

Phase formation and catalytic activity of Cu-Co-spinel catalyst deposited on Al/Si/Mg – support

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The paper is devoted to Cu-Co oxide catalysts deposited on a commercially available high-temperature support containing aluminum, magnesium and silicon, calcined at different temperatures: 350, 450, 550, 650 and 750°C. The samples are prepared by impregnation with aqueous solutions of Cu and Co nitrates. The catalysts are characterized by X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), scanning electron microscopy (SEM), differential thermal analysis (DTA), and BET method. Chemical analysis of the samples is also carried out. The phase formation at different preparation temperatures and the catalytic activity of copper cobaltite in the reaction of CO oxidation with oxygen at two space velocities (20 000 and 100 000 h⁻¹) are investigated. The stability of the specimens to catalytic poisons at the same space velocities is studied. It is established that the most promising catalysts regarding the catalytic activity and stability to poisoning with SO₂ are the catalysts calcined in the range 350 – 550°C. At these temperatures a Cu-Co oxide spinel - like phase is formed. These results give us the reason to suppose a possible implementation of the high-temperature support in the preparation of catalysts for the purification of toxic emissions of gaseous fluids in the practice.

Key words: Cu-Co oxide catalysts, oxidation of CO + O₂, CO + O₂ + SO₂

INTRODUCTION

An important task in the investigations focused on the development of catalysts designed for environment protection, is to elaborate supports of various shape, porosity and thermal stability. The chemical nature of the support plays an important role for the catalytic activity, stability and resistance of the supported active phase to catalytic poisons. Therefore, the creation of such a support is decisive with a view to the industrial application of the catalyst. The spinels based on 3d-transition metals are known to possess high catalytic activity in reactions of complete oxidation of organic compounds, CO and also in the reduction of nitrogen oxides [1–4]. This enables their utilization in processes of neutralization of harmful components in industrial waste gases and in transport exhaust gases. For example, such a catalyst is the spinel copper cobaltite (CuCo₂O₄) applied as active phase in the MPB-11 and RILA catalysts [5, 6]. The activities of these catalysts depend on the formation of spinel structure and on the stoichiometry of the active phase. For this reason, the above cited patents are making use of γ -Al₂O₃ as support, which also possesses spinel

structure thus enabling epitaxial growth of the copper cobaltite active phase on the surface of the γ -Al₂O₃ support. It was ascertained that when the spinel stoichiometry is close to that of CuCo₂O₄, the activity is high and the sensitivity to catalytic poisons is low, including even SO₂ [7-9]. The question arises: is there another type of support, having different nature of the surface in comparison to that of γ -Al₂O₃, which would allow the formation of a spinel phase of copper cobaltite on the carrier surface manifesting high activity in oxidation reactions and at the same time non-sensitivity (resistance) to catalytic poisons including SO₂.

For this reason, the aim of the present work was to investigate the phase formation process at various synthesis temperatures and to observe the respective catalytic activity of copper cobaltite supported on an alumina-silica-magnesia carrier.

EXPERIMENTAL

Preparation of the catalyst samples

Ceramic filter containing alumina, silica and magnesia, manufactured by the company “Hofmann Consult Bulgarien GmbH” was used as a support in the synthesis of the catalyst samples. Its parameters were as follows: diameter 95 mm, thickness 22 mm, porosity 10 PPI, specific surface area $S = 0.7 \text{ m}^2\text{g}^{-1}$

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and it was calcined at 1450°C. The deposition of the active phase containing Cu and Co was accomplished by the impregnation method using an aqueous solution – mixture of the nitrates of the two components. The technology involves the following steps: preliminary heating of the impregnating solution containing 7 g Cu/100 ml + 14 g Co/100 ml, up to 80°C, immersion of the filters in the solution, whereupon the temperature was maintained constant for 1 h. There follows drying at room temperature for 24 h and consecutive calcinations for 3 h at five different temperatures: 350, 450, 550, 650 and 750°C. The obtained catalyst samples are denoted as follows below:

AMC_350 – catalyst sample calcined at T=350°C

AMC_450 – catalyst sample calcined at T=450°C

AMC_550 – catalyst sample calcined at T=550°C

AMC_650 – catalyst sample calcined at T=650°C

AMC_750 – catalyst sample calcined at T=750°C

Characterization of the catalyst samples

The structure and the phase composition of the support and of the so prepared catalyst samples were identified on the X-ray diffractometer Bruker Advance, using CuK α radiation equipped with SolX detector, whose 2 θ value comprises 20 - 800 at a step of 0.04°.

