



Article Influence of Magnetic Fields Assisted for Preparation of Ferromagnetic Mono- and Bi-Metallic Co and Co–V SHS Catalysts on Their Activity in Deep Oxidation and Hydrogenation of CO₂

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Abstract: Co–Al and Co–V–Al intermetallics produced by centrifugal self-propagating high-temperature synthesis (SHS) were used as precursors for preparation of catalysts for deep oxidation and hydrogenation of CO₂. Leaching in NaOH solution and stabilization with H_2O_2 solution of precursors were carried out in permanent magnetic field (MF) (0.24 T) and alternating magnetic field (0.13 T, 50 Hz). Prepared Co μ Co–V (95Co–5V, 90Co–10V) granular catalysts with size of 100–300 μ m were characterized by XRD, SEM, EDS, and BET method and revealed to have a scaly surface structure. It was shown that the type of MF affects phase composition and surface morphology, as well as specific surface and activity in deep oxidation of CO and hydrocarbons as an important part of the neutralization of gas emissions, and hydrogenation of CO₂, the processing of which would reduce atmospheric pollution with this greenhouse gas. Catalysts obtained in alternating MF was found to possess higher activity in the process of deep oxidation.

Keywords: magnetic field; self-propagating high-temperature synthesis; ferromagnetic catalysts; deep oxidation; CO₂ hydrogenation

1. Introduction

Of particular interest is to study various aspects of magnetic field (MF) influence on different substances and materials (ferromagnets, paramagnets and diamagnets). Studies showed significant effects of such action on metals and their alloys (both ferrous and non-ferrous ones), melts during their crystallization, and in some cases on the solid phase. To data, in Fe–C alloys [1,2], a strong MF changes the eutectoid composition and increases austenite/ferrite temperature that leads to a shift in phase equilibrium. The application of external MF increases the temperature and accelerates the bainitic transformation for Fe–3.6Ni–1.45Cr–0.5C steel and the martensitic transformation for 18Ni steel [3]. In case of Fe73.5Si13.5B9Nb3Cu1 amorphous precursor crystallized by annealing at temperatures between the Curie temperatures of amorphous and crystalline phases [4], the existence of MF increases the nucleation rate of ferromagnetic α -Fe(Si) grains from paramagnetic amorphous phase; their volume fraction grows with increasing MF strength. As was shown in [5], the precipitation of nanocrystalline α -Fe(Si) phase in Fe–Si–B amorphous alloy is favorable to an increase in magnetization. MF was found in [5,6] to suppress the crystallization process. The use of pulsed MF in solidification of alloys (1Cr18Ni9Ti austenitic stainless steel [7], Mg-Al-Zn [8], Al-Cu [9], pure Al [10]) leads to refinement and modification of the microstructure that is accompanied by improving mechanical properties [8,11]. As noted in [7], the grain size of solidified structure grows as the MF induction increases. In addition,



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the action of pulsed MF markedly reduces the solidification time and increases the initiation and finishing solidification temperature. Results from diamagnetic zinc [12], zinc-based alloy [13], and bismuth [14] showed magnetically induced grain boundary migration in the direction of the grain with higher diamagnetic susceptibility followed by change in their crystallographic orientation. There is a number of experimental studies devoting to an impact of strong MF on annealing behavior of cold-rolled materials. Magnetic annealing of cold-rolled interstitial-free steel was shown in [15] to inhibit crystallization and to cause elongation of recrystallized grains along the MF direction. However, in case of cold-rolled pure Cu, the annealing promotes the recovery and recrystallization processes [16]. Grain growth was also observed for cold-rolled commercial pure Zn [17] and Ti [18], iron alloys, non-ferrous materials, and steels [19], which was attributed to an enhanced mobility of grain boundaries.

