3</sup>/m<sup>3</sup>, and 0.03–47.47 μg/m<sup>3</sup>, respectively.

When investigating the elemental composition of fine particles, both individual particles and their aggregates were analyzed (Fig. 4).

Based on the results of the elemental analysis, 22 elements from the periodic table were identified in the composition of nanoparticles (Table 3).

Elemental analysis of the nano-sized aerosol generated during crude lead smelting revealed significant heterogeneity and variability in the elemental composition of the nanoparticles depending on the smelting stage and the operations carried out simultaneously (see Table 1). The most common elements in the nanoparticles were Pb, Na, Zn, As, Sn, and S. Their frequency of occurrence varied depending on the smelting stage; however, they were detected in the nanoparticles of all the samples tested. Furthermore, 90–100% of the NPs contained Pb, whilst the occurrence of other priority elements varied significantly (from 7–34 to 49–100%).

Taking into account the results of the preliminary analysis of the elemental composition of the NPs and the morphological heterogeneity of the aerosol particles, it was decided to identify NP compositional units (Fig. 3). The identification of the nanoparticle composition was based on data obtained during the elemental composition identification stage using the EDX method. For each aerosol sample, a list of nanoparticle compositions was determined. The number of nanoparticles belonging to each identified nanoparticle composition was then calculated. The number concentration of the nanoparticle composition ( $C_{n\text{ con.np}}$ , units/m<sup>3</sup>) was determined using the formula:



Рис. 3. Электронная микрофотография частиц аэрозоля, образующихся при плавке черного свинца (чем светлее объект, тем больше атомная масса химических элементов, из которых он состоит).

Fig. 3. Electron micrographs of aerosol particles generated during crude lead smelting (the lighter the object, the greater the atomic mass of the chemical elements of which it is composed).

$$C_{n\text{ con.np}} = C_{n\text{ np}} \cdot (n_{\text{con.np}} / n_{\text{np}}), \tag{5}$$

where  $C_{n\text{ np}}$  is the total number concentration of nanoparticles, units/m<sup>3</sup>;  $n_{\text{con.np}}$  is the number of identified nanoparticle compositions in the sample;  $n_{\text{np}}$  is the total number of identified nanoparticles in the sample.