The express BET method (single point) was applied for the evaluation of the specific surface area, based on low-temperature adsorption of nitrogen at the boiling point of liquid nitrogen 77K. The relative error of the method amounts to about 8%.

The chemical analysis of the samples was carried out on a FAAS M5-Thermo Fisher apparatus.

The X-ray photoelectron spectra (XPS) were recorded using monochromatic radiation AlK α (1486.6eV) on a VG ESCALAB MK II spectrometer at base pressure of 1×10^{-8} and 1 eV resolution of the apparatus. The charge effect was corrected (calibrated) based on the C1s peak, related to binding energy of 285 eV. The photoelectron signals of C 1s, O 1s, Al 2p, Mg 2p, Cu 2p $_{3/2}$, Co 2p $_{1/2}$ were recorded and corrected by subtraction of the background of the Shirley type and they were evaluated quantitatively on the basis of the peak area and the cross-sections of photo-ionization following Scofield's algorithm.

The morphology of the surface of the studied catalyst samples was observed by means of a scanning electron microscope JEOL JSM – 5300.

The differential thermal analysis was performed on a combined DTA/TG apparatus

LABSYSTM EVD manufactured by the SETARAM Company (France). Synthetic air was used as carrier gas, fed at a flow rate of 20 ml/min. The rate of heating was 10°C/min. A corundum crucible and a Pt/Pt-Rh thermocouple were used. The maximum achieved temperature upon heating reached 750°C.

The catalytic activity tests were carried out on a continuous-flow catalytic reactor described in [10]. The activities of the catalyst samples were measured in the reaction of CO oxidation by oxygen, whereupon the concentration of CO in the feed was 0.5 vol. %. The experimental runs were carried out at two different space velocities – $W = 20\,000\text{ h}^{-1}$ and $100\,000\text{ h}^{-1}$. The stability of the contact masses with respect to the catalyst poison SO $_2$ was ascertained in the reaction of CO oxidation by air, where the CO concentration was 0.5 vol%, while that of SO $_2$ was 0.03 vol%. This test was also carried out at the same two space velocities $W = 20\,000\text{ h}^{-1}$ and $100\,000\text{ h}^{-1}$.

RESULTS AND DISCUSSION

In order to determine the conditions of synthesis, DTA of dried catalyst (sample AMC_350) was carried out after depositing a phase of the precursors – nitrates of copper and cobalt (Figure 1).

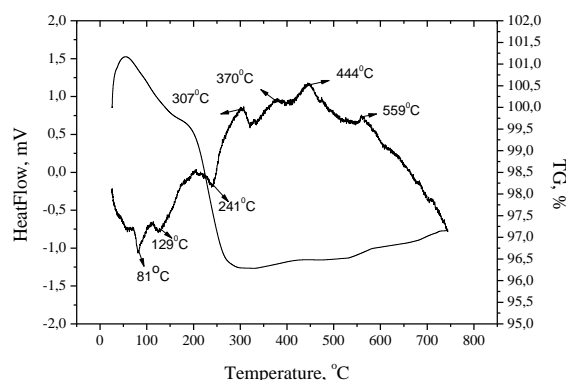


Fig. 1. DTA and TG curves of dry AMC_350 sample, after deposition of the precursors (Cu, Co nitrates)

The low-temperature region displays several endothermic effects at temperatures of 81 and 129°C, which could be attributed to processes of desorption of the water molecules, pre-adsorbed on the sample. Upon increasing the temperature, another endothermic peak is registered at 241°C, which could be attributed to the decomposition of the nitrates and is probably an initial step in the formation of the phases. The endothermic phase transition is accompanied by loss of mass $\Delta m = 4.83\%$ (TG curve), which starts at T~180°C and is completed at T~295°C. Going higher beyond this temperature there are no changes observable in the TG curve. The next increase in the temperature is leading to several exothermic effects, registered at the following temperatures: 307, 370,

