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ИЗВЕСТИЯ

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РЕСПУБЛИКИ КАЗАХСТАН

АО «ИНСТИТУТ ТОПЛИВА, КАТАЛИЗА И
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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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ELECTRON MICROSCOPY SURFACE STUDY OF CATALYSTS BASED ON FERROALLOY PRODUCTION WASTE

Abstract: In the paper results of electron microscopy study of catalysts based on ferroalloy production waste from ash-slime storage of Aksu ferroalloy plant (Aksu, Kazakhstan). The surface morphology of catalyst granules as well pattern of surface distribution of crystallites of catalytically active metals (Fe, Cr and Mn) were described. It was shown, that during the process of catalyst obtaining, uniform porous surfaces without visible large agglomerates of metal crystallites were obtained. Distribution of metallic components was equable, particles were fine disperse, sizes of the crystallites were of one order.

Keywords: catalyst, waste, ferroalloy production, electron microscopy, surface.

Introduction

In result of activity of a ferroalloy plant various types of waste form. The main of them are slime and dust from gas cleaning equipment [1–3]. The waste is accumulated in slime storages. Usually the waste are dumped under the layer of water in artificial ponds. These materials are very disperse and unusable for remelting of ferroalloy, that is, they unusable for direct use in metallurgy [4]. However, earlier, it was shown that their elemental and phase content as well as surface structure make it possible to use them as catalysts for various chemical processes, in particular, in the processes of oil chemistry and oil refining [5].

Earlier we studied waste dumped in ash-slime storage of Aksu ferroalloy plant (Aksu, Kazakhstan) [6–8]. We investigated elemental content, phase content, structure of the surface with ammonia thermoprogrammed desorption and electron microscopy. A series of granulated catalyst was prepared based on obtained waste. In the paper, we are presenting electron micrographs of obtained catalyst samples. The aim of the study is to reveal morphological peculiar surface properties of catalyst samples prepared from samples of waste picked up from different locations of the ash-slime storage.

Methods

Catalyst preparation

For catalyst preparation ferroalloy production waste samples were picked up from the territory of ash-slime storage of Aksu ferroalloy plant according to GOST «17.4.3.01-83 Soils. General Sampling Requirements». Totally 80 samples from 16 locations were taken. From every location we took 5 samples using the method of «envelope» and well mixed them for obtaining of joint sample. So, we have obtained

16 joint samples. Each sample was assigned a serial number. Catalysts were named using serial number of joint sample used for preparing of it. In the research we used according names of catalysts Kt-1, Kt-2 ... Kt-16. For preparing of catalysts obtained ash-slime mass was washed by distilled water in order to separate small organic particles. Then the washed mass was dried on airtight petridishes to obtain a pasty mass. Laboratory extruder molded mass to obtain cylindrical granules with diameter of 3-4 mm and length 10-15 mm. After that granules were dried at 100-150 °C for 5 hours at the rate of temperature rise 25-30 °C per hour, then it was calcinated at 200 °C for 1 hour, 300 °C - 1 hour, 400 °C - 1 hour, 500 °C - 5 hours.

Electron microscopy

For studying of structure and surface of researched materials scanning electron microscope with thermionic cathode (LaB6) JSM-6610LV ("JOEL", Japan) was used. The device was kitted with the system of energy dispersive microanalysis, wave dispersion microanalysis system, backscattered electron diffraction analysis system using a reflected electron detector, Everhart-Thornley Secondary Electron Detector, secondary electron detector for low vacuum mode and equipment for sample preparation. The research was carried out at x1000 and x3000 magnifications.

Results and discussion

The broadness of the application of the method of electron microscopy is associated with its high informativity and versatility, as well as the simplicity and convenience of equipment managing [9]. Scanning electron microscopy has several advantages over other methods. For example, compared with traditional light microscopy, it is characterized by a significantly higher resolution and depth of sharpness; relative ease of interpretation of the images due to their three-dimensional representation; the ability to connect additional devices for analysis in the micro-range with sufficient ease of adaptation and control of these devices [10]. It is also necessary to note the relatively low requirements for sample preparation. Compared with scanning probe microscopy scanning electron microscopy allows you to explore significantly large areas of the surface; work with highly relief surfaces; use a much wider range of magnifications; obtain information not only about the surface, but also about the adjacent to the surface "subsurface" layers [11, 12].