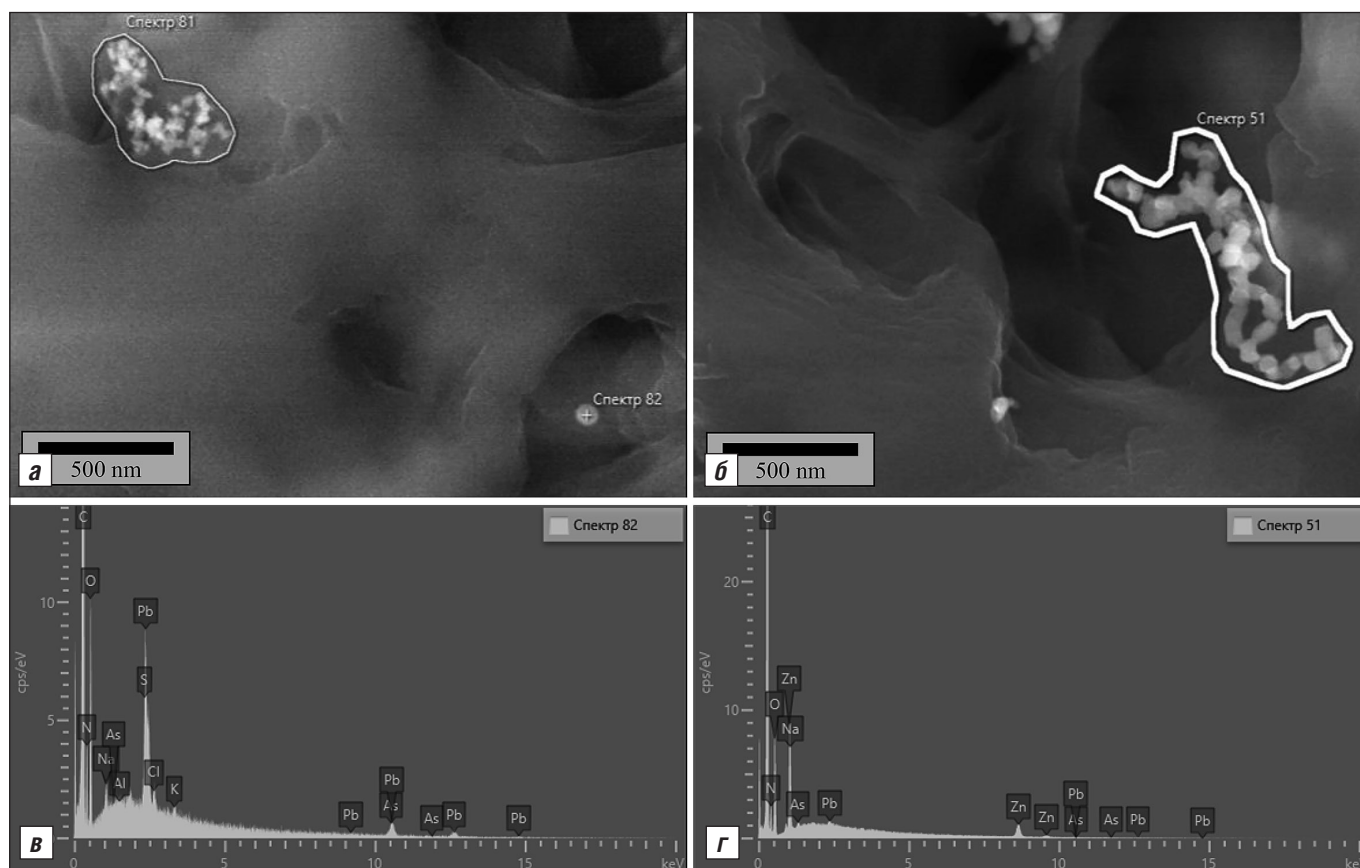
Thus, it was established that during the smelting process, up to 153 NP compounds (ranging from 8 to 45 types depending on the smelting stage) were generated in the workplace air, comprising

Таблица 2 / Table 2

Count, volume, and mass concentrations of nanoparticles (NPs) and total aerosol at different stages of crude lead smelting

Счётные, объёмные и массовые концентрации НЧ и общего аэрозоля на разных этапах плавки черного свинца