444 and 559°C. According to the literature data, these can be explained by decomposition of the copper cobaltite into Co_3O_4 and CuO phases [11-13]. The latter exothermal peak at 559°C can be attributed to consecutive reduction of Co_3O_4 to CoO , as well as to crystallization processes of the oxides and growth of the crystallites. A slight increase in the mass is observed at the same temperature in the TG curve, which is attributed to the same processes. Table 1 lists the data from the chemical analysis of the catalyst samples, the specific surface area and the observed phases in the XRD patterns. As it is seen from the data in the table, the content of copper and cobalt increases with the increase of the synthesis temperature up to 550°C. The ratio between the contents of the two elements is preserved but the ratio does not reach the value 1:2 as in the case of normal spinel composition. This fact gives evidence that the stoichiometric spinel CuCo_2O_4 cannot be formed on the support but non-stoichiometric spinel may be formed. The dependence of the specific surface area of the samples also passes through a maximum, which is due to the formation of new phases on the surface of the support. The data from the XRD analysis of the support and the obtained catalyst samples reveal the presence mainly of the high-temperature modification of Al_2O_3 (i.e. $\alpha\text{-Al}_2\text{O}_3$) and a spinel phase of composition $\text{Cu}_{0.15}\text{Co}_{2.8}\text{O}_4$ judging from the XRD patterns (Figure 2 and Table 1).

It was established that at the different temperatures of calcination of the catalyst samples the degree of dispersion of the spinel phase is changing, whereupon in the case of

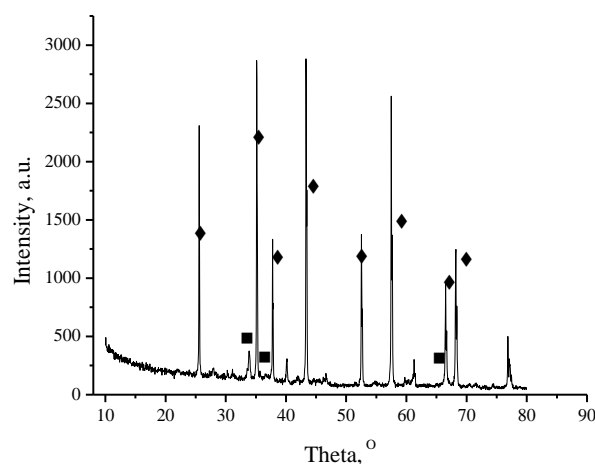


Fig. 2. XRD prevailing phases: \blacklozenge - $\alpha\text{-Al}_2\text{O}_3$, \blacksquare - $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$ and SiO_2 -quartz

catalyst sample AMC_350 the size is 28.4 nm, while for the catalyst sample AMC_750 it reaches 89 nm (Figure 2). In the catalyst samples AMC_650 and AMC_750 calcined at the highest temperatures, there occurs decomposition of the copper cobaltite and the products of this decomposition Co_3O_4 and CuO are so finely dispersed, that it is impossible to register them by XRD measurements. Partial reduction of Co_3O_4 to CoO is also possible at the high temperatures. The analysis of the surface of the formed copper-cobalt mixed oxides on the alumina supports calcined at various temperatures was made by the XPS method. The obtained photoelectron spectra of copper and cobalt are shown in Figure 3, while the data from the analysis are systematized in Table 2.

Table 1. Cu, Co contents, phase composition, specific surface area of the synthesized samples and mechanical strength of the support

Sample Support	Cu, wt %	Co, wt %	Co/Cu	Phase composition, XRD	S_{BET} , m^2g^{-1}	Mechanical strength, kg cm^{-2}
AMC_350	0.84	1.33	1.58	Al_2O_3 , SiO_2 quartz - traces, $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$	0.7 1.32	650 -
AMC_450	1.30	2.15	1.65	Al_2O_3 , SiO_2 quartz - traces, $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$	1.14	-
AMC_550	1.45	2.48	1.71	Al_2O_3 , SiO_2 quartz - traces, $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$	1.13	-
AMC_650	1.55	2.47	1.59	Al_2O_3 , SiO_2 quartz - traces, $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$ - traces	1.01	-
AMC_750	1.40	2.35	1.68	Al_2O_3 , SiO_2 quartz - traces, $\text{Cu}_{0.15}\text{Co}_{2.84}\text{O}_4$ - traces	0.48	-

