ELECTRON MICROSCOPY SURFACE STUDY OF CATALYSTS BASED ON FERROALLOY PRODUCTION WASTE

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АО «ИНСТИТУТ ТОПЛИВА, КАТАЛИЗА И
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каб. 310, тел. 291-62-80, факс 291-57-22, e-mail:orgcat@nursat.kz
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Zh.K. Shomanova[1], R.Z. Safarov[2], A.S. Zhumakanova[3], Yu.G. Nosenko[4], A.T. Zhanibekova[1], N.L. Shapekova[2], D. Lorant[5]

[1]Pavlodar State Pedagogical Institute, Pavlodar, Kazakhstan
[2]L.N. Gumilyov Eurasian National University, Astana, Kazakhstan
[3]D.V.Sokolsky Institute of Fuel, Catalysis and Electrochemistry, Almaty, Kazakhstan
[4]Innovative University of Eurasia, Pavlodar, Kazakhstan
[5]Eötvös Loránd University, Budapest-Szombathely, Hungary
e-mail: ruslanbox@yandex.ru

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). Thesurface 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]. Thewasteisaccumulatedinslimestorages. 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, itwasshownthattheirelementalandphasecontentaswellassurfacestructuremakeitpossibletousethemascatalysts 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 thermoprogrammeddesorption 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 airin petridishesto 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.

Electronmicroscopy

Forstudyingof 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.

Resultsanddiscussion

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 ×1000 and ×3000 (Fig. 1-20).

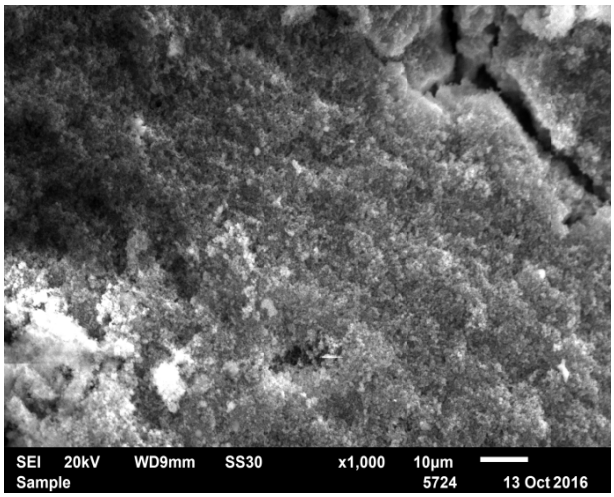

Figure 1 –Micrographof cross section of catalyst Ct-2 granule (×1000)

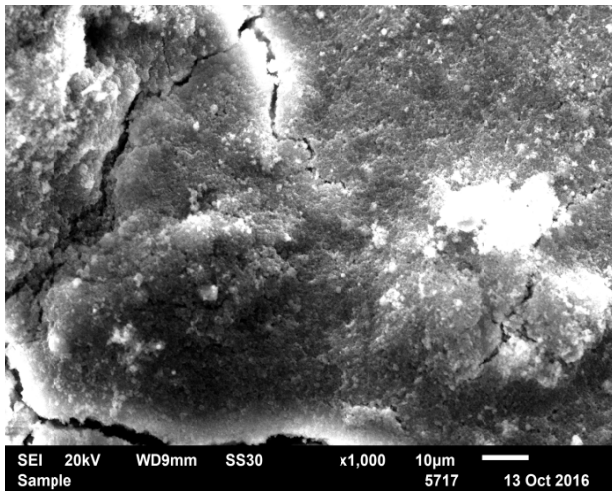

Figure2 - Micrographofside surface of catalyst Ct-2granule(×1000)

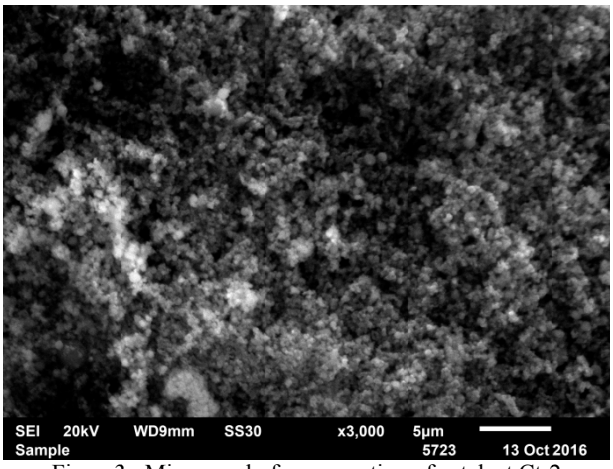

Figure3 –Micrographof cross section of catalyst Ct-2 granule(×3000)