| Этап плавки<br>Smelting stage | Счётная концентрация частиц, млрд/м <sup>3</sup><br>Particle count concentration, b/m <sup>3</sup> |                | Объёмная концентрация частиц, млн мкм <sup>3</sup> /м <sup>3</sup><br>Volume concentration, μm <sup>3</sup> /m <sup>3</sup> |                | Массовая концентрация частиц, мкг/м <sup>3</sup><br>Mass concentration, μg/m <sup>3</sup> |                |
|-------------------------------|----------------------------------------------------------------------------------------------------|----------------|-----------------------------------------------------------------------------------------------------------------------------|----------------|-------------------------------------------------------------------------------------------|----------------|
|                               |                                                                                                    |                |                                                                                                                             |                |                                                                                           |                |
|                               | НЧ<br>NP                                                                                           | Общая<br>Total | НЧ<br>NP                                                                                                                    | Общая<br>Total | НЧ<br>NP                                                                                  | Общая<br>Total |
| ЗП / FL                       | 27.30                                                                                              | 37.12          | 2.987                                                                                                                       | 146.8          | 14.24                                                                                     | 700            |
| П-1 / M-1                     | 1.78                                                                                               | 2.50           | 0.200                                                                                                                       | 124.5          | 0.96                                                                                      | 593            |
| П-2 / M-2                     | 0.13                                                                                               | 0.21           | 0.013                                                                                                                       | 157.7          | 0.06                                                                                      | 752            |
| П-3 / M-3                     | 0.08                                                                                               | 0.13           | 0.008                                                                                                                       | 123.2          | 0.04                                                                                      | 587            |
| ДЗП / AFL                     | 0.06                                                                                               | 0.14           | 0.006                                                                                                                       | 501.7          | 0.03                                                                                      | 2392           |
| П-4 / M-4                     | 15.09                                                                                              | 19.92          | 1.437                                                                                                                       | 186.6          | 6.85                                                                                      | 890            |
| П-5 / M-5                     | 57.11                                                                                              | 87.58          | 4.797                                                                                                                       | 839.1          | 22.87                                                                                     | 4000           |
| П-6 / M-6                     | 16.22                                                                                              | 21.44          | 1.490                                                                                                                       | 199.5          | 7.11                                                                                      | 951            |
| П-7 / M-7                     | 1.50                                                                                               | 2.01           | 0.132                                                                                                                       | 113.2          | 0.63                                                                                      | 540            |
| П-8 / M-8                     | 0.67                                                                                               | 0.94           | 0.059                                                                                                                       | 65.0           | 0.28                                                                                      | 310            |
| П-9 / M-9                     | 0.31                                                                                               | 0.62           | 0.034                                                                                                                       | 995.3          | 0.16                                                                                      | 4745           |
| ВШП-1 / PSMM-1                | 1.60                                                                                               | 3.09           | 0.186                                                                                                                       | 1542.8         | 0.89                                                                                      | 7355           |
| ВШП-2 / PSMM-2                | 5.73                                                                                               | 11.14          | 0.798                                                                                                                       | 689.1          | 3.80                                                                                      | 3285           |
| ВШП-3 / PSMM-3                | 12.65                                                                                              | 18.87          | 1.619                                                                                                                       | 472.4          | 7.72                                                                                      | 2252           |
| BC-1 / PML-1                  | 39.03                                                                                              | 58.70          | 3.898                                                                                                                       | 432.2          | 18.58                                                                                     | 2060           |
| BC-2 / PML-2                  | 100.08                                                                                             | 138.58         | 9.958                                                                                                                       | 533.9          | 47.47                                                                                     | 2545           |
| ВШП / PSMM                    | 6.7                                                                                                | 11.04          | 0.87                                                                                                                        | 901.5          | 4.14                                                                                      | 4297           |
| BC / PML                      | 69.6                                                                                               | 98.6           | 6.93                                                                                                                        | 483.0          | 33.03                                                                                     | 2303           |
| Среднее / Mean                | 13.8                                                                                               | 19.8           | 1.310                                                                                                                       | 358.3          | 6.24                                                                                      | 1708           |



**Рис. 4.** Электронные микрофотографии НЧ и спектры ЭДРС: *а* и *в* – операция ЗП; *б* и *г* – операция ВС.

**Fig. 4.** Low frequency electron micrographs and EDX spectra: *a* and *в* – FL operation; *б* and *г* – PML operation.

Таблица 3 / Table 3

Mass concentrations of the main elements in the composition of NPs at various stages of smelting,  $\mu\text{g}/\text{m}^3$

Массовые концентрации основных элементов в составе НЧ на различных этапах плавки,  $\mu\text{кг}/\text{м}^3$