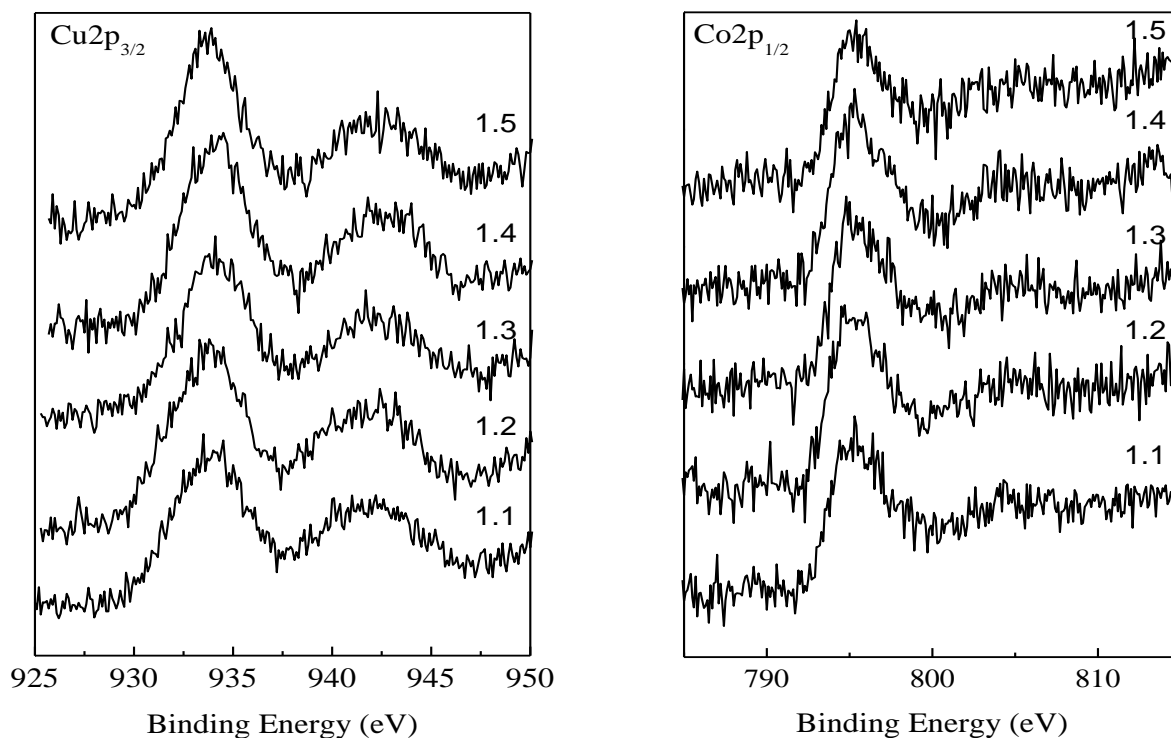


Fig. 3. The Cu2p_{3/2} and Co2p_{1/2} photoelectron spectra of AMC₃₅₀ (1.1), AMC₄₅₀ (1.2), AMC₅₅₀ (1.3), AMC₆₅₀ (1.4) and AMC₇₅₀ (1.5) catalysts.

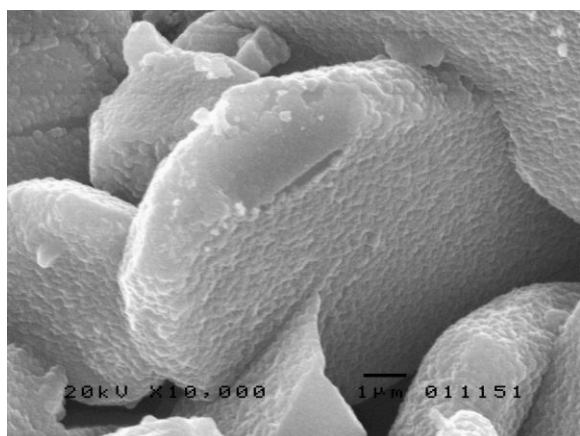
Table 2. XPS binding energies (eV) in the synthesized samples

Samples	Binding energies, eV			
	Co2p _{1/2}	Cu2p _{3/2}	Is/Im	Co-Cu
AMC ₃₅₀	795.1	933.9	0.70	1.41
AMC ₄₅₀	795.3	933.9	0.64	1.47
AMC ₅₅₀	795.0	934.2	0.59	1.44
AMC ₆₅₀	795.2	934.3	0.58	1.11
AMC ₇₅₀	795.1	933.8	0.57	1.33
CuO Merck	-	934.2	0.56	-

