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Metallurgy

Vacuum sublimators with a rheological displacement of the dispersed medium

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	ABSTRACT
Received: May 27, 2022 Peer-reviewed: June 3, 2022 Accepted: July 27, 2022	Based on the analysis of designs and technological processes carried out in vacuum electric
	furnaces developed to date for the processing of dispersed materials by sublimation of volatile
	components from them, and problems associated with technological processes, technical
	solutions are proposed in the present work in which the movement of concentrate is carried out
	due to rheological properties with direct heating by radiation from the heater to the surface of the
	transported and mixing raw materials. During the development of the equipment, a concept was
	adopted in which the movement of material in the sublimation zone of the furnace is ensured due
	to rheological properties, which opens up the possibility of using materials inert to the sulfide
	atmosphere and realizing heat transfer by radiation to open areas of dispersed material.
	Technological tests on the sublimation of arsenic sulfide compounds from granulated concentrates
	of Nezhdaninsky and Bakyrchik deposits have confirmed the prospects of such a constructive
	design of sublimation processes. The application of the developed equipment in practice will
	ensure the technical and economic efficiency of production in compliance with all environmental
	·····
	requirements with minimal impact on the environment, which is currently a fundamental indicator
	when choosing a particular technology.
	Keywords: Rheology, dispersed material, electric furnace, heating, dearsenation.
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Introduction

Currently, there is a large number of deposits of polymetallic sulfide ores and concentrate productions that contain arsenic, antimony, and mercury in the form of sulfides or other compounds [[1], [2], [3], [4], [5]]. Extraction of valuable components from these types of raw materials with

the help of classical methods is not safe from an environmental point of view due to the significant toxicity of accompanying elements [[6], [7], [8]].

The analysis of the studies performed in the field of arsenic extraction technologies corresponding to the current requirements enables us to make the following conclusion. The arrangement of the preliminary stage of arsenic extraction allows excluding its circulation in the processed products, which, in its turn, contributes to the improvement of the quality of the finished product. That is why the most promising technologies are those which provide for preliminary extraction of arsenic in the form of low-toxic and low-soluble compounds [[9], [10]].

For example, these methods include oxidationsulfiding roasting of granulated concentrate with arsenic removal in a low-toxic form [[11], [12]], and heating arsenopyrite-pyrite-containing of concentrates in reducing or sulfur-containing media with arsenic conversion to sulfide sublimates [[13], [14]]. However, these methods have not found industrial application due to the complexity of strict control over the oxidative and sulfur potential of the gas phase. This problem can be solved using a vacuum-thermal method of arsenic removal [15] in combination with traditional methods of extraction of noble metals from the obtained cinders [16]. It should be noted that the implementation of the process in a vacuum, among other things, enables to reduce the process temperature, and improve the working conditions of the personnel of the enterprise and the environmental situation in the region as a whole.

The employees of the laboratory of vacuum processes in the Institute of Metallurgy and Ore Beneficiation JSC are engaged in the development of vacuum-thermal and vacuum-distillation equipment for the processing of various types of raw materials for several decades [17].

To date, the movement of bulk solids in vacuum apparatuses is provided forced, mainly by fluidization of the directed vibration [18] and sucking of gas flow through a dispersed material layer [19].

Vibro-vacuum electric furnaces are the most advanced design of disperse material processing by fluidization. They have been validated by a pilot and industrial tests in the processing of gold-arsenic concentrates [17]. The material is moved and heated on a screw vibrating conveyor having an internal cavity that is not communicated with the vacuum furnace space. A furnace heater is placed in the inner cavity of the vibrating conveyor.

The main disadvantage of this equipment type is the process of heat transfer to the dispersed material through the central steel tube of the vibrating conveyor resulting in severe overheating from the heater and residual deformations in it. Analyzing the technical design of sublimation processes during the vacuum processing of dispersed raw materials, we can highlight some of the difficulties affecting the quality of the resulting product and the operating time of the equipment. They include the following drawbacks:

- pressure reductions inherent for the vacuum sublimation layer;
- increase in the process temperature during heat transfer to the processed material;
- thermal and corrosion resistance of special steels in vacuum vibro-vacuum furnaces.

It is required to conduct research in the field of new, modern, effective, and environmentally safe equipment for the dearsenation of mineral and manmade materials by vacuum method to solve these problems which is the purpose of this work.