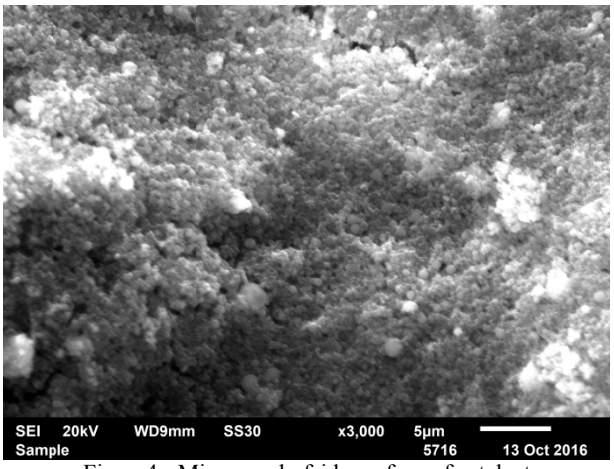

Figure4 - Micrographofside surface of catalyst Ct-2granule(×3000)

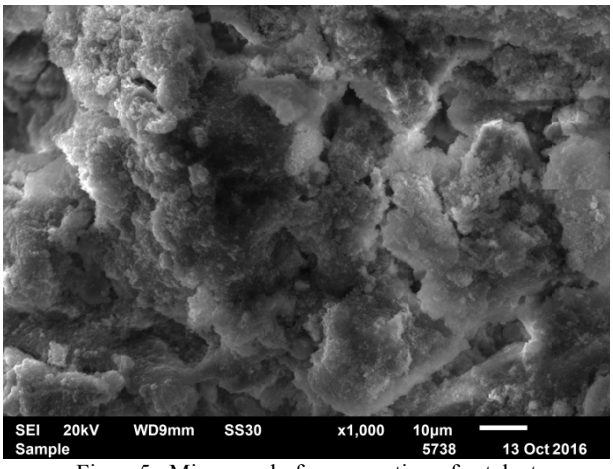

Figure5 –Micrographof cross section of catalyst Ct-3 granule(×1000)

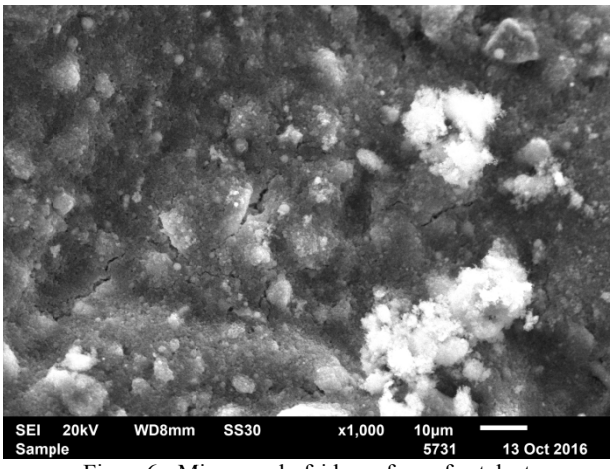

Figure6 - Micrographofside surface of catalyst Ct-3granule(×1000)

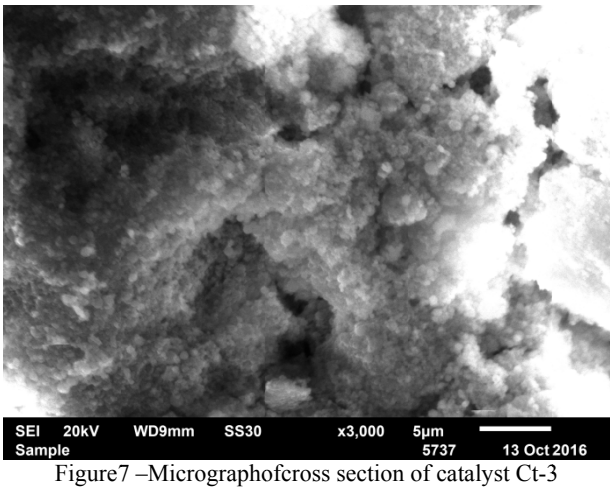

Figure7 –Micrographofcross section of catalyst Ct-3 granule(×3000)