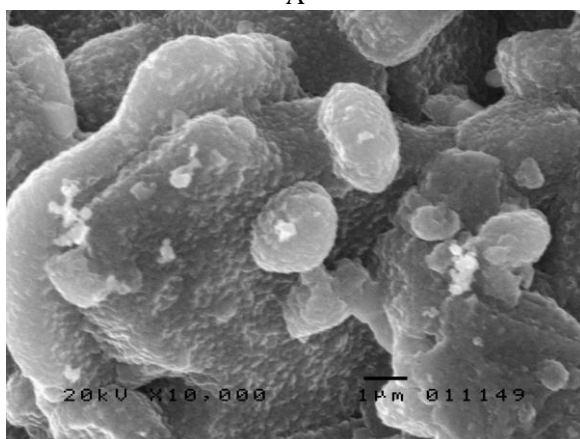
It is obvious from the results for the photoelectron signal of cobalt that it is in the Co³⁺ state, but one cannot exclude the presence of cobalt in the Co²⁺ oxidation state. It is clearly observable in the figure that the copper peak displays a satellite, characteristic of copper in the Cu²⁺ state. The catalyst samples AMC₃₅₀ to AMC₅₅₀ have a distinctly expressed asymmetry of the main photoelectron peak, which is missing in the other two samples AMC₆₅₀ and AMC₇₅₀ calcined at the highest temperatures. The single oxygen peak at about 541.4 eV was estimated to belong to the alumina phase. The calculated ratio between the intensity of the satellite and that of the main copper peak is decreasing and it becomes near to that of CuO - Merck (0.56). This fact gives us the reason to suppose that at low temperatures of calcination copper cobaltite is being formed on the surface, as it has already been observed in our previous studies

[14], while the increase in the temperature of calcination is probably leading to formation of separate phases of copper and cobalt oxides on the surface. These results coincide with the data from the XRD analysis of the same samples. Figure 4 presents SEM micrographs of the catalyst samples AMC₄₅₀, AMC₆₅₀ and AMC₇₅₀, respectively (Fig. 4A, 4B and 4C).

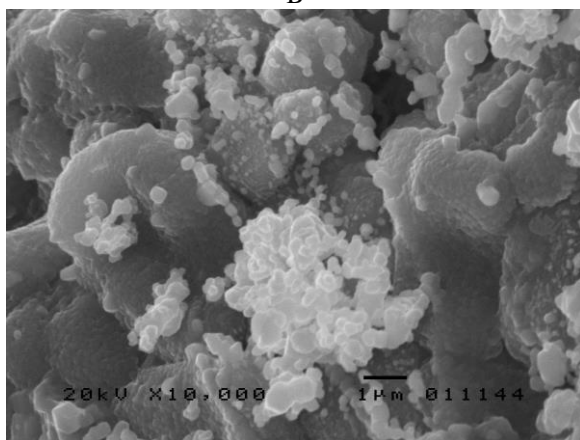
Separate particles of prevailing spherical form are observed in the catalyst sample AMC₄₅₀ (Fig. 4A), whereupon the size of the biggest ones is below 0.36 μm . In the case of catalyst sample AMC₆₅₀ (Fig. 4B) the shape of the particles is also spherical, whereupon the size of the smallest ones is below 0.7 μm . The largest particles have sizes less than 2 μm . Agglomeration of the particles is observed at this temperature. Further, in the case of the highest temperature sample AMC₇₅₀ (Fig. 4C) agglomerates are observed consisting of small particles of spherical shape, with sizes of the order of 0.15 – 0.30 μm and some larger ones of dimensions 0.50 – 0.80 μm , with prevailing spherical shape, which form chain clusters of size below 3 μm . The analysis of the results from the SEM micrographs shows that the particle size is dependent on the calcination temperature of the catalyst samples and it corresponds to the calculations made on the basis of Scherrer's rule in the course of the XRD investigations.



A



B



C

Fig. 4 SEM images for catalysts A – (AMC_450), B – (AMC_650) and C – (AMC_750)

The activities (conversion degrees η) of the catalyst samples in the CO oxidation reaction at different space velocities are shown in Figures 5 and 6.

