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Thermodynamic modeling and production of FeCo alloy from mill scale through metallothermic reduction

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ABSTRACT

Mill scale is a waste material arises during the continuous casting of steel. It contains approximately 70% iron by mass. Mill scale is used as an iron source in many applications to reduce costs. In this study, it was aimed to produce FeCo alloy through the metallothermic reduction of mill scale and Co_3O_4. The metallothermic reduction was chosen because of its advantages such as minimum energy need, short process time and no need for high-tech equipment. The system was thermochemically simulated with FactSage and HSC software. In the experimental studies, the effects of the use of aluminium, as a reductant, in different stoichiometries on total metal recovery yield were investigated. The effects of aluminium stoichiometry on hardness and microstructure were also investigated. As a result of the experimental studies, it was seen that highest metal recovery yields were obtained as 81.25% for iron and 93.78% for cobalt with the mixture including 105% stoichiometric aluminium.

1. Introduction

Alloying elements significantly affect the properties of steels and each alloying element has a different effect on properties. Cobalt is not a commonly used alloying element in low-alloy steels because of its high price. On the other hand, it is used in the alloying of specialty steels. Cobalt is used for increasing the hot hardness of high-speed tool steels. It is used in the range of 5% to 12% in molybdenum and tungsten high-speed steels. In maraging steels, cobalt is used between 8.5% and 12% to decrease the solubility of molybdenum in the martensitic matrix. Cobalt is also used in high-fracture toughness steels to enhance the precipitation of $(Mo, Cr)_2C$ phase. This phase increases the strength and raises the martensitic transformation temperature of these steels. In some high-performance carburizing martensitic stainless steels, cobalt is used for increasing fracture toughness, hot hardness, metal-to-metal wear resistance and corrosion resistance. Cobalt also improves the wear resistance and resistance to thermal fatigue cracking of martensitic stainless steel hardfacing alloys [1].

The alloying of steels is carried out by using ferroalloys. The elements used in alloying of the steels are difficult to obtain in pure form. Also, considering that the material to be alloyed is steel, it is unnecessary to eliminate iron components in the structure of the alloying element. Furthermore, these alloying elements may not maintain their stability at steel production temperatures. Besides, the oxygen and nitrogen affinity of the alloying elements is high, which creates the possibility of reaction before the alloying process. Additionally, the costs of alloying elements in the form of ferroalloy are lower than their elemental forms. For the reasons mentioned herein, cobalt and other alloying elements are added to the steel structure in the form of ferroalloys [2].

Ferrocobalt (FeCo) is a soft magnetic material. Soft magnetic materials can be easily magnetized and made magnetic under a low magnetic field and they are considered as an important material class because of this property [3]. Magnetic properties of these materials are important for applications involving power generation and distribution, actuating mechanism, magnetic imaging, data storage and microwave communication [4]. For the development of motors and generators used in aerospace and high power applications, it is necessary to use soft magnetic materials with high mechanical strength. A large number of researches were carried out about high strength soft magnetic materials and significant improvements have been achieved in this field [5]. Literature shows that FeCo intermetallic alloys have an excellent combination of high saturation magnetization, high Curie temperature, low crystalline anisotropy and good strength [4]. These materials are quite suitable for applications requiring high current density [4]. Due to FeCo's unique magnetic properties, this material can also be used in high-speed pumps [6].

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Lawrence et al. produced FeCo by electrolytic method from copper ores initially [7]. Powder metallurgy, ball milling, direct co-precipitation, combustion wave, electric arc melting, induction melting, polyol and controlled chemical synthesis are the methods used to produce FeCo and nano FeCo alloys [6,8–12]. White and Wahl inspected FeCo alloys' properties and they stated that presence of elements such as S, P, N and O are detrimental to machinability, and to the mechanical and magnetic properties of FeCo alloys. Therefore, control of contaminations is necessary. In metallothermic reduction process, which was used to synthesize desired alloys in this study, final product can't include S, N and P contaminations, because these elements are not present in the raw materials (reactants and reductants) [13].

In the Kawahara's study, in order to produce FeCo, metallic form of the element was melted at 1565 °C and desired alloy composition was obtained by adding cobalt and vanadium to liquid metal bath [14]. Amount of energy needed to reach 1565 °C is quite high. On the other hand, metallothermic reduction is an exothermic process and it uses the energy that is produced by the reaction itself. This is one of the important advantages of metallothermic reactions.