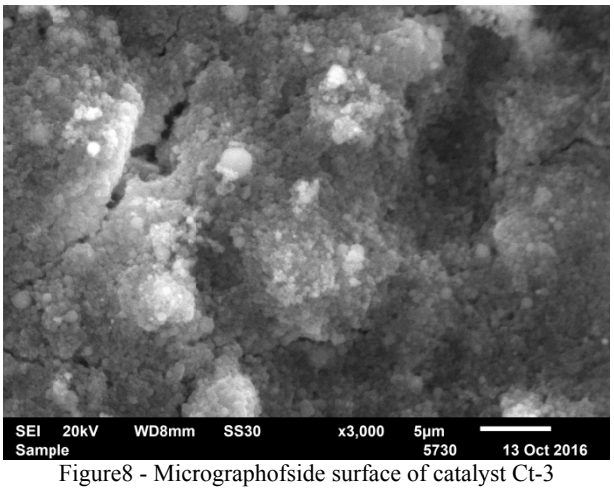

Figure8 - Micrographofside surface of catalyst Ct-3 granule(×3000)

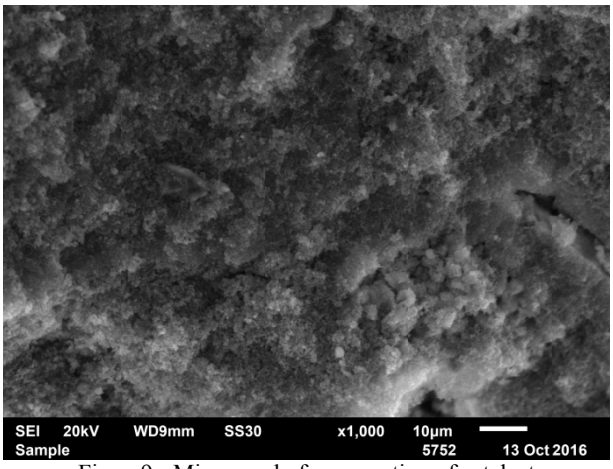

Figure9 - Micrographofcross section of catalyst Ct-4 granule(×1000)

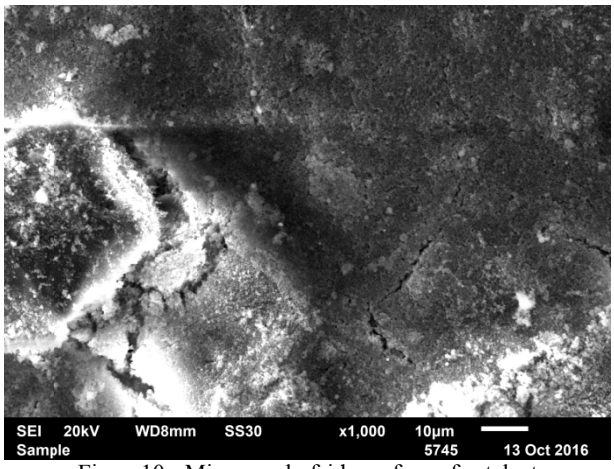

Figure10 - Micrographofside surface of catalyst Ct-4granule(×1000)

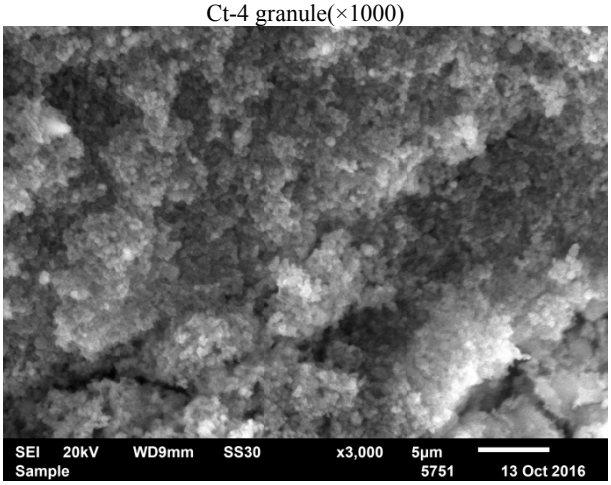

Figure11 - Micrograph of cross section of catalyst Ct-4 granule (×3000)