One can observe the close values of the temperatures at which η is 50% with the catalyst samples AMC_350, AMC_450 and AMC_550 – $T_{50\%} = 175^\circ\text{C}$ at space velocity $W = 20\,000\text{ h}^{-1}$ (contact time $0.53 - 0.54\text{ h g-cat/mol}$). This is due to the formation of copper cobaltite spinel at these

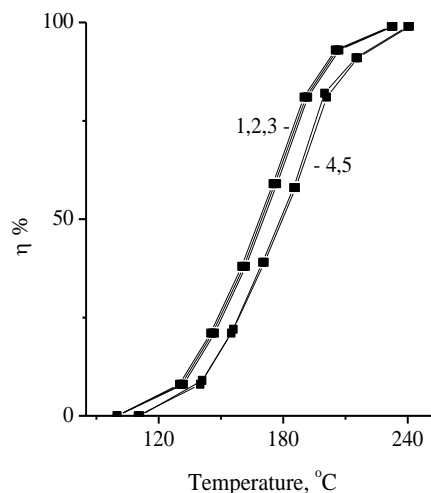


Fig. 5. CO conversion (η %) as a function of temperature of the catalysts: 1-AMC_350, 2-AMC_450, 3-AMC_550, 4-AMC_650, 5-AMC_750, space velocity, $W = 20\,000\text{ h}^{-1}$

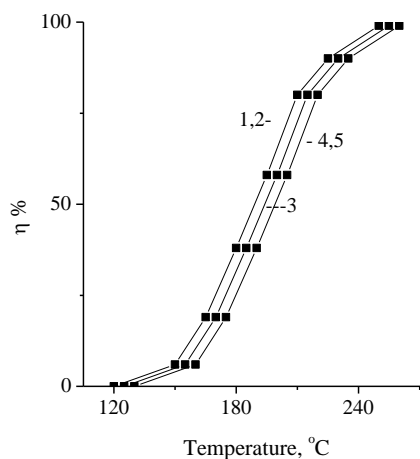


Fig. 6. CO conversion (η %) as a function of temperature of the catalysts: 1-AMC_350, 2-AMC_450, 3-AMC_550, 4-AMC_650, 5-AMC_750, space velocity, $W = 100\,000\text{ h}^{-1}$

temperatures. In the cases of the catalyst samples, calcined at 650 and 750°C, the temperature of the 50% conversion degree is shifted to higher values. Increasing the space velocity up to $100\,000\text{ h}^{-1}$ (contact time $0.11 - 0.12\text{ h g-cat/mol}$), the tendency is preserved, whereupon the catalyst samples AMC_350, AMC_450, AMC_550 are slightly more active than the samples calcined at the higher temperatures.

An analogous picture is obtained in regard to the activity, if the experimental results are presented by means of I_g , which denotes the amount of CO oxidized by 1g of catalyst per hour. These data are shown in Figures 7 and 8. This index also indicates AMC_350 as the most active catalyst sample. Similar activities were manifested by the catalyst samples AMC_450, AMC_550. The samples AMC_650 and

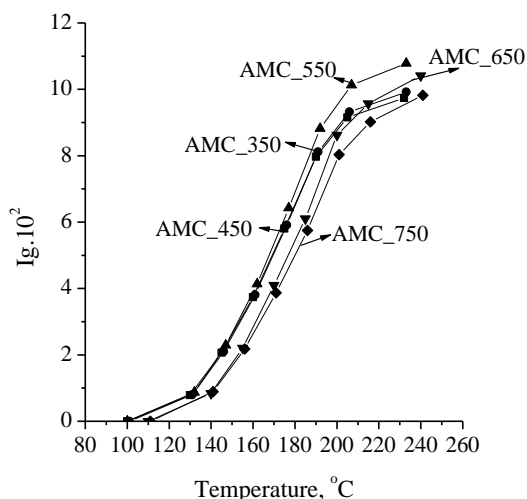


Fig. 7. CO oxidation (milligrams) by 1 g catalyst (I_g) versus temperature for the catalysts: AMC_350, AMC_450, AMC_550, AMC_650, AMC_750 space velocity, $W = 20\,000\text{ h}^{-1}$

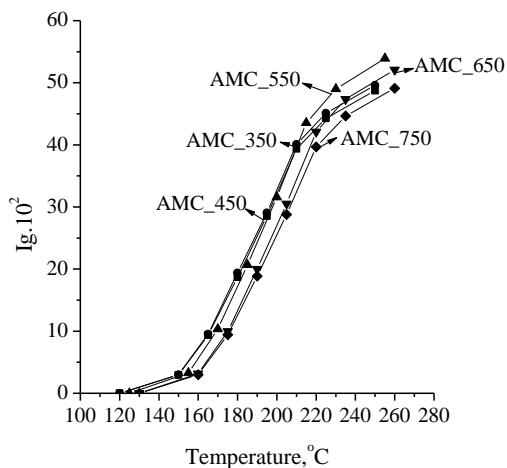


Fig. 8. CO oxidation (milligrams) by 1 g catalyst (I_g) versus temperature for the catalysts: AMC_350, AMC_450, AMC_550, AMC_650, AMC_750 space velocity, $W = 100\,000\text{ h}^{-1}$

AMC_750 display lower activities. It should be noted that the content of copper and cobalt on the surface of the AMC_350 sample (as a sum of the two) is the lowest one - 2.17 wt. %, while in the other catalyst samples it is higher than 3 wt. % and nevertheless, this catalyst sample is the most effective.