| Этап плавки<br>Smelting stage | Pb    | Zn     | Na    | As    | Sn    | S     | Sb     | Прочие / Other |
|-------------------------------|-------|--------|-------|-------|-------|-------|--------|----------------|
| ЗП / ZP                       | 11.46 | 0.450  | 1.041 | 0.328 | 0.084 | 0.566 | —      | 0.309          |
| П-1 / M-1                     | 0.712 | 0.034  | 0.104 | 0.014 | 0.027 | 0.044 | 0.001  | 0.019          |
| П-2 / M-2                     | 0.043 | 0.005  | 0.007 | 0.001 | 0.003 | 0.002 | 0.0001 | 0.001          |
| П-3 / M-3                     | 0.027 | 0.003  | 0.005 | 0.001 | 0.002 | 0.001 | 0.0001 | 0.0003         |
| ДЗП / AFL                     | 0.017 | 0.003  | 0.003 | 0.001 | 0.001 | 0.001 | 0.0001 | 0.0002         |
| П-4 / M-4                     | 4.140 | 0.943  | 0.880 | 0.235 | 0.424 | 0.165 | 0.025  | 0.041          |
| П-5 / M-5                     | 9.399 | 7.684  | 4.126 | 0.106 | 1.115 | 0.255 | —      | 0.182          |
| П-6 / M-6                     | 3.499 | 2.400  | 0.856 | 0.158 | 0.107 | 0.036 | —      | 0.049          |
| П-7 / M-7                     | 0.288 | 0.201  | 0.074 | 0.028 | 0.014 | 0.008 | 0.0002 | 0.014          |
| П-8 / M-8                     | 0.158 | 0.054  | 0.030 | 0.022 | 0.011 | 0.003 | 0.0005 | 0.001          |
| П-9 / M-9                     | 0.065 | 0.020  | 0.024 | 0.033 | 0.003 | 0.001 | 0.011  | 0.003          |
| ВШП-1 / PSMM-1                | 0.188 | 0.227  | 0.205 | 0.163 | 0.049 | 0.010 | 0.034  | 0.013          |
| ВШП-2 / PSMM-2                | 0.732 | 1.432  | 0.686 | 0.844 | 0.065 | 0.031 | —      | 0.012          |
| ВШП-3 / PSMM-3                | 1.683 | 3.446  | 1.287 | 1.151 | 0.106 | 0.030 | —      | 0.014          |
| ВС-1 / PML-1                  | 3.503 | 9.491  | 3.310 | 1.960 | 0.158 | 0.044 | 0.047  | 0.070          |
| ВС-2 / PML-2                  | 9.632 | 19.42  | 7.930 | 9.351 | 0.879 | 0.131 | 0.129  | 0.000          |
| ВШП / PSMM                    | 0.867 | 1.702  | 0.726 | 0.720 | 0.074 | 0.024 | 0.011  | 0.013          |
| ВС / PML                      | 6.567 | 14.455 | 5.620 | 5.656 | 0.518 | 0.088 | 0.088  | 0.035          |
| Среднее / Mean                | 2.641 | 1.883  | 0.946 | 0.433 | 0.205 | 0.083 | 0.010  | 0.043          |

Примечание. Среднее – средневзвешенная за весь период плавки. Прочие – остальные идентифицированные элементы в составе НЧ (Al, Mo, Cl, Cd, Te, F, Br, Se, K, Fe, Cu, Ca, Si, Ti, P).

Notes: Mean – weighted average for the entire smelting period. Other – Other identified elements in the composition of NPs (Al, Mo, Cl, Cd, Te, F, Br, Se, K, Fe, Cu, Ca, Si, Ti, and P).

Таблица 4 / Table 4

**Priority types of unique nanoparticle combinations (UNPC) (> 1.5% of the total number of NPs) generated in the course of crude lead smelting**  
**Приоритетные виды УС НЧ (> 1,5% от общего числа НЧ), образующихся при плавке черного свинца**