Thus, this suggests that applying the MF during the synthesis of catalysts with a metal-containing active phase (AF) can significantly modify the texture and microstructure of AF, as well as its chemical composition. As is known, the synthesis of catalysts is often carried out in the liquid media including water. Therefore, let us consider the impact of MF on water and water solutions. It was shown in [20,21] that under such influence, the number of hydrogen bonds increases, bonding between the water molecules becomes tighter, and structure of the liquid water changes. The self-diffusion coefficient of the water molecules in this case reduces thus inducing changes in thermal conduction and viscosity. Treatment in 0.65 T MF favors an increase in evaporation rate and a slightly decrease in surface tension [22]. The refractive index of water was found in [23] to increase by 0.1%; meanwhile that of aqueous electrolyte solutions decreases. An increase in melting temperatures of H₂O and D₂O treated in 6 T MF up to 5.6 mK and 21.9 mK, respectively, was observed in [24]. The action of permanent MF was revealed to slow down the rate of crystal growth of calcite suspended in a fluidized bed [25]. In summary, the action of MF on water and water solutions causes the variations in their physicochemical characteristics. In this context, there is good reason to believe that the structure and properties of catalysts are also modified by applying external MF during their synthesis. This point is poorly studied. There are just a few works touching on both properties of catalysts [26] and processes with their participation, in particular, electrocatalysis [27,28] when external MF is applied. In [29], Ni-Fe bimetallic catalysts prepared by chemical reduction with MF was shown to possess high activity for CO2 methanation. Application of electromagnetic field to Fe₃O₄ (as magnetic) and ZnO (as dielectric) nanocatalysts during green urea synthesis was considered in [30]. It was shown that this impact reduced the activation energy of Fe_3O_4 by 36.6% as compared to ZnO due to its higher saturation magnetization, this improves the singlet to triplet conversion. Improvement of photoelectrochemical and photocatalytic properties of Ag–BiVO₄–MnO_x photocatalyst was explained in [31] by the generation of local electric and magnetic field formed by plasmonic Ag under ultraviolet light. Similar effects of generation of porous madnetoplasmonic Ag/Fe_3O_4 was observed in [32]. By applying permanent MF, the hydrogen evolution activity of Weyl semimetal catalysts (NbP, TaP, NbAs, and TaAs) was found in [33] to increase by up to 95%.

Previously, we prepared polymetallic catalysts from complex multicomponent intermetallics produced by centrifugal self-propagating high-temperature synthesis (SHS), which possess high activity and stability in the processes of deep oxidation of carbon monoxide and hydrocarbons, as well as in the processes of CO₂ hydrogenation [34–36]. Iron-group metals with promoters —V, Zr, Mn, and other transition and rare earth metals —are the main components of the above-mentioned catalysts. Their active phase is a highly disordered, to a large degree X-ray amorphous, and oxo-metallic layer with nanostructured surface, which was deposited on the residual non-leached intermetallic compound with reduced aluminum content. During synthesis in water solution, there is a transition from dia- or paramagnets (intermetallics) to ferromagnetic catalysts. Taking it into consideration, it is reasonable to expect a marked influence of MF (permanent or alternating) on the mechanism of this transition and accompanying changes in the structure and properties of catalysts. Studying this effect will make it possible to control the properties of catalysts. In [37], we first considered the effect of MF assisted for synthesis of polymetallic Ni–Co–Mn catalysts on its physicochemical and catalytic properties.

We pioneered in applying permanent and alternating MF during liquid-phase synthesis of Co and Co–V catalysts from intermetallic SHS precursors. This research was aimed at studying the MF effect on chemical, physical, and catalytic properties of Co, 95Co–5V, and 90Co–10V catalysts. The idea lied in the fact that the ferromagnetic properties of catalysts enabled to reveal results of the impact of MF already at moderately low induction of both permanent and alternating MFs. Catalytic activity of prepared catalysts was studied in the processes of deep oxidation of CO and propane as an important part of the neutralization of gas emissions, as well as of hydrogenation of CO₂. Carbon dioxide is a promising renewable carbon-containing raw material, the processing of which would reduce atmospheric pollution with this greenhouse gas.

2. Materials and Methods

Intermetallic precursors used in this work as starting materials were prepared by centrifugal SHS. The SHS reaction yielding higher intermetallic compounds MAl₃ can be represented by the following scheme (1):

$$Co_3O_4 + xV_2O_5 + (35 + 28x)/3Al \rightarrow 3CoAl_3 \cdot 2xVAl_3 + (4 + 5x)/3Al_2O_3$$
 (1)

The synthesis was carried out in a SHS machine at the centrifugal acceleration *a* of up to 400 g. (Figure 1).