In order to check the sensitivity of the catalyst samples with respect to the catalyst poison sulfur dioxide, after reaching 98% conversion degree 30 ppm of SO_2 are fed into the inlet gas mixture flow. After a certain time interval the feeding of SO_2 is discontinued in order to observe whether the poisoning is reversible or irreversible. As it is seen in Figure 9 at space velocity $W = 20\,000\text{ h}^{-1}$, the

activities of the catalyst samples are gradually decreasing with the increase of the time interval of feeding the poison, but they are completely recovered after discontinuing the SO_2 flow. The difference in the performance of the high-temperature catalyst samples and that of the other samples is negligibly small. Quite different is the behavior of the samples at the higher space velocity $W = 100\,000\text{ h}^{-1}$ (Figure 10).

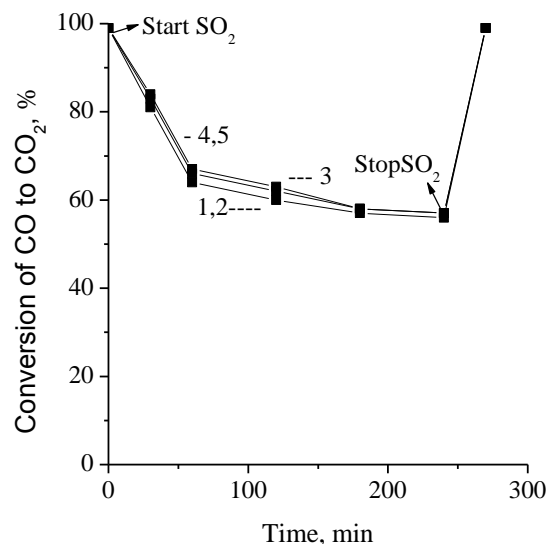


Fig. 9. Poisoning of the catalysts: 1-AMC_350, 2-AMC_450, 3-AMC_550, 4-AMC_650, 5-AMC_750 during the reaction oxidation of $\text{CO} + \text{O}_2$, space velocity, $W = 20\,000\text{ h}^{-1}$

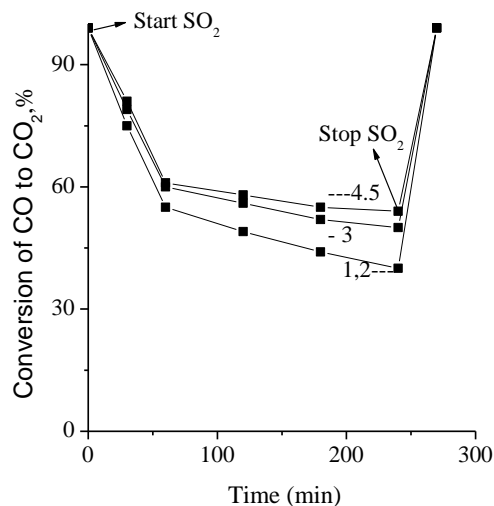


Fig. 10. Poisoning of the catalysts: 1-AMC_350, 2-AMC_450, 3-AMC_550, 4-AMC_650, 5-AMC_750 during the reaction oxidation of $\text{CO} + \text{O}_2$, space velocity, $W = 100\,000\text{ h}^{-1}$

During the feeding of the SO_2 poison the catalyst samples AMC_350, AMC_450 lower their activities down to 40%, while in the cases of the other samples the activities decrease to 50 – 55%. In this case it is also observable that the poisoning of the catalyst

samples is reversible. This is probably due to blocking of a fraction of the active sites participating in the CO+O₂ reaction, by the poison, which occurs as a result of formation of unstable superficial sulfate complexes under the effect of SO₂, which is followed by their desorption after stopping the SO₂ flow.