Iron oxide layer formed as result of oxidation occurred on steel slab and billet surfaces during annealing process in rolling mills, continuous casting plants and annealing furnaces is called mill scale [15]. At high temperatures, three types of iron oxide are formed by the iron surface. These are wustite (FeO), hematite (Fe_2O_3) and magnetite (Fe_3O_4) [16]. Approximately 3500 tons of mill scale is accumulated in mill scale pits per year and it is used as sintering input [17]. Mill scale can be a useful resource when it is recycled. 13.5 million metric tons of mill scale is formed annually in the world [18]. Mill scale was used as iron source in metallothermic reduction experiments in order to utilize this waste material.

In casting applications, the use of master alloy rather than the addition of alloying components in the elemental form to the liquid steel bath is advantageous. Densities and melting points of the alloying components can create problems in the alloying process. If melting points of the alloying components are higher than the liquid metal bath, they will not melt in the liquid metal bath. On the other hand, if melting points of the alloying components are much lower than the liquid metal bath, they would evaporate in a short time. If densities of the alloying components and liquid steel bath are much different from each other, alloying components will float or sink to the bottom of the liquid metal bath. Master alloys are used to overcome these problems. One of the FeCo master alloys is Permendur 24. Permendur 24 contains 24 mass % Co, 76 mass % Fe. In this study, Permendur 24 FeCo alloy which is used in casting applications was chosen as target alloy.

In this study, production of FeCo by metallothermic reduction was investigated for the first time. Metallothermic reduction is a good alternative to other methods mentioned here, because it is an environmentally friendly process and it doesn't produce any CO_2 emission.

2. Materials method

2.1. Materials

In this study, production of iron and cobalt based alloys by using metallothermic reduction was investigated. In experimental studies, mill scale and cobalt oxide (Co_3O_4) was reduced by aluminium. The aim of the experimental studies is to produce cobalt based ferroalloys which meet the quality standards obtained in the carbothermic method. High purity raw materials used in metallothermic experiments were supplied from Nanokar Company. Purity and grain size values of the raw materials are given in Table 1.

Table 1
Purity and grain size values of the raw materials which were used in the experiments.

Component	Purity, %	Grain Size
Co_3O_4	99.70	<38 μm
Al	99.50	<45 μm

Mill scale was used as iron source and its chemical analysis is given in Table 2. Mill scale contains 71.04% iron in total. 24.59% of total iron is Fe^{+2}, 42.80% of it is Fe^{+3} and 3.65% of it is Fe^0.

Table 2
Chemical analysis of mill scale (mass %).

Cu	SiO_2	Mn	Fe_2O_3	Fe_3O_4	Fe	Total Iron
0.75	0.39	0.14	24.59	42.80	3.65	71.04

2.2. Method

Although there are disadvantages of the process such as slight differences in the product due to some possible different reaction developments in each process and loss of the material used as reductant; metallothermic production technique was chosen because of its advantages such as minimum energy consumption (energy is only needed to start the reaction), completion of the process in a few seconds, no need for high-tech equipment and possibility of controlling the cooling stage.

This study, which was carried out for producing cobalt and iron-based alloys by metallothermic reduction method, is executed in three main steps. These steps are thermodynamic studies, reduction experiments and characterization studies. Flowchart of the studies is given in Fig. 1.

Metallothermic reduction is a simple and cost-effective method used in the production of high-tech ceramics, alloys and intermetallic materials. In this method, an exothermic reaction is triggered and then, this reaction self-propagates as a wave in the reactant mixture. For these events to take place, reaction must have a relatively high activation energy and generate extremely high heat [19]. This technique has many features that can be distinguished from conventional production methods. Some of these features are; high heat released as result of chemical reaction, high reaction rate (0.15 m/s) (enables the production of materials between 800 °C and 4500 °C without using an external heat source), high conversion rate of reactants to products, evaporation of impurities because of high reaction temperature [20]. In the metallothermic reduction process, the first heat is supplied to the reactant mixture to increase the heat to ignition temperature. Afterward, the reaction starts and proceeds spontaneously without the need of an external heat source. The heat generated in each reacting layer is transferred to the next unreacted layer, and this heat causes the reaction to take place in the unreacted layer. While metallothermic reaction leaves the reaction products behind, reaction starts in the unreacted mixture. The high amount of energy generated during the reaction affects the reaction rate in direct proportion. High reaction rate enables an economic and highly efficient production [21,22].