Figure 1. Schematic of experimental setup: *1* rotor, *2* removable reaction chamber, *3* centrifuge frame, *4* electric motor support, *5* sleeve-pin coupling, *6* electric motor, *7* tachometer, *8* reaction mold, *9* collector with current leads, and *10* bearing unit cover.

The action of gravity forces favors the separation of combustion product into two layers—complex intermetallic compound and aluminum oxide slag with traces of undereducated target elements. In order to prepare catalysts, intermetallic ingot was crashed followed by sieving out 100–300 μ m fraction, and then was subjected to leaching with NaOH solution (spontaneous reaction for 1 h, boiling for 1 h, and holding at room temperature for 24 h), wash-out, and stabilization with 10% H₂O₂ solution (for 0.5 h). The last step removed residual hydrogen after leaching of catalysts and contributed to the formation of a thin layer of chemisorbed oxygen on their surface in order to prevent oxidation by air. Dry freshly leached air-unstabilized catalysts are pyrophoric. The leaching and stabilization stages were carried out in permanent (0.24 T, samples **D**) or alternating (0.13 T, samples **A**) MF. The preparation of catalysts under study was described more fully in our previous papers [34–37]. The MF action method was presented in [37].

Prepared catalysts —Co (samples I), 95Co–5V (wt %, samples II), and 90Co–10V (wt %, samples III) —were characterized by XRD (DRON-3 diffractometer, Fe K α radiation) and SEM/EDS (Zeiss Ultra plus microscope + JCXA-733 Superprobe JEOL). Specific surface was determined by BET method.

The catalytic activity of obtained samples was determined using the flow fixed bed silica reactor described in detail elsewhere [35–39].

Deep oxidation was performed in a gaseous mixture containing (vol %) 0.2 propane, 0.6 CO, 2 O₂, and nitrogen the rest at a gas hour space velocity (GHSV) of 120,000 h⁻¹. Hydrogenation tests were carried out at GHSV = 6000 h⁻¹ using the mixture containing (vol %) 5 CO₂, 20 H₂ (H₂: CO₂ = 4: 1), and helium the rest. The composition of initial gas mixtures and reaction products was analyzed by AVTOTEST 02.03.P gas analyzer (META, Zhigulevsk, Russia) and 3700 gas liquid chromatography machine (Moscow, Russia).

The conversion of CO, propane, and CO₂ in deep oxidation and hydrogenation was calculated using the following formula:

$$\chi = [(x_0 - x_T)/x_0] \cdot 100\%$$
⁽²⁾

where x_0 is the component concentration in the initial mixture, x_T is the component concentration in the reaction products at temperature *T*. The higher is the conversion at given temperature, the greater is the activity of catalyst.

3. Results

3.1. XRD Analysis

Phase compositions of intermetallic precursors were given in [39]. We showed that intermetallic $Al_{13}Co_4$ (monoclinic) is the major phase of (100Co) Al_x precursor; Co–V–Al precursors consist of Al_5Co_2 , $Al_{0.52}Co_{0.48}$, and $Al_{80}V_{20}$.

Table 1 presents the XRD data of prepared catalysts.

 Table 1. Phase composition of precursors and catalysts.

Sample	Phase Composition				
IA	Co, Al, Co(OH) ₂ , Al ₂ O ₃ , Al ₁₃ Co ₄ (orthorhombic)				
ID	Co, Al, Co(OH) ₂ , Al ₂ O ₃ , Al ₁₃ Co ₄ (orthorhombic)				
IIA	Co, Al ₁₃ Co ₄ (orthorhombic), Al _{4.85} Co _{5.15} , Co ₃ O ₄ , CoAl ₂ O ₄				
IID	Co, Al ₁₃ Co ₄ (orthorhombic), AlVCo ₂ , AlCo				
IIIA	Co, AlVCo ₂ , Al ₁₃ Co ₄ (orthorhombic), CoV ₃				
IIID	Co, Al ₁₃ Co ₄ (orthorhombic), AlVCo ₂ , AlCo				

In sample I, the type of MF effects no phase composition of prepared catalysts. These samples (IA and ID) are seen (Table 1) to consist of Co, Al, Co(OH)₂, Al₂O₃, as well as of Al₁₃Co₄ just as in non-leached precursor. Note that residual intermetallic Al₁₃Co₄ transformed from monoclinic structure into orthorhombic one as a result of post-combustion process in MF.