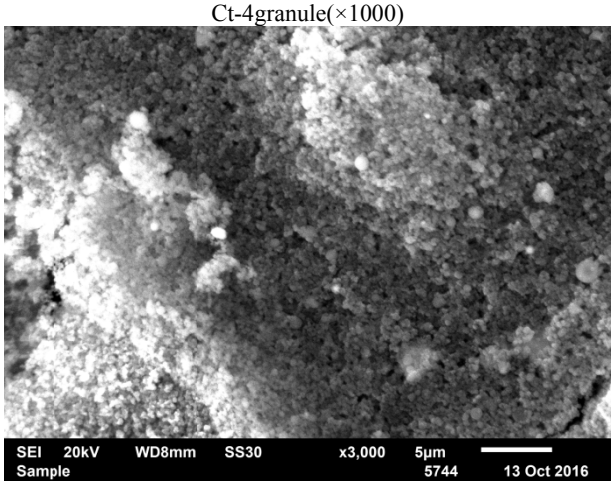

Figure12 –Micrographofside surface of catalyst Ct-4 granule(×3000)

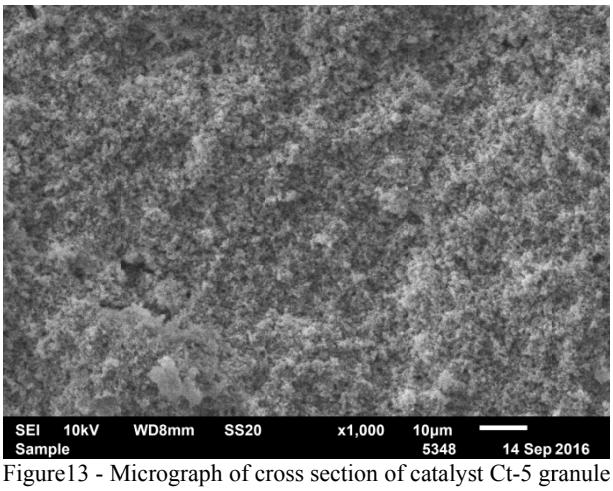

Figure13 - Micrograph of cross section of catalyst Ct-5 granule (×1000)

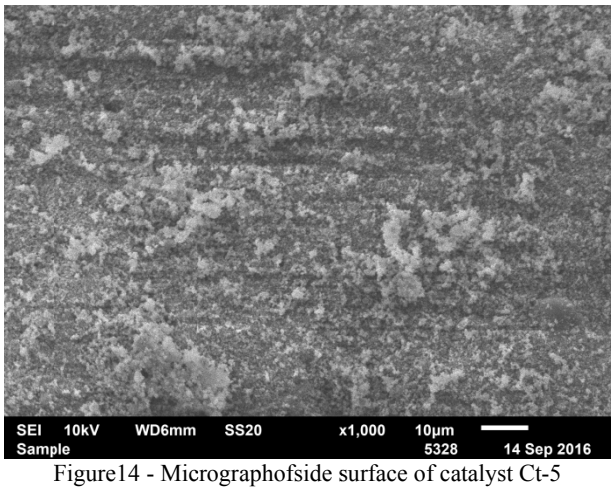

Figure14 - Micrographofside surface of catalyst Ct-5 granule(×1000)

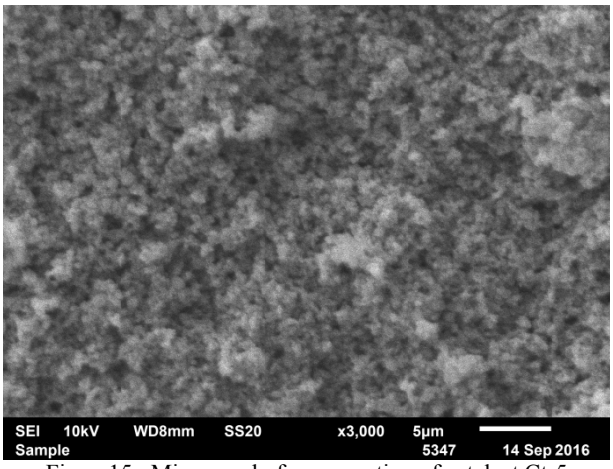

Figure15 –Micrographof cross section of catalyst Ct-5 granule(×3000)