Experimental part

In this regard, we have developed two equipment designs where the movement of bulk materials in the sublimation zone of the furnace is provided by rheological properties. This method of movement opens up the possibility to use materials that are inert in relation to the sulfide atmosphere and to realize the transfer of heat by radiation to the exposed areas of the dispersed material.

The scheme of the first version of the sublimation unit intended to process dispersed materials and the principle of its operation are considered and given by us in [20]. Further design developments were aimed to simplify the manufacturing process of inclined surfaces, where the processed material descends, their placement, and mounting of the column in the sublimator volume. In the first version, the inclined surfaces were made in the form of a column of truncated conical shells facing upwards with a large base. The inclined surfaces in the new version are made in the form of polyhedral truncated pyramids or inclined plates assembled with inclined grooves in the form of a vertical shaft (Figure 1).

The vacuum sublimator scheme with these inclined surfaces is shown in Figure 2. The unit is a sublimator (1) heated by an electric furnace (2), inside which a shaft formed by truncated pyramids (3) is placed. The pyramids are located with their larger bases upwards with a slope angle of 60° that significantly exceeds the natural slope angle of almost all types of bulk materials.

The truncated pyramids are turned on half of the face one relative to the other, and the larger base

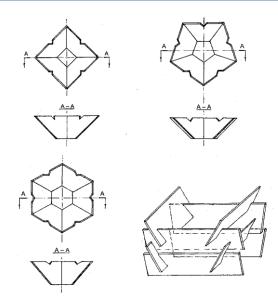
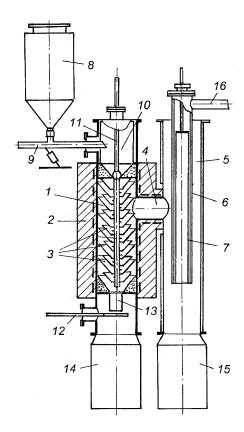


Figure 1 – Type of inclined surfaces in the form of truncated pyramids or plates, forming a shaft in the sublimator



1 – sublimator; 2 – electric furnace; 3 – inclined surface; 4 – steam pipeline; 5 – condenser; 6 – pipe; 7 – fabric filter; 8 – hopper for raw materials; 9 – vibrating loader; 10 – intermediate vessel; 11 – a rod with cone shutters; 12 – vibrating unloader; 13 – mouth; 14 – receiving hopper residue; 15 – receiving hopper condensate; 16 – vacuum wire.

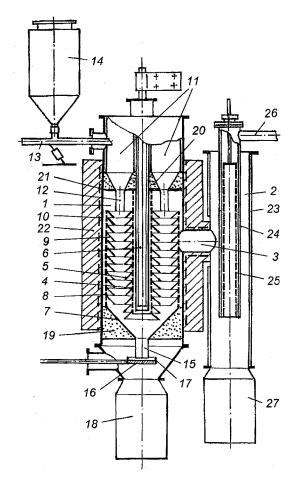
Figure 2 – Schematic diagram of a vacuum sublimation unit with a rheological displacement of dispersed materials

corners touch the sublimator body that ensuring their fixation in the column. The gaps are correlated so that the material pouring out of the upper pyramid or shell plate at the natural slope angle does not "overflow" the upper edge of the larger base below the pyramid or plate. The sublimator is connected by a heated steam line (4) to a watercooled condenser (5). There is a water-cooled coaxially mounted tube (6) inside the condenser inside which a fabric filter for catching fine condensate (7) is placed. The apparatus is equipped with a hopper for raw materials (8) and a vibrating feeder (9) for feeding the latter to an intermediate vessel (10). The intermediate vessel is separated from the sublimator space by a movable hollow rod a cone gate (11), inside which the thermocouple is placed. A vibrating unloader (12) is placed below the sublimator, which together with the bulk material in the mouth (13) forms a gate that separates the receiving hopper of the residue from the processing (14) from the sublimator (1). Arsenic sulfides crumbling from the walls of the condenser accumulate in the hopper (15). The gases are evacuated from the apparatus through a vacuum conduit.

Technological tests of the unit were performed on the model material. When they have performed the dispersed material from the bunker for initial raw materials was fed by means of the vibrating feeder into the intermediate vessel above a sublimator, from where they filled the internal cavity of the shaft formed by truncated pyramids or plates. The treated material was heated by radiation on the exposed surface areas. Such areas are formed by dispersed material due to natural oversaturation and face the external heater.