Catalysts obtained from V-containing precursors are seen to contain different phases, save for main Co phase and $Al_{13}Co_4$ phase with residual Al. Residual intermetallic $Al_{13}Co_4$ was most likely formed during leaching. In addition to basic phases in sample **IIA**, there are also oxide and complex oxide phases. Note that samples **IID** and **IIID** have the same phase composition.

3.2. Elemental Mapping

Figures 2 and 3 show the SEM images and corresponding elemental maps of $(95Co-5V)Al_x$ and $(90Co-10V)Al_x$ precursors. Elemental maps presented in Figure 2 and EDS analysis of $(95Co-5V)Al_x$ precursor revealed the following structural constituents: AlCo

matrix, Al–V dendrite precipitations, and Co-rich particles matched to intermetallic Al₅Co₂ from XRD data. As it easy to see, there are inclusions of pure V, which are not detected by XRD analysis.



Figure 2. SEM image and EDS elemental maps (Al, V, and Co) for (95Co-5V)Al_x precursor.



Figure 3. SEM image and EDS elemental maps (Al, V, and Co) for (90Co-10V)Al_x precursor.

In case of (90Co–10V)Al_x precursor (see Figure 3), two Co-rich and Co-depleted AlCo phases and Al–V particles are observed. No other phases in SEM image were found.

3.3. Precursor Surface Modification by Etching

In order to study the leaching-affected surface morphology, we etched the mechanically polished sections of intermetallic precursors in 20-% NaOH solution with a holding time varying from 5 to 30 min with subsequent wash-out and stabilization in H_2O_2 solution. Figure 4 shows the SEM images of the modified surface of (90Co–10V)Al_x samples. As follows from Figure 4a–e, the treated surfaces represent spherical particles with their size about 100 nm. However, in case of 30-min holding (Figure 4f), the surface starts to be

covered with a layer of nanoscaled species, thus forming a branched structure. On this basis, to prepare the catalysts under study, we increased a leaching time to 2 h with additional holding for 24 h.



Figure 4. SEM images of the surface of samples of $(90Co-10V)Al_x$ precursor affected by leaching for (a) 5, (b) 10, (c) 15, (d) 20, (e) 25, and (f) 30 min followed by wash-out and stabilization.

3.4. Specific Surface and Morphology of Catalysts

Specific surface values for catalysts are given in Table 2. Samples I–III obtained in permanent MF have higher specific surface that that obtained in alternating MF. Note that catalysts containing 5% V (samples IID and IIA) are characterized by maximum specific surface values, no matter what type of MF was applied.

Table 2. Specific surface of catalysts.

Sample	ID	IID	IIID	IA	IIA	IIIA
Specific surface s , m ² /g	7.0	17.5	6.7	2.4	10.5	3.1

SEM images of surfaces of catalyst granules obtained in MFs are presented in Figures 5 and 6. These granules are seen to contain cracks and pores, which were formed when removing aluminum from precursor powder by leaching. The granule surfaces have a scaly structure manifested as interconnected plates with a thickness below 100 nm (as one can clearly see in Figure 5b). As was shown in [33], these plates have the form of distorted hexahedrons. In all cases, distorted plates are seen to be clearly perpendicular to the granule surface. It should be noted that the permanent and alternating MFs act in different way on the surface structure. So, sample **IID** contains crystallites on the surface of plates as differentiated from catalyst prepared in alternating MF. However, the catalysts containing 10% V (Figures 5c and 6c) are covered by denser layer of thinner plates.