We have obtained micrographs of cross sections and side surfaces of granules of catalysts Ct-2, Ct-3, Ct-4, Ct-5, Ct-15 at magnifications $\times 1000$ and $\times 3000$ (Fig. 1-20).

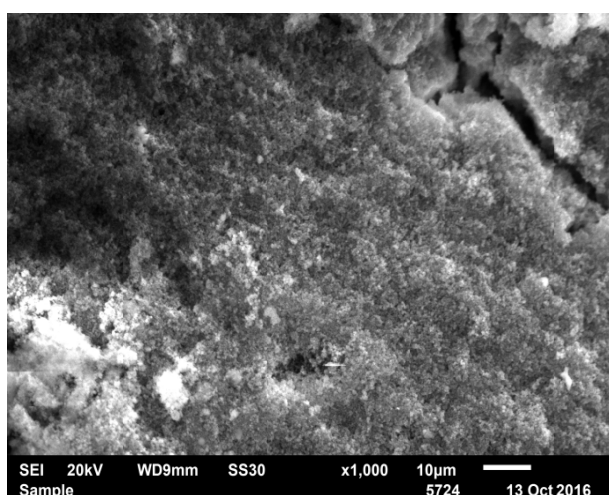


Figure 1 – Micrograph of cross section of catalyst Ct-2 granule ($\times 1000$)

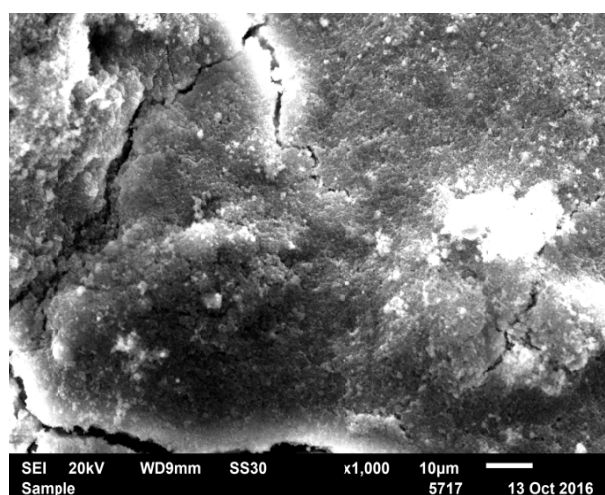


Figure 2 - Micrograph of side surface of catalyst Ct-2 granule ($\times 1000$)

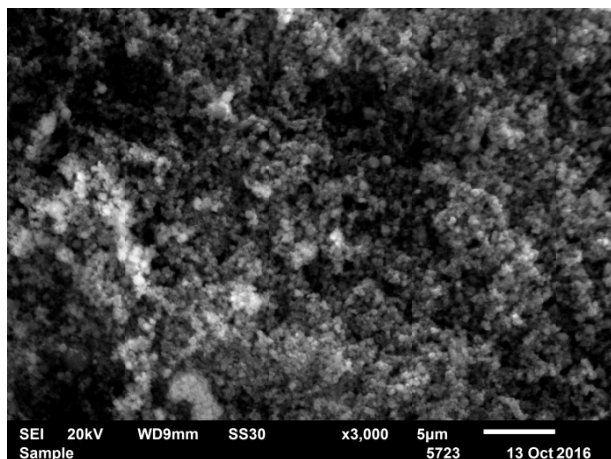


Figure3 –Micrographof cross section of catalyst Ct-2 granule($\times 3000$)

