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Investigation of the phase composition and structure of stationary catalysts to increase safety of agrarian-and-food production

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Abstract. The phase composition and structure of stationary nickel-aluminum promoted catalysts have been studied. It is shown that the phase composition of the catalysts consists of intermetallic compounds NiAl, NiAl3, Ni2Al3. Changes in the microscopic structure of the alloys are determined. Modern methods for evaluating hydrogenation catalysts, as well as methods for analyzing raw materials and products of catalytic hydrogenation, have been established. The highest hydrogenation performance is characteristic of stationary catalysts whose alloys consist of Ni2Al3×4NiAl3 intermetallic compounds. New modifications of stationary alloyed nickel-copper-aluminum catalysts with the addition of vanadium (0.5-2.5 %), rhodium (0.5 %) and palladium (0.05 %) in the process of pre-contact hydrogenation of cottonseed oil have been studied and developed. It has been shown that the lowest content of transisomerized acids in tallow is achieved at a content of 1.5 % vanadium in a stationary promoted catalyst. The content ratio of the fatty-acid composition in the triglycerides of target dietary fats was determined, ensuring their increase in nutritional value and safety. The conditions under which the highest results were obtained are presented.

1 Introduction

In the process of catalytic hydrogenation of oils and fats, the hydrogenating properties of catalysts depend on the phase composition [1-3], the content of intermetallic compounds [4-6] and the structure of alloys [7-10]. In this regard, the study of the features of the phase composition and structure of new types of stationary alloy catalysts is of practical scientific interest. The purpose of the study is aimed at studying the features of the phase composition and content of intermetallic compounds in new modifications of stationary alloy promoted nickel-copper catalysts, as well as the microscopic structure of alloys.

2 Research methods

The work uses modern methods of X-ray diffraction analysis of the phase composition of catalysts, microscopic surveys of the active surface of the alloy structure.

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The phase composition of the alloyed catalysts was studied using a URS-50 IM X-ray device operating on copper radiation [11].

The studied areas of radiographs were 20-82°. Some samples of stationary catalysts were subjected to metallographic studies, some were carried out on microscopes MIM-7 and MIM-8 [12].

The phase composition of typical samples of alloy catalysts is shown in Figures 1-6.

The diffraction pattern of nickel-copper-aluminum and nickel-copper-aluminum catalysts is compared in Figures 1 and 3.

The diffractogram of fresh and regenerated nickel-copper-aluminum catalysts promoted with rhodium and germanium is shown in Figures 2 and 4,5,6.

The diffraction peaks and corresponding intensities of the nickel-copper-aluminum catalyst (alloy) with the addition of rhodium are shown in Table 1.

Table 1. Change in diffraction peaks and corresponding intensities of unpromoted and rhodium-promoted catalysts.

Unpromoted catalyst		Promoted catalyst		Phase composition
J/J_0. %	d/n. dA^0	J/J_0. %	d/n. dA^0	
45	4.93	50	4.95	*Ni_2Al_3*
40	3.53	45	3.53	*Ni_2Al_3. $(Al_3Ni_2)5H$*
40	2.87	37	2.86	*NiAl*
100	2.23	100	2.07	*NiAl. Ni_2Al_3. $(Al_3Ni_2)5H$*
60	2.02	65	2.02	*Ni_2Al_3. NiAl. $(Al_3Ni_2)5H$*
100	2.01	100	2.01	*$NiAl_3$*
7	1.87	14	1.87	*Ni_2Al_3. $(Al_3Ni_2)5H$*
70	1.75	75	1.79	*$NiAl_3$*
35	1.60	40	1.60	*NiAl. Ni_2Al_3*
20	1.42	30	1.46	*$NiAl_3$. $(Al_3Ni_2)5H$*
60	1.28	65	1.28	*Ni_2Al_3*
70	1.16	74	1.16	*NiAl. $(Al_3Ni_2)5H$*
60	1.04	65	1.07	*Ni_2Al_3*

Table 1 analysis. indicates that the addition of rhodium to the original alloy did not lead to the appearance of additional lines in the diffraction patterns, indicating the formation of new phases. However, the diffraction patterns change with increasing catalyst leaching after reuse.