The processing time of the bulk material (staying in the sublimator) was regulated by the productivity of the vibrating unloader, the plane of which is located with a gap relative to the mouth. Dispersed material in the mouth and on the unloader with sides formed a gate, separating the sublimation volume from the bunker space, which prevented penetration and condensation of the sulfide vapor phase in the latter. The vapor phase from the sublimator through a steam pipeline was directed to the solid-phase condensation of arsenic sulfides in a cyclone-type condenser. The design shown in Figure 3 was developed to increase the performance of the sublimator.

The technology for processing dispersed raw materials containing volatile components is similar to that for the sublimator presented in Figure 2. The difference is that the dispersed material in the sublimator is located in a tube-shaped shaft formed by an inner column of shells of smaller diameter, with the slope of the formation downward, and the outer column of shells of larger diameter, facing upward with their large bases. The sublimated steam exits inside the perforated tube where the heater is located, and outside through the perforated screen into the gap between the shell and the screen, and then through the steam line to the condenser. The external heater prevents the condensation of steam on the sublimator body.



1 – sublimator; 2 – condenser; 3 – steam pipeline; 4 – perforated tube; 5 – shells turned downward; 6 – heater; 7 – collapsible cone; 8 – perforated screen; 9 – shells with the larger base upward; 10 – the body of sublimator; 11 – receiving hopper; 12 – mouth of the receiving hopper; 13 – vibrating loader; 14 – hopper of raw materials; 15 – mouth of collecting cone; 16 – shaker; 17 – sides of the vibration discharger plane; 18 – hopper of the rest; 19, 20, 22 – heat-insulation; 21 – outdoor heater; 23 – cooled condenser housing; 24 – cooled pipe; 25 – fabric filter; 26 - vacuum-pipe; 27 – receiving condensate hopper.

Figure 3 – Scheme of the sublimator with a rheological displacement of increased productivity material

Discussion of results

Technological tests of the equipment on the model material showed the fundamental possibility of abandoning the forced movement of bulk materials in the reaction zone of the sublimator by imposing directional vibrations with the achievement of high raw material dearsenation parameters [[15], [16]].

The tests were conducted at temperatures in the sublimator reaction zone of 600-700 °C. The residual pressure in the system was 0.13 kPa. The residence time of the material in the isothermal zone of the furnace was regulated by changing current loads on the electromagnet windings of the vibration unloader, which, in turn, determined the material unloading rate, and thus the volume of a new portion of the material fed into the reaction space. It was determined that the time when the material was in the reaction zone of the furnace could vary from 5 to 18 minutes. It should be noted that no dust escape was observed in the used design of the sublimation electric furnace with the model material work which, in turn, excludes the additional design of a heated system for rough cleaning of the gas flow from the sublimator against fine dust.

The proposed rheological movement of the dispersed material in the sublimation zone of arsenic sulfides in combination with direct radiation heating of the open surface of the treated material confirmed the prospects of such a design of the technological process. Sublimators of such design can be used for processing both arsenic- and mercury- and antimony-containing raw materials.

Conclusions

Several variants of equipment designs with the rheological movement of the processed material designed to process dispersed arsenic-containing concentrates are proposed as a result of research and design developments.

For practical use of research results, the authors proposed a variant of design with external heating of the sublimator body and the use of inclined planes to transfer the material in the form of plates with grooves, assembled in pairs in a certain way in the form of a vertical shaft.

This sublimation electric furnace design provides a more efficient transfer of heat to the material from

the heated surfaces of the furnace, eliminating mechanical stress on the elements of the furnace, resulting from the imposition of directional vibrational motions occurring in the vibration-vacuum apparatus.

The main advantage of the proposed sublimation electric furnace design is its simplicity which is one of the most important aspects when the equipment is used on an industrial scale.

Conflict of interest

On behalf of all authors, the correspondent author declares that there is no conflict of interest.