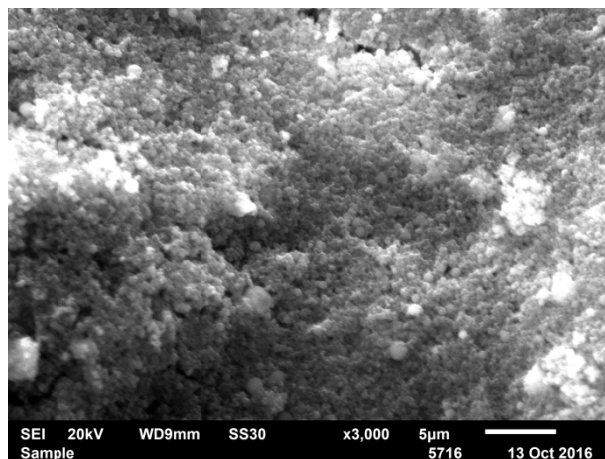


Figure4 - Micrographofside surface of catalyst Ct-2granule($\times 3000$)

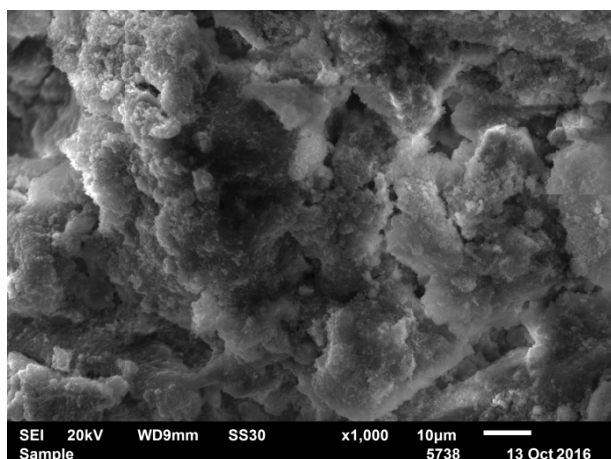


Figure5 –Micrographof cross section of catalyst Ct-3 granule($\times 1000$)

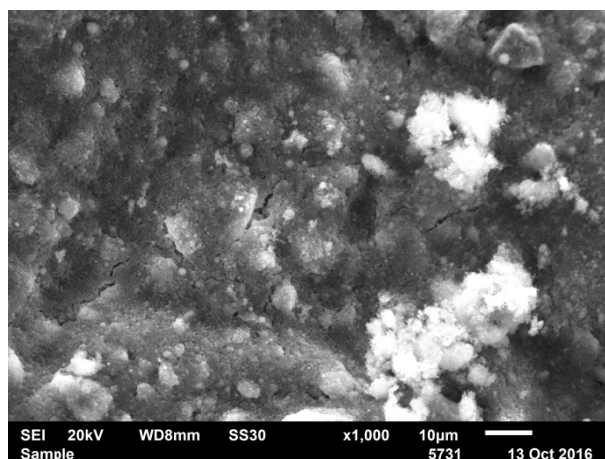


Figure6 - Micrographofside surface of catalyst Ct-3granule($\times 1000$)

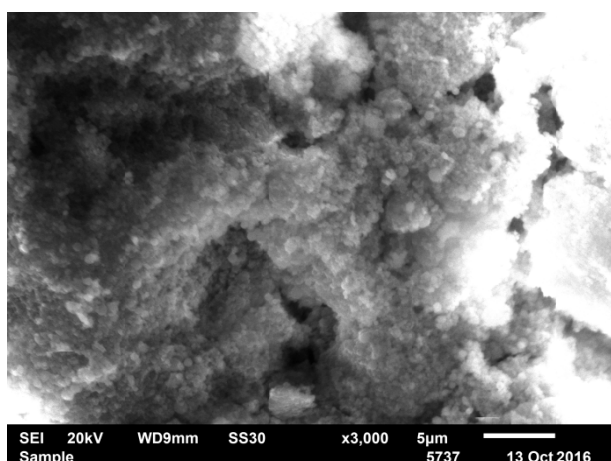


Figure7 –Micrographofcross section of catalyst Ct-3 granule($\times 3000$)