Studies of the phase composition of the prepared nickel-aluminum catalyst (Figure 1) showed that this catalyst consists mainly of two metallides - Ni_2Al_3 and NiAl, $NiAl_3$, as well as a small amount of metallic nickel.

3 Research results

With the introduction of relatively small amounts of copper (about 10 %) into the nickel-aluminum alloy, the phase composition of the alloy practically does not change. In the investigated nickel-copper-aluminum catalyst (12.5 % copper), we also did not find new phases (Figure 3).

X-ray diffraction patterns of catalysts promoted with germanium show that they mainly consist of intermetallic combined Ni_2 Al_3; $NiAl_3$, that is, similar to the structure of the previous catalysts (Figure 5). An increase in the amount of germanium in the nickel-copper-aluminum alloy leads to additional formation of the Ni_2 Al_3 phase. Repeated leaching has little effect on the metal compound Ni_2 Al_3 (Figure 6).

The diffraction peaks and their respective intensities of the investigated germanium-promoted nickel-copper-aluminum catalyst depending on the activation repetition are given in Table 2.

Table 2 data. indicate that the intensity of the formation of diffraction peaks on a fresh catalyst is much higher than on a spent catalyst. This characterizes the high degree of selectivity of the process on the reused catalyst.

Table 2. Changes in the phase composition and intensity of the catalyst depending on its leaching.

First leaching				Repeated (second) leaching			
$NiAl$		Ni_2Al_3		$NiAl$		Ni_2Al_3	
dA^0	J/J$_0$	dA^0	J/J$_0$	dA^0	J/J$_0$	dA^0	J/J$_0$
1.17	70	1.05	9	1.17	70	1.04	6
1.43	20	1.16	74	1.43	20	1.05	9
1.88	14	1.48	3	1.88	14	1.16	2
2.01	100	2.01	100	2.01	100	1.28	3
2.87	40	2.02	100	2.87	40	1.48	3
3.54	40	5.59	17	4.19	50	2.01	100
4.94	50	-	-	-	-	2.02	100

The diffraction peaks and corresponding intensities of the nickel–copper–aluminum catalysts with rhodium additives indicate that these catalysts also consist of intermetallic compounds NiAl, Ni_2Al_3, $NiAl_3$ and the active form (Ni_2Al_3) 5Ĥ. However, the diffraction peaks and intensity of the formation of these phases differed somewhat from the diffraction pattern of the unpromoted catalyst.

4 Discussion

With an increase in the degree of leaching of the alloy, the intensity of all characteristic phases of intermetallic compounds decreases significantly, and the decrease in the Ni_2 Al_3 phase occurs somewhat more strongly. In addition, on the diffraction patterns of the nickel-copper-aluminum catalyst with a rhodium content of 0.5 % after the third leaching, line broadening was observed, indicating some decrease in the crystal size. Therefore, it can be assumed that the role of rhodium in this case is that it prevents the growth of crystals of the existing phases in the alloy. This is also indicated by a comparison of micrographs of the surface of nickel-aluminum, promoted nickel-copper-aluminum and nickel-copper-rhodium-aluminum catalysts (Figures 7-9). The finest crystalline structure is characteristic of a rhodium-promoted catalyst.

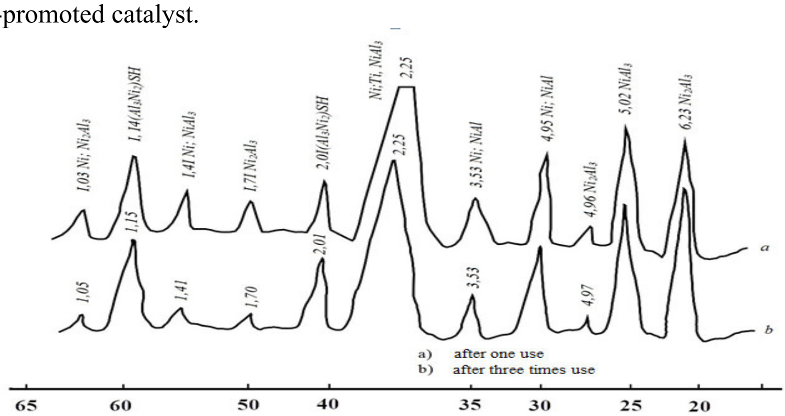

Fig. 1. X-ray diffraction pattern of a nickel-aluminum catalyst.