These results show that the formation of a spinel phase of copper cobaltite alters the sensitivity towards this strong catalyst poison – sulfur dioxide. It is known that the oxides of the transition metals, in particular the oxides of copper and cobalt, are active catalysts in oxidation reactions, but in the presence of this poison they lose 100 % of their activity. The poisoning is also reversible in the case of these two metal oxides and the initial activity is restored after discontinuing the poison flow. In the present investigation, the oxides present in the catalyst samples lose their activity and effectiveness completely, but the spinel phase is preserved and it continues its action, as it is non-sensitive to sulfur dioxide and therefore these catalyst samples preserve a comparatively high activity.

Additional information can be obtained if the data on the activity of the catalyst samples are presented by means of the quantity I_g, at one and the same reaction temperature, in dependence of the synthesis temperature of the catalyst samples.

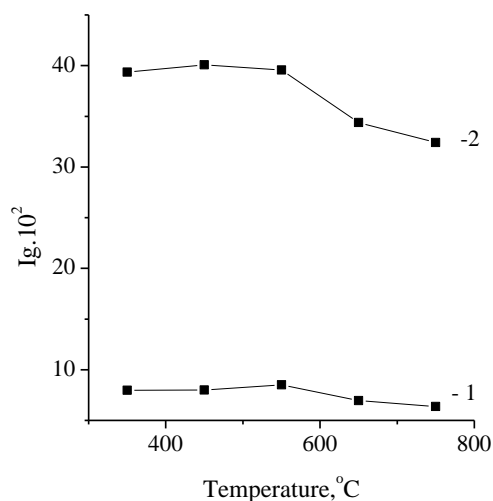


Fig. 11. CO oxidation by 1 g catalyst versus temperature and different space velocity:

- at T=190°C, space velocity W = 20 000 h⁻¹ (curve 1)
- at T=210°C, space velocity W = 100 000 h⁻¹ (curve 2)

Figure 11 illustrates this dependence for the studied catalyst samples at the two space velocities. As it can be seen from the figure, the catalyst samples obtained at lower temperatures, having a higher content of the spinel phase, preserve their high effectiveness. In the cases of higher synthesis

temperatures – above 650°C, at which the spinel is probably starting to decompose, the activities are lower and the sensitivity towards catalytic poisons increases.

CONCLUSION

On the basis of the results from the present investigation it was ascertained that:

The phase formation process in the synthesized catalyst samples deposited on high-temperature alumina-silica-magnesia support plays an essential role with respect to both the catalytic activity in the reaction of CO oxidation by oxygen from the air and the stability/resistance to catalyst poisons, in particular SO₂.

Of substantial importance for obtaining a spinel-like active phase are the structure and the texture of the support, as well as the solid-state processes occurring during the thermal treatment of the contact mass.

Most promising with a view to the activity in the studied reaction and stability to poisoning by SO₂ are the catalyst samples calcined within the temperature interval 350 – 550°C, enabling the synthesis of a spinel-like Cu/Co oxide phase.

This gives us the reason to propose practical application of the high-temperature support for depositing catalysts for purification of toxic emissions of gaseous fluids.

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ФАЗООБРАЗУВАНЕ И КАТАЛИТИЧНА АКТИВНОСТ НА Cu – Co ШПИНЕЛЕН КАТАЛИЗАТОР НАНЕСЕН ВЪРХУ Al/Si/Mg – НОСИТЕЛ

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(Резюме)

В работата са представени Cu-Co оксидни катализатори, нанесени върху фирмен високотемпературен носител, съдържащ алуминий, магнезий и силиций и накалиени при различни температури: 350, 450, 550, 650 и 750⁰C. Образците са получени по метода на пропиването от водни разтвори на нитрати, съдържащи Cu и Co. Катализаторите са охарактеризирани посредством рентгенови фотоелектронни спектри, рентгенофазов анализ, диференциален термичен анализ, сканираща електронна микроскопия и по метода на БЕТ. Направен е химически анализ на образците. Изследвано е фазообразуването, при различни температури на получаване и каталитичната активност на меден кобалтит в реакцията на окисление на CO с кислород при две обемни скорости (20 000 и 100 000h⁻¹). Проследена е и устойчивостта на образците към каталитични отрови като SO₂ при същите обемни скорости (20 000 и 100 000h⁻¹). Установено е, че най-добра перспективност по отношение активността на разглежданата реакция и стабилност спрямо отравяне и активност съчетана с SO₂ показват катализаторите, накалиени в температурен интервал 350 – 550⁰C, при която температура се синтезира шпинелоподобната Cu-Co оксидна фаза. Това ни дава основание за възможната приложимост на високотемпературния носител за нанесени катализатори за очистване от токсични емисии на газове флуиди.