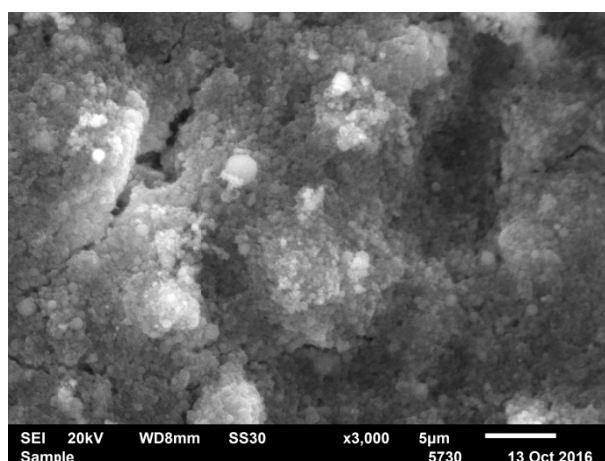


Figure8 - Micrographofside surface of catalyst Ct-3 granule($\times 3000$)

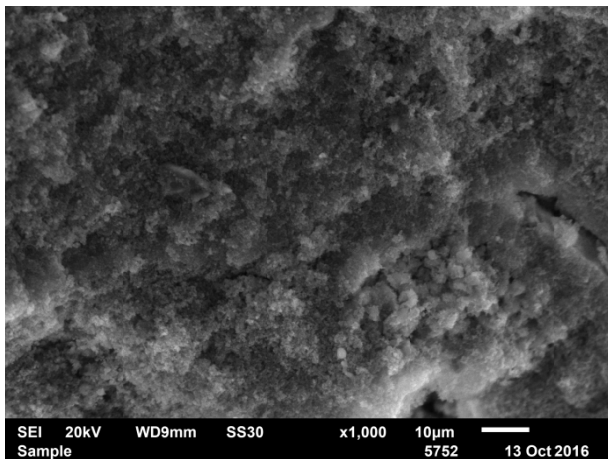


Figure9 - Micrograph of cross section of catalyst Ct-4 granule (×1000)

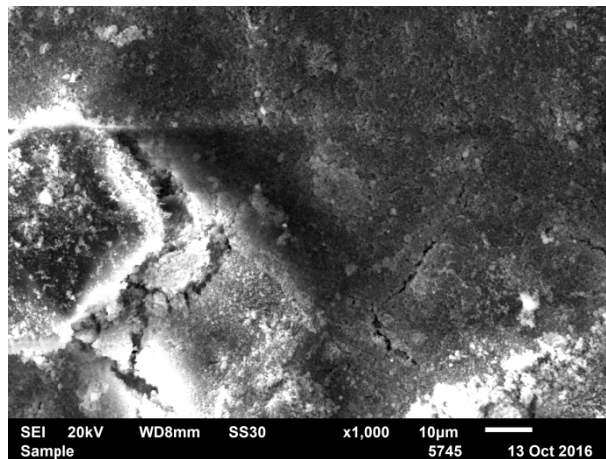


Figure10 - Micrograph of side surface of catalyst Ct-4 granule (×1000)

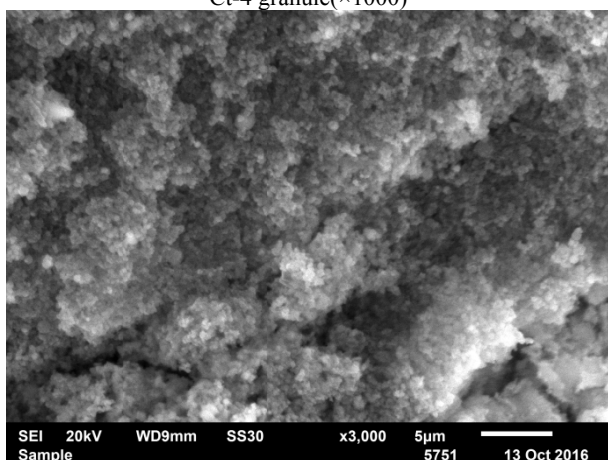


Figure11 - Micrograph of cross section of catalyst Ct-4 granule (×3000)