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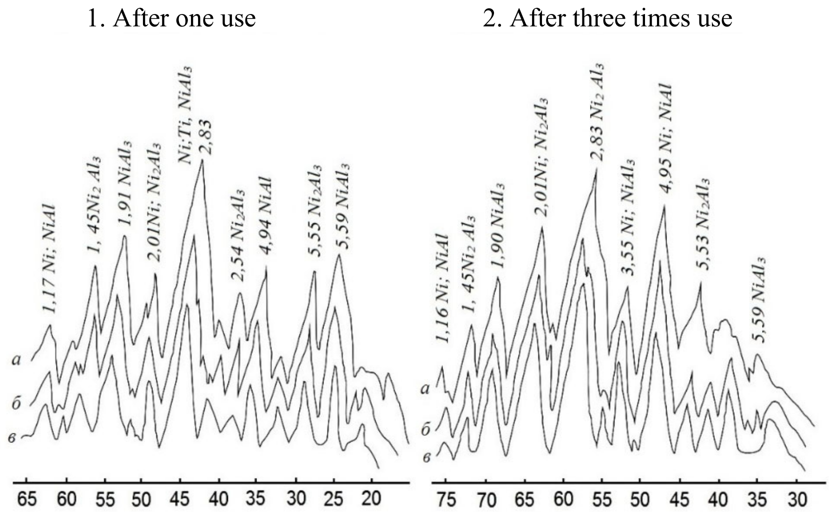

Fig. 2. Diffractogram of promoted nickel -aluminum catalysts.

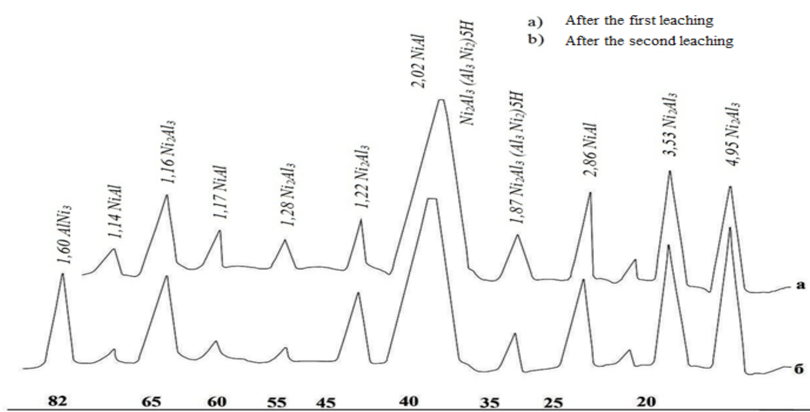

Fig. 3. Diphractograms of nickel -med -alumine catalaliver.

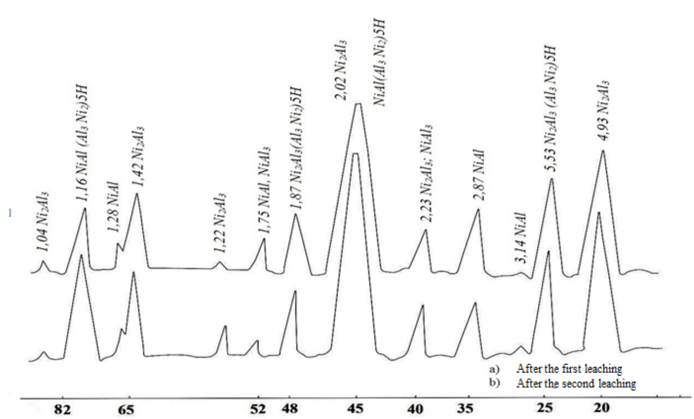

Fig. 4. Diphractograms promoted by nickel -medal -aluminum catalyst.