Figure 5. SEM images of (a) Co, (b) 95Co–5V, and (c) 90Co–10V catalysts prepared in permanent MF.



Figure 6. Cont.



Figure 6. SEM images of (a) Co, (b) 95Co-5V, and (c) 90Co-10V catalysts prepared in alternating MF.

From the above reasoning we can get the conclusion that the catalyst granule may be imagined as a core of residual intermetallic compound with an outer porous shell consisting of the plates observed above. According to EDS results, the catalyst surface is covered with oxide layer. Therefore, conceivably these plates might represent oxide or oxometallic products of post-combustion processing; however, they could not be identified within this study.

3.5. Catalytic Activity in Deep Oxidation and Hydrogenation of CO₂

Figures 7 and 8 show the conversion of CO and propane in deep oxidation over samples I–III obtained in permanent and alternating MF, respectively, as a function of temperature.



Figure 7. Cont.



Figure 7. Conversion in deep oxidation of *1*, 2 CO and *3*, 4 propane over samples (**a**) **ID**, (**b**) **IID**, and (**c**) **IIID** as a function of temperature. *1*, 3 first cycle; 2, 4 s cycle.



Figure 8. Conversion in deep oxidation of *1*, 2 CO and *3*, 4 propane over samples (**a**) **IA**, (**b**) **IIA**, and (**c**) **IIIA** as a function of temperature. *1*, 3 first cycle; 2, 4 s cycle.

In cycle 1, 80% propane conversion for all samples is seen to be reached already at temperature of 250°C. Samples **ID** and **IA** exhibited 100% CO conversion around 175°C. In other cases, including data of cycle 2, this temperature was 200 °C. For all catalysts, propane oxidation got started after complete oxidation of CO. Cycle 2 results indicated that if the catalyst activity in propane oxidation markedly drops, so the activity in CO oxidation significantly increases. This change in activity can be caused by oxidation of catalyst surface during cycle 1, thus forming metal–oxide phases showing higher activity in CO oxidation and lower one in propane oxidation.

It is worth noting that the catalysts obtained in alternating MF are characterized by higher activity than that prepared in permanent MF.

For all catalysts, hydrogenation of CO_2 occurred with the formation of predominantly methane. CO_2 conversion–temperature dependencies are seen in Figure 9 to have a maximum corresponding to maximum methane yield. Then there is a decrease in CO_2 conversion that was accompanied by dropping methane yield; as a result, a CO byproduct was formed.



Figure 9. Conversion of CO₂ in hydrogenation process over catalysts: (a) 1 ID, 2 IID, and 3 IIID; (b) 1 IA, 2 IIA, and 3 IIIA as a function of temperature.

Catalysts **ID** and **IA** possess the highest hydrogenating activity among studied samples: 67.7% and 70% at 300 °C, respectively. CO₂ conversion curves for samples **IID** and **IIA**, **IIID** and **IIIA** are close to each other. The catalyst activity was unaffected by type of MF.

4. Conclusions

The first Co II Co–V (95Co–5V, 90Co–10V) catalysts to be prepared by leaching of SHS-produced granular intermetallics in NaOH solution followed by stabilization with H_2O_2 solution, which were carried out in permanent magnetic field (0.24 T) and alternating magnetic field (0.13 T, 50 Hz), for deep oxidation of CO and propane, and methanation of CO₂. These reactions underlie the processes of purification of gas emissions and prevent an increase in the CO₂ content in the atmosphere as the most important greenhouse gas.

The granular catalysts have a scaly surface structure manifested as interconnected distorted plates with a thickness below 100 nm and show specific surface in the range of 2.4–17.5 m²/g. The type of magnetic field used during the preparation of catalysts effects on (a) phase composition and surface morphology of V-containing catalysts; (b) specific surface, (c) activity in deep oxidation of CO and propane, and (d) activity in hydrogenation of CO₂ of V-free catalysts. The influence of MF is more pronounced in the process of deep oxidation; in this case, higher activity is observed for alternating magnetic field.

The proposed route is appropriate in the case of ferromagnetic catalysts; however, it is of limited application for diamagnetic catalysts as to do this would require stronger magnetic fields.

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