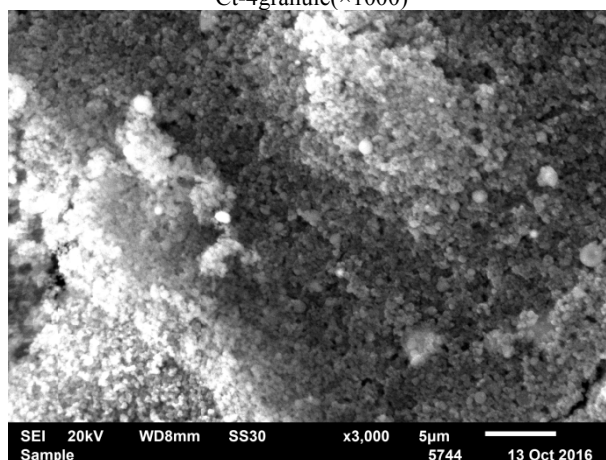


Figure12 - Micrograph of side surface of catalyst Ct-4 granule (×3000)

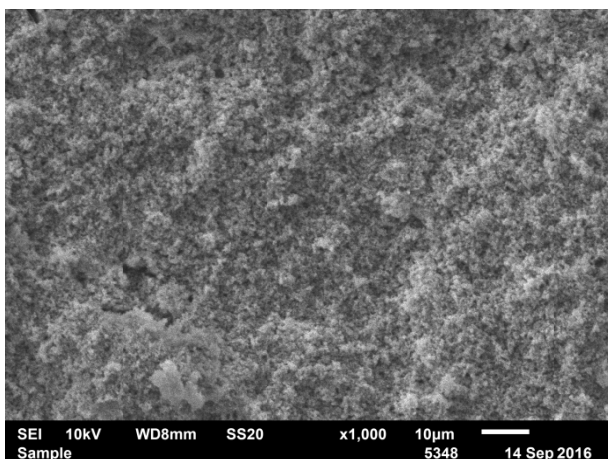


Figure13 - Micrograph of cross section of catalyst Ct-5 granule (×1000)

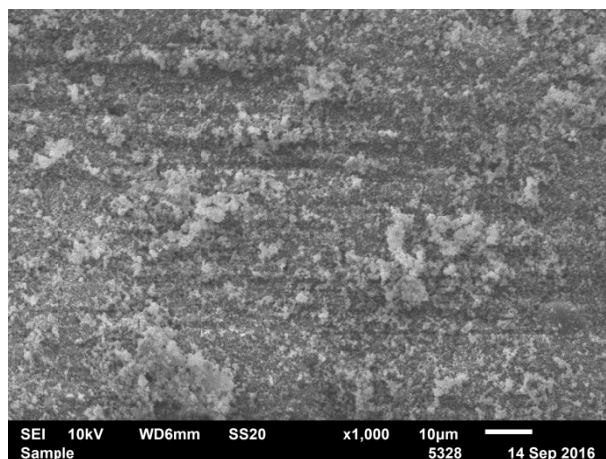


Figure14 - Micrograph of side surface of catalyst Ct-5 granule (×1000)

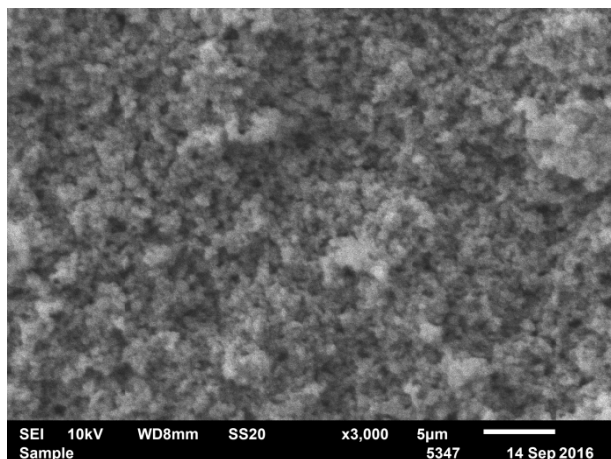


Figure15 –Micrograph of cross section of catalyst Ct-5 granule($\times 3000$)