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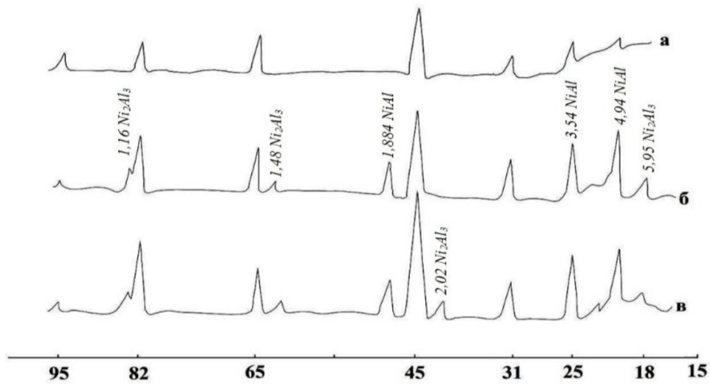

Fig. 5. Diphractograms of nickel -med - hermanium -alumien catalalivers.

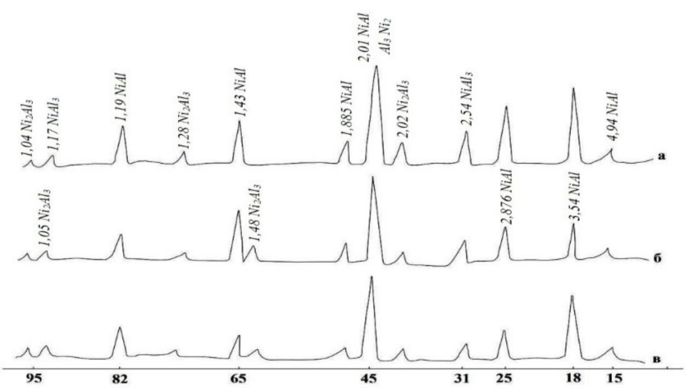

Fig. 6. Diphractograms of nickel -med - hermanium - alumien catalalivers after the second leaching.

Fig. 7. Microscopic images of the surface of the nickel -aluminum catalyst.

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Fig. 8. Microscopic images of the surface of the nickel - honey - aluminum catalyst.

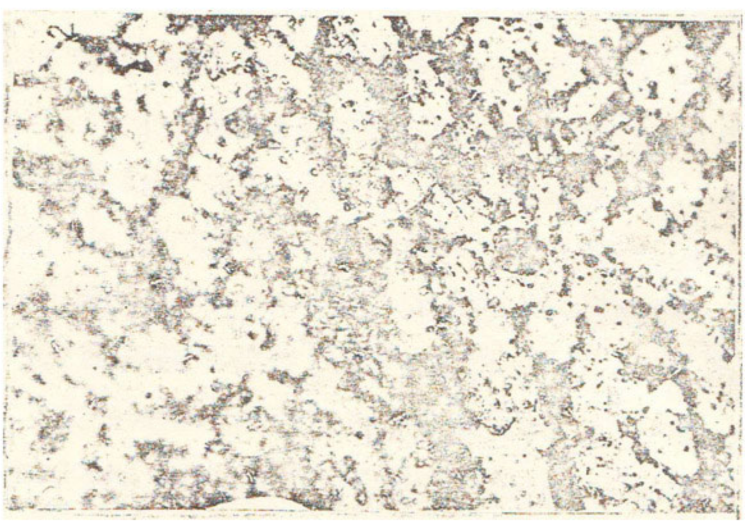

Fig. 9. Microscopic images of the surface of the nickel - rodi - aluminum catalyst.

5 Conclusions

Thus, the additive of Germany in nickel-honey aluminum alloy affects the structural formation of intermetallic compounds, which ultimately leads to an increase in active centers at which the concentration of dissolved hydrogen increases.

A continuous technology of pre-contact hydrogenation of cottonseed oil on stationary and powdered nickel-copper catalysts has been studied and developed for the first time. It has been established that the recommended technology can significantly increase the productivity of hydrogenation plants and reduce the content of trans-isomerized fatty acids in tallow. This provides an increase in the physiological and nutritional value of margarine products based on food fats.

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