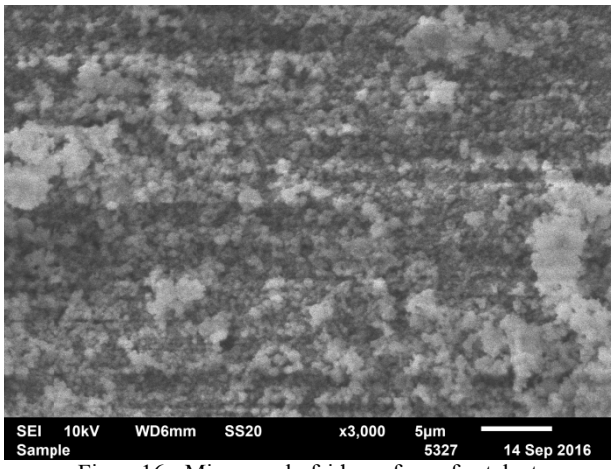

Figure16 - Micrographofside surface of catalyst Ct-5granule(×3000)

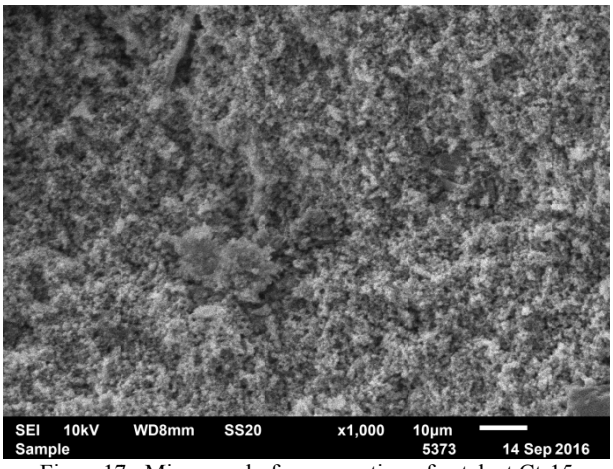

Figure17 –Micrographof cross section of catalyst Ct-15 granule(×1000)

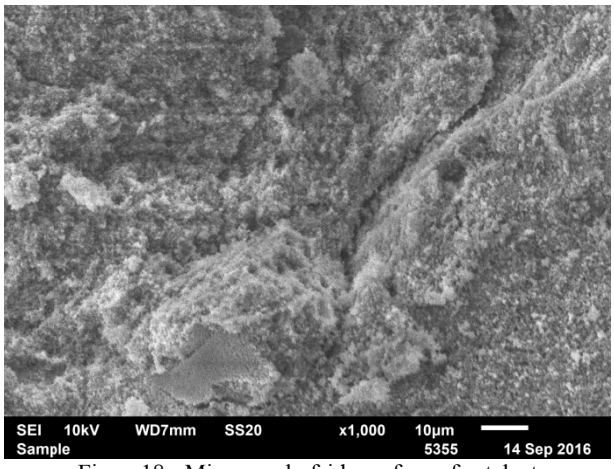

Figure18 - Micrographofside surface of catalyst Ct-15granule(×1000)

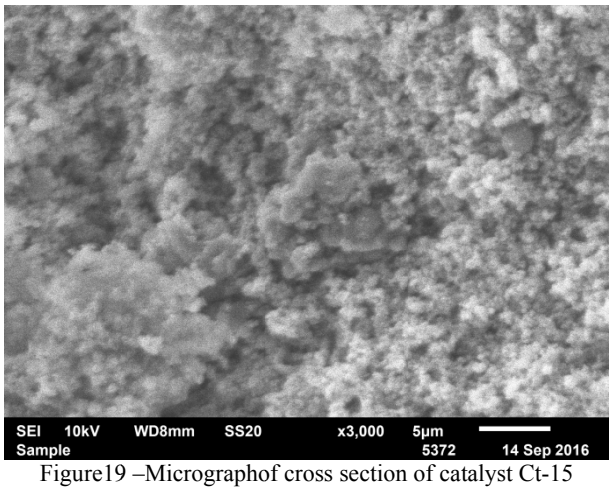

Figure19 –Micrographof cross section of catalyst Ct-15 granule(×3000)