| Операция<br>Operation                                                                                                                          | УС НЧ<br>UNPC    | Счётная концентрация, млрд/м <sup>3</sup><br>Particle count, b/m <sup>3</sup> | Доля УС НЧ от общего числа НЧ, %<br>UNPC proportion of total NPs, % |
|------------------------------------------------------------------------------------------------------------------------------------------------|------------------|-------------------------------------------------------------------------------|---------------------------------------------------------------------|
| За весь период плавки,<br>380 мин (153 УС НЧ)<br>For the entire smelting period,<br>380 min (153 UNPC)                                         | Na/Zn/As/Pb      | 3.41                                                                          | 24.7                                                                |
|                                                                                                                                                | Na/Zn/Pb         | 2.19                                                                          | 15.9                                                                |
|                                                                                                                                                | Na/Zn/Sn/Pb      | 0.94                                                                          | 6.8                                                                 |
|                                                                                                                                                | Na/S/Zn/Sn/Pb    | 0.81                                                                          | 5.9                                                                 |
|                                                                                                                                                | Na/Zn/As/Sn/Pb   | 0.75                                                                          | 5.5                                                                 |
|                                                                                                                                                | Zn/Pb            | 0.52                                                                          | 3.8                                                                 |
|                                                                                                                                                | Pb               | 0.43                                                                          | 3.1                                                                 |
|                                                                                                                                                | Na/S/Pb          | 0.43                                                                          | 3.1                                                                 |
|                                                                                                                                                | Na/S/Zn/As/Sn/Pb | 0.41                                                                          | 2.9                                                                 |
|                                                                                                                                                | Na/Pb            | 0.33                                                                          | 2.4                                                                 |
|                                                                                                                                                | Na/S/Zn/As/Pb    | 0.27                                                                          | 1.9                                                                 |
|                                                                                                                                                | Na/Al/S/Zn/Pb    | 0.20                                                                          | 1.5                                                                 |
| Выпуск шлакоштейнового<br>промпродукта, 30 мин<br>(36 УС НЧ)<br>Pouring of slag, matte,<br>and middlings from the furnace,<br>30 min (36 UNPC) | Na/Zn/As/Pb      | 4.21                                                                          | 56.9                                                                |
|                                                                                                                                                | Na/Zn/As/Sn/Pb   | 0.70                                                                          | 9.4                                                                 |
|                                                                                                                                                | Na/S/Zn/As/Pb    | 0.57                                                                          | 7.7                                                                 |
|                                                                                                                                                | Na/S/Zn/As/Sn/Pb | 0.41                                                                          | 5.6                                                                 |
|                                                                                                                                                | Zn/As/Sn/Pb      | 0.26                                                                          | 3.6                                                                 |
|                                                                                                                                                | Na/Cl/Zn/As/Pb   | 0.15                                                                          | 2.0                                                                 |
| Выпуск свинца, 20 мин<br>(20 УС НЧ)<br>Pouring of lead from the furnace,<br>20 min (20 UNPC)                                                   | Zn/As/Pb         | 0.13                                                                          | 1.7                                                                 |
|                                                                                                                                                | Na/Zn/As/Pb      | 50.51                                                                         | 70.8                                                                |
|                                                                                                                                                | Na/Zn/As/Sn/Pb   | 8.13                                                                          | 11.4                                                                |
|                                                                                                                                                | Na/S/Zn/As/Sn/Pb | 3.60                                                                          | 5.0                                                                 |
|                                                                                                                                                | Na/S/Zn/As/Pb    | 2.64                                                                          | 3.7                                                                 |
|                                                                                                                                                | Zn/As/Pb         | 2.28                                                                          | 3.2                                                                 |
|                                                                                                                                                | Na/Zn/As/Sb/Pb   | 1.37                                                                          | 1.9                                                                 |

between one and eight elements. NP compounds containing three to five elements prevailed. **Table 4** shows the most common types of identified NP compounds and their concentrations.

Despite the diversity of the elemental composition of the nanoparticles, only six types of single-element nanoparticles were detected over the entire observation period – Na, Al, Zn, As, Sb, and Pb. Furthermore, the proportion of Pb, Sb, and Zn particles reached 12.6%, 1.4%, and 0.6% of the total number of particles at certain stages of smelting, respectively, whilst the proportions of other types of single-element particles did not exceed 0.22%.

## Discussion

Microscopic findings showed that samples of suspended particles collected at the smelter's workplace in a lead-containing scrap processing plant were characterized by size heterogeneity: particle diameters varied over a wide range of 0.01–25 µm. The study by Rovin et al. also indicates an upper size limit of 25 µm. However, in the present study, a large number of particles smaller than 100 nm were detected [30].