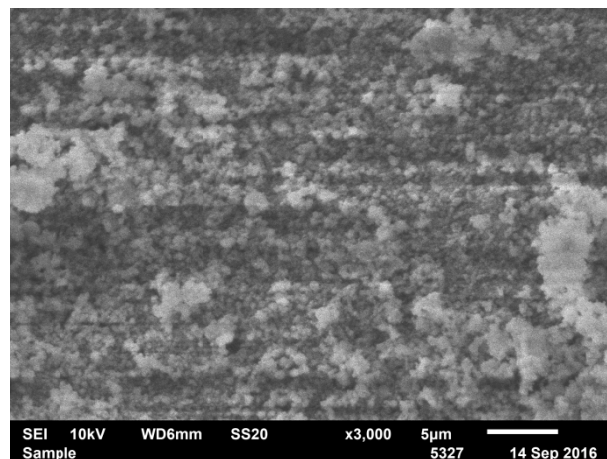


Figure16 - Micrograph of side surface of catalyst Ct-5 granule($\times 3000$)

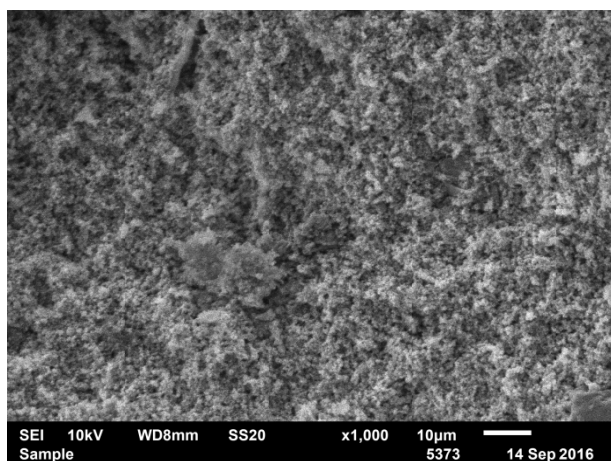


Figure17 –Micrograph of cross section of catalyst Ct-15 granule($\times 1000$)

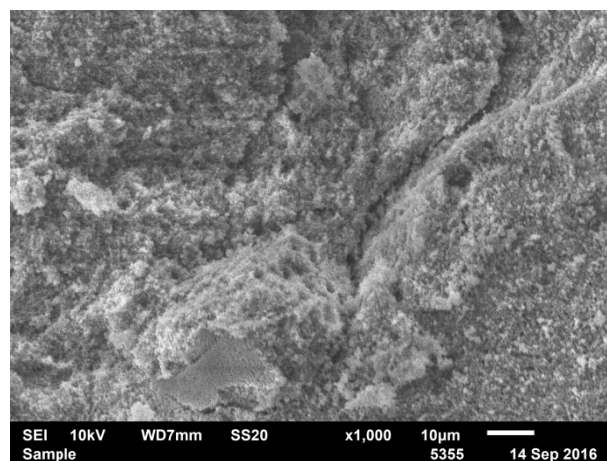


Figure18 - Micrograph of side surface of catalyst Ct-15 granule($\times 1000$)

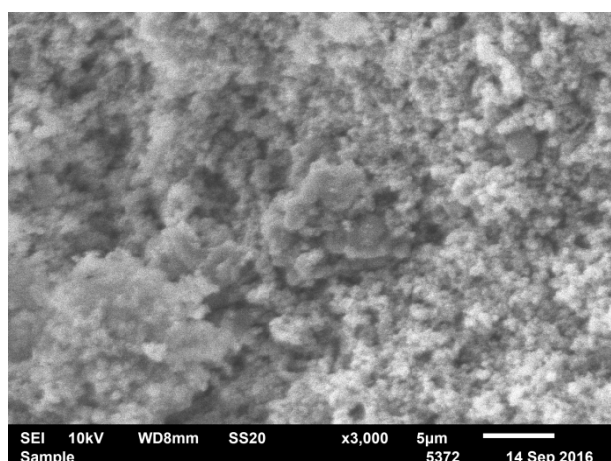


Figure19 –Micrograph of cross section of catalyst Ct-15 granule($\times 3000$)