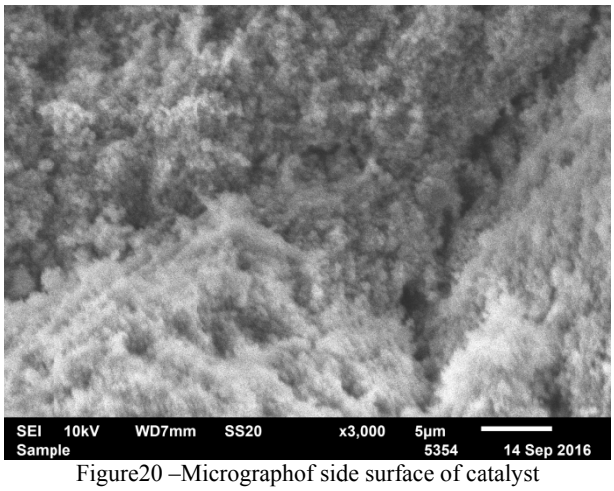

Figure20 –Micrographof side surface of catalyst Ct-15granule(×3000)

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 obviousdue to the fact thatformation of granules was performed with laboratory extruder, which forms more or less smooth surface of granules. Thesametimeatmagnificationfor 3000 timesthedifference in cross section surface and side surface morphology practically unnoticeable. The surface is finely porous, represented by fine

granular particles. Poroussurfacecanbeformedduring 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 revealdistribution of crystallites of metallic components on surface. Onpresentedmicrographsitisvisible, that metal components distribution is uniform, particles are finedispersed, sizes of crystallites are of one order.

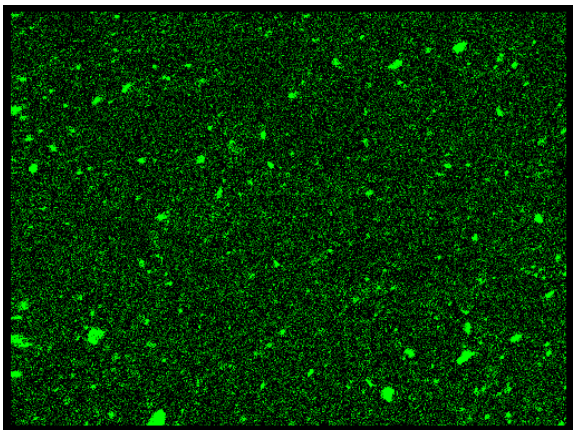

Figure21 - Micrographofside surface of catalyst Ct-15granulewith representation of Cr crystallites distribution

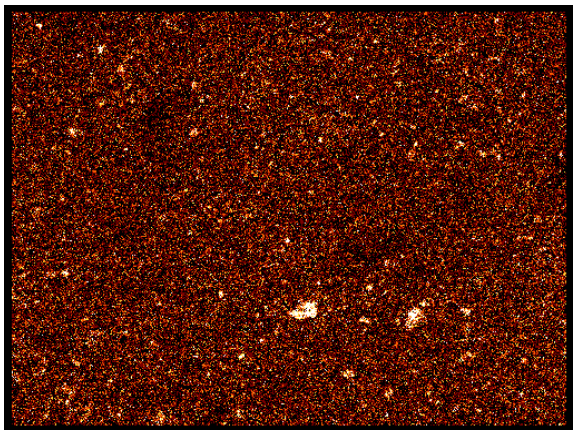

Figure22 - Micrographofside surface of catalyst Ct-15granulewith representation of Mn crystallites distribution

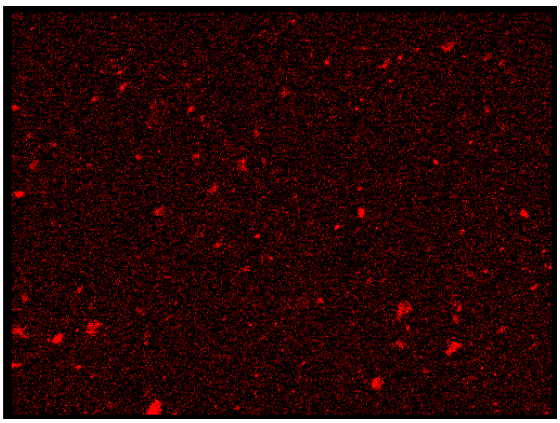

Figure23 - Micrographofside surface of catalyst Ct-15granulewith representation of Fe crystallites distribution