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Дисперсті ортаның реологиялық орнын ауыстыратын вакуумдық сублиматорлар

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ТҮЙІНДЕМЕ Дисперсті материалдарды олардан Ұшпа құрауыштарды сублимациялау арқылы өңдеу үшін қазіргі уақытта әзірленген вакуумдық электр пештерінде жүзеге асырылатын конструкциялар мен технологиялық процестерді талдау және технологиялық процестермен байланысты проблемалар негізінде ұсынылған жұмыста концентраттың орын ауыстыруы жылытқыштан орын ауыстырылатын және тасымалданатын жер бетіне тікелей сәулеленумен реологиялық қасиеттер есебінен жүзеге асырылатын техникалық шешімдер ұсынылған аралас шикізат. Жабдықты әзірлеу кезінде тұжырымдама қабылданды, онда Мақала келді: 27 мамыр 2022 пештің сублимациялық аймағында материалдың қозғалысы реологиялық қасиеттерге Сараптамадан өтті: 03 маусым 2022 байланысты қамтамасыз етіледі, бұл сульфидті атмосфераға қатысты инертті Қабылданды: 27 шілде 2022 материалдарды қолдануға және дисперсті материалдың ашық жерлеріне сәуле арқылы жылу беруді жүзеге асыруға мүмкіндік береді. Нежданинск және Бақыршық кен орындарының түйіршіктелген концентраттарынан мышьяк сульфидті қосылыстарын сублимациялау бойынша технологиялық сынақтар сублимациялық процестердің осындай құрылымдық дизайнының болашағын растады. Әзірленіп жатқан жабдықты практикада қолдану қоршаған ортаға ең аз әсер ете отырып, барлық экологиялық талаптарды сақтай отырып, өндірістің техникалық-экономикалық тиімділігін қамтамасыз етеді, бұл қазіргі уақытта қандай да бір технологияны таңдау кезінде негізгі көрсеткіш болып табылады. *Түйін сөздер:* Реология, дисперсті материал, электр пеші, қыздыру, деарсенация. Авторлар туралы ақпарат: Техника ғылымдарының кандидаты, қауымдастырылған профессор (доцент), "металлургия және кен байыту институты" АҚ Вакуумдық процестер зертханасының Требухов Сергей Анатольевич жетекші ғылыми қызметкері, Сәтбаев атындағы университет, 050010, Шевченко к-сі, 29/133, Алматы, Қазақстан. Email: s.trebukhov@satbayev.university; ORCID ID: https:// orcid.org/0000-0001-9708-0307 Техника ғылымдарының докторы, профессор, "металлургия және кен байыту институты" АҚ Вакуумдық процестер зертханасының бас ғылыми қызметкері, Володин Валерий Николаевич Сәтбаев атындағы университет, 050010, Шевченко к-сі, 29/133, Алматы, Қазақстан. Email: volodinv_n@mail.ru; ORCID ID: https://orcid.org/0000-0003-0116-1423 Техника ғылымдарының кандидаты, "металлургия және кен байыту институты" АҚ Вакуумдық процестер зертханасының меңгерушісі, Сәтбаев атындағы университет, Ниценко Алина Владимировна 050010, Шевченко к-сі, 29/133, Алматы, Қазақстан. Email: alina.nitsenko@gmail.com; ORCID ID: https://orcid.org/0000-0001-6753-0936

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	RN µАТОННА
Поступила: <i>27 мая 2022</i> Рецензирование: <i>03 июня 2022</i> Принята в печать: <i>27 июля 2022</i>	На основании анализа конструкций и технологических процессов, осуществляемых в разработанных к настоящему времени вакуумных электропечах для переработки дисперсных материалов сублимацией из них летучих составляющих, и проблем, связанных с технологическими процессами, в представленной работе предложены технические решения, в которых перемещение концентрата осуществляется за счет реологических свойств с прямым нагревом излучением от нагревателя к поверхности перемещаемого и перемешивающегося сырья. При разработке оборудования принята концепция, в которой перемещение материала в сублимационной зоне печи обеспечивается за счет реологических свойств, что открывает возможность использовать инертные по отношению к сульфидной атмосфере материалы и реализовать передачу тепла излучением на открытые участки дисперсного материала. Технологические испытания по сублимации сульфидных соединений мышьяка из гранулированных концентратов Нежданинского и Бакырчикского месторождений подтвердили перспективность подобного конструктивного оформления сублимационных процессов. Применение разрабатываемого оборудования на практике обеспечит технико-экономическую эффективность производства с соблюдением всех экологических требований с минимальным воздействием на окружающую среду, что в настоящее время является основополагающим показателем при выборе той или иной технологии.
	<i>Ключевые слова:</i> Реология, дисперсный материал, электропечь, нагрев, деарсенация.
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