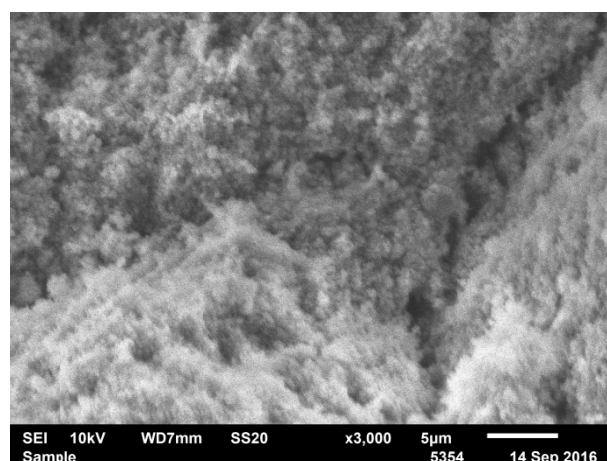


Figure20 –Micrograph of side surface of catalyst Ct-15 granule($\times 3000$)

Analysis of electronic micrographs allows us to establish that at magnification for 1000 times the lateral surfaces of the granules is more uniform than the cross-section surfaces. That is obvious due to the fact that formation of granules was performed with laboratory extruder, which forms more or less smooth surface of granules. The same time at magnification for 3000 times the difference in cross section surface and side surface morphology practically unnoticeable. The surface is finely porous, represented by fine

granular particles. Porous surface can be formed during the process of high temperature treatment as a result of elimination of volatile components.

In figures 21-23 micrographs of side surfaces of catalyst Ct-15 granules in the mode of frequencies filtration are represented. That allows to reveal distribution of crystallites of metallic components on surface. On presented micrographs it is visible, that metal components distribution is uniform, particles are finely dispersed, sizes of crystallites are of one order.

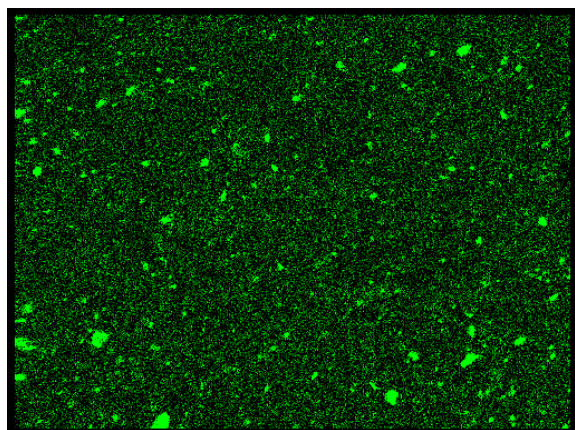


Figure 21 - Micrograph of side surface of catalyst Ct-15 granule with representation of Cr crystallites distribution

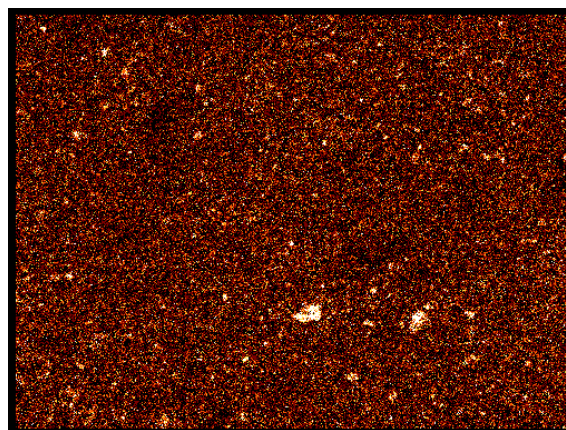


Figure 22 - Micrograph of side surface of catalyst Ct-15 granule with representation of Mn crystallites distribution

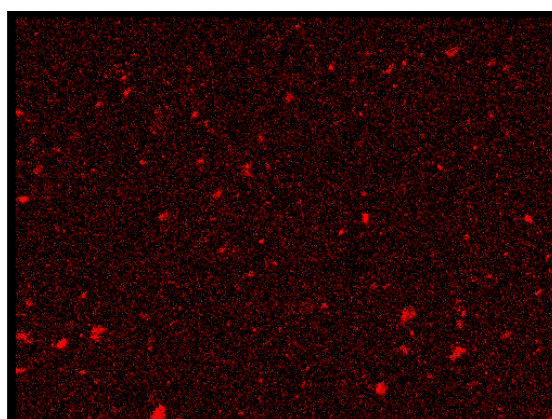


Figure 23 - Micrograph of side surface of catalyst Ct-15 granule with representation of Fe crystallites distribution