Conclusion

Thus, asaresult of catalyst preparation, uniform, porous surface without visible large agglomerates of metal (or of metal compounds) crystallites was obtained. Takingintoaccountcharacteristiccontentofferroalloy production waste with increased concentration of catalytically active elements – Fe, Cr, Mn, specific for heterogeneous catalysts surface morphology [13,14], trendofdistributionanddegreeofdispersityofmetalcrystallitesoncatalystsurface[15], itcanbeconcludedthatobtainedmaterialscanbeusedasheterogeneous 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 confirmpossibilityof using of ash-slime storage as secondary source for mining of raw material for obtaining of catalyst. Thesamesurfacemorphologyofwastetakenfromdifferent 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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Ж.К. Шоманова[1], Р.З. Сафаров[2], А.С. Жумаканова[3], Ю.Г. Носенко[4], А.Т. Жанибекова[1], Н.Л. Шапекова[2], Д. Лорант[5]

[1]Павлодар мемлекеттік педагогикалық университеті, Павлодар, Қазақстан;
[2]Л.Н. Гумилев атындағы Еуразиялық ұлттық университеті, Астана, Қазақстан;
[3]Д.В. Сокольский атындағы жанармай, катализ және электрохимия институты, Алматы, Қазақстан;
[4]Инновациялық еуразия университеті, Павлодар,Қазақстан;
[5]EötvösLorándUniversity, Будапешт - Сомбатхей, Венгрия

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, Cr 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, кристалиттердің өлшемі бірдей екендігі, бөлшектердің ұсақ дисперстілігі көрсетілді.

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

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Ж.К. Шоманова[1], Р.З. Сафаров[2], А.С. Жумаканова[3], Ю.Г. Носенко[4], А.Т. Жанибекова[1], Н.Л. Шапекова[2], Д. Лорант[5]

[1]Павлодарский государственный педагогический университет, Павлодар,Казахстан;
[2]Евразийский национальный университет им. Л.Н. Гумилева, Астана, Казахстан;
[3]Институттоплива, катализаиэлектрохимии им. Д.В. Сокольского, Алматы, Казахстан;
[4]Инновационный евразийский университет, Павлодар,Казахстан;
[5]EötvösLorándUniversity, Будапешт - Сомбатхей, Венгрия

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.

Information about authors:

Zhanat Kairollinovna Shomanova – doctoroftechnicalsciences, professor of the Geography and chemistry department, Pavlodar state pedagogical institute, zshoman@yandex.ru, https://orcid.org/0000-0001-8346-9688

RuslanZairovichSafarov – candidateofchemicalsciences, the Acting Associate Professor of the Department of Chemistry, vice-dean in science at Natural Sciences Faculty, L.N. Gumilyov Eurasian national university, ruslanbox@yandex.ru, https://orcid.org/0000-0003-2158-6330

Ardak Sydykovna Zhumakanova – candidate of chemical sciences, scientist secretary, D.V. Sokolsky Institute of Fuel, Catalysis and Electrochemistry, a.jumakanova@ifce.kz, https://orcid.org/0000-0003-4983-4199

Yuri Gennadievich Nosenko – Candidate of Chemical Sciences, acting Associate Professor of the Department of Chemical and Biological Technologies, Innovative University of Eurasia, nosenko1980@yandex.ru, https://orcid.org/0000-0002-2491-7337

Aisulu Talgatovna Zhanibekova – bachelor of environmental science, zshoman@yandex.ru, https://orcid.org/0000-0002-5739-6267

Nelya Lukpanovna Shapekova – doctor of medical sciences, professor of the Department of Biotechnology and microbiology, Dean of Natural Sciences Faculty, L.N. Gumilyov Eurasian national university, shapekova_nl@enu.kz, https://orcid.org/0000-0003-2534-7951

Dr. habil. habil. habil. Dávid Lóránt Dénes – full-professor, Jean Monnet Professor, PhD, EötvösLorándUniversity, davidlo@ektf.hu, https://orcid.org/0000-0001-7880-9860

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