Furthermore, heterogeneity in the morphology of the aerosol particles was observed. The presence of multiple phases within the aerosol particles points to a mechanism of heterogeneous nucleation, in which vapours of more volatile elements form a surface layer on pre-existing particles of a different elemental composition.

The study of changes in the concentrations and particle size distribution of aerosols over time has established the following:

- the aerosol formed is capable of persisting in the air for a long period even after the relevant process operations have ceased;
- despite the presence of local ventilation systems, operations carried out simultaneously in other areas of the workshop can significantly affect the overall (background) concentration and composition of aerosols throughout the entire workshop;
- the highest concentrations of large particles ( $\geq 1 \mu\text{m}$  in size) were observed during furnace charging (re-charging) and mechanical operations (e.g. cutting of tap holes) as disintegration aerosols; and
- peak concentrations of submicron particles ( $< 1 \mu\text{m}$ ), including fine particles, occurred during the discharge and pouring of molten materials (slag, dross, and crude lead), when the particles formed mainly by condensation.

Furnace operators, who monitored the smelting process and tapped the metal, were most exposed as they worked at the emission source. Thus, the spikes in fine particle concentrations were caused precisely by operations involving the molten metal – its tapping and transfer through the runners.

The study showed that the entire smelting period can be divided into four phases based on the qualitative elemental composition of the molten metal: 1) during the stages from ZP to P-4, Pb predominated in the molten metal, with a gradual decrease and increase in the proportions of Zn, Na, and Sn; 2)

during the period from P-5 to P-7, Pb also predominated in the composition of the molten metal, but its proportion decreased significantly. At the same time, the proportion of Zn increased whilst that of Sn gradually decreased; 3) during stages P-8 and P-9, Pb continued to predominate in the composition of the crude lead, but the proportion of Zn decreased whilst that of As increased, and 4) during the period from VPSH-1 to VS-2, Zn has already predominated in the composition of the crude lead.

This diversity of qualitative characteristics of the elemental composition of crude lead is explained by differences in raw materials used during the smelting process. Under continuous production conditions, an average of four smelting runs were carried out per shift (two furnaces, each with two runs), during each of which either identical or unique raw materials could be used depending on production requirements (production volume targets, order volumes for the supply of specific products by the enterprise). These uncertainties introduce significant distortions into the identification of contributing factors and determination of their levels, thus preventing an objective assessment of the potential impact on workers' health and elaboration of effective preventive measures.

No correlations were found between the size of ultrafine particle agglomerates and the number of all identified elements in the sample, nor with the particle concentrations. This may indicate the specific nature of the technical process (temperature regime, additives), leading to the formation of ultrafine particle agglomerates with a complex composition. Furthermore, as described earlier, there are possible scenarios in which the particles formed had a non-homogeneous composition rather

than a multilayered structure. Primarily, nanoparticles might have formed from elements or substances with a high condensation temperature, whilst substances with a lower condensation temperature condensed directly onto their surfaces.

## Conclusion

Application of a suite of electron microscopy techniques in combination with EDS enabled the detailed identification of nanoscale components within complex polydisperse aerosols. Owing to their high spatial resolution and elemental microanalysis, these methods provided reliable information not only on the morphology and dispersibility of the particles, but also on their unique multicomponent composition, enabling establishment of the patterns governing the formation of multicomponent particles and determination of their distribution across the phases of the technological process.

The approaches to identifying fine particles in the workplace air presented in this paper have provided valuable additional information on the risk factors for metallurgical workers; however, the methodology is labour-intensive and is suitable only for expert studies. Further development of the methodology should focus on its optimization. The most promising approach appears to be the integration of the proposed method with techniques for determining real-time particle counts in aerosols (for particles larger than 5 nm). Such an approach would significantly reduce labour and financial costs of investigating nanoparticles within polydisperse aerosols of complex composition.

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