Conclusion

Thus, as a result of catalyst preparation, uniform, porous surface without visible large agglomerates of metal (or of metal compounds) crystallites was obtained. Taking into account characteristic content of ferroalloy production waste with increased concentration of catalytically active elements – Fe, Cr, Mn, specific for heterogeneous catalysts surface morphology [13,14], trend of distribution and degree of dispersity of metal crystallites on catalyst surface [15], it can be concluded that obtained materials can be used as heterogeneous nanosized catalysts for various processes of hydrocarbon-containing raw materials refining.

Important conclusion of electron microanalysis is the fact that morphology of surfaces of catalysts Ct-2, Ct-3, Ct-4, Ct-5, Ct-15 is practically the same. This fact allows indirectly confirm possibility of using of ash-slime storage as secondary source for mining of raw material for obtaining of catalyst. The same surface morphology of waste taken from different locations of ash-slime storage is the consequence of the fact that the waste was accumulated during many years of performing of regular processes of ferroalloys melting with strict adherence to regulations. In the other words, conditions of these wastes are constant and are controlled during the melting process. The spread of waste over the area of the ash-

slimestorage lake occurs as a result of hydrodynamic processes of mass transfer, diffusion. Waste material does not mix with ground, the presence of flora on the perimeter of the lake storage is minimal.

Based on the data obtained, it can be concluded that the catalysts obtained are promising materials and can be used in various processes of processing of hydrocarbon-containing raw materials, in particular, in the processes of cracking, hydrogenation, oxidation.

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ФЕРРО ҚОРЫТПАНЫ ӨНДЕУ ҚАЛДЫҚТАРЫ НЕГІЗІНДЕ АЛЫНҒАН КАТАЛИЗАТОРЛАР БЕТІН ЭЛЕКТРОНДЫҚ МИКРОСКОПИЯ ӘДІСІМЕН ЗЕРТТЕУ

Аннотация. Мақалада Ақсу ферроқорытпа зауытының күлшлам қалдықтарынан ферроқорытпа өндірісі қалдықтары негізінде алынған катализаторлардың электронды - микроскопиялық зерттеулерінің нәтижелері келтірілген. Катализатор түйіршіктер бетінің морфологиясы мен каталитикалық белсенді Fe, Сг және Mn

металдар кристаллиттерінің беткі таралуы сипатталған. Катализаторды дайындау барысында металл кристаллиттерінің ірі агломераттарының көрінбейтін біркелкі, кеуекті беті алынғаны, металл компоненттері құрамдас бөліктерінің біркелкі болуы, кристаллиттердің өлшемі бірдей екендігі, бөлшектердің ұсақ дисперстілігі көрсетілді.

Түйін сөздер: катализаторлар, қалдықтар, феррокорытпаны өңдеу, электронды микроскопия, бет.

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ИССЛЕДОВАНИЕ МЕТОДОМ ЭЛЕКТРОННОЙ МИКРОСКОПИИ ПОВЕРХНОСТИ КАТАЛИЗАТОРОВ, ПОЛУЧЕННЫХ НА ОСНОВЕ ОТХОДОВ ФЕРРОСПЛАВНОГО ПРОИЗВОДСТВА

Аннотация. В статье приведены результаты исследования методом электронной микроскопии катализаторов, полученных на основе отходов ферросплавного производства с золошламонакопителя Аксуского ферросплавного завода. Описана морфология поверхности гранул катализатора, а также характер поверхностного распределения кристаллитов каталитически активных металлов Fe, Cr и Mn. Показано, что в ходе приготовления катализатора, получена равномерная, пористая поверхность без видимых крупных агломератов кристаллитов металлов, распределение металлических компонентов является равномерным, частицы мелкодисперсные, размеры кристаллитов одного порядка.

Ключевые слова: катализаторы, отходы, производство ферросплавов, электронная микроскопия, поверхность.

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