In the experimental studies, it was aimed to obtain Fe and Co by reducing mill scale and Co_3O_4 with aluminium. Chemical analysis of the mill scale shows that 100 g of mill scale contain 70.95 g of Fe. 3.56 g of iron is transferred into product without using aluminium (Eq. (1)). Molecule weight of Fe_2O_3 is 159.7 g and 2Fe formed in products are 112 g. Chemical analysis of mill scale shows that amount of Fe^{+3} is 42.8 g. According to Eq. (2), it is necessary to

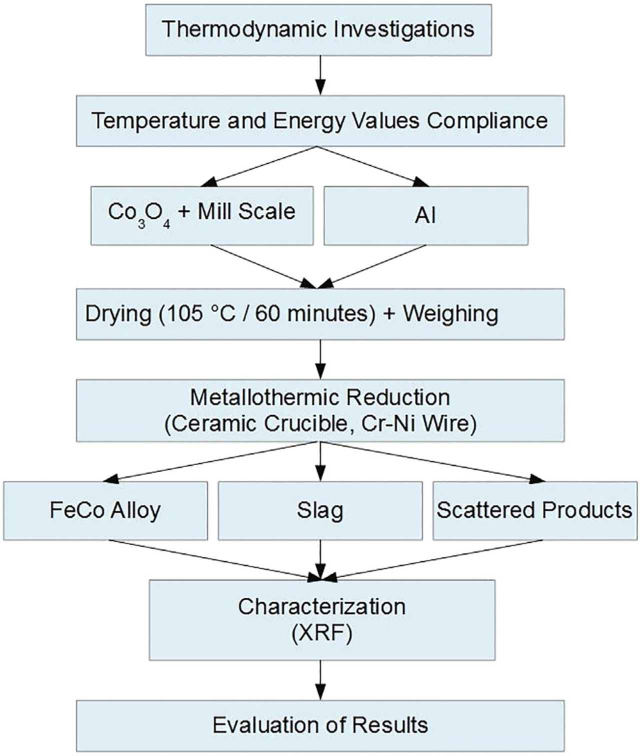

Fig. 1. Flowchart of the experimental studies.

use 61.19 g of Fe_2O_3 to obtain 42.8 g of Fe and it is required to use 20.69 g of Al for reduction of Fe_2O_3. Molecule weight of 3FeO is 215.55 g and weight of 3Fe formed in the products is 168 g. Chemical analysis of the mill scale shows that Fe^{+2}'s weight is 24.59 g and according to Eq. (3), it is required to use 31.63 g of FeO to obtain 24.59 g of Fe. Eq. (3) shows that 7.92 g of Al is needed for reduction of 3FeO. Co_3O_4 is added to alloy composition to obtain an alloy which includes 24% cobalt (Eq. (4)). Total aluminium quantity is calculated by adding the required Al amount for reduction of Co_3O_4. In the experiments, the amount of Al that can reduce the reactants was calculated as 100% stoichiometric Al ratio. Then, other reaction stoichiometry's was calculated from this ratio.

$$Fe \rightarrow Fe \tag{1}$$

$$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3 \tag{2}$$

$$3FeO + 2Al \rightarrow 3Fe + Al_2O_3 \tag{3}$$

$$3Co_3O_4 + 2Al \rightarrow 3Co + 4Al_2O_3 \tag{4}$$

Mill scales, Co_3O_4 and aluminum powders were weighed to prepare the mixture. The weighed raw materials were dried in an oven for one hour at 105 °C. Dried samples were mixed until homogeneous mixture was obtained, then resulting mixture was charged in a ceramic crucible. CrNi resistance wire, which was connected to power cable, was embedded inside the mixture. In order to reduce scattering during experiment, ceramic crucible was covered with a lid and a weight was placed on that lid. The electric current provided by the power supply was transferred to the resistance wire, and the energy released from the wire triggered the reaction. Power supply was turned off after the reaction started. The reaction was finished in a short time and the ceramic crucible was broken with metal tongs, then FeCo and slag were separated from each other. Fig. 2 presents the first, third, sixth and ninth second of the reaction (Table 3).

3. Characterization techniques

Thermoscientific Qualitative XL2 plus model XRF was used to determine the chemical composition of the alloys and slag. Raw materials were analyzed by using chemical analysis and atomic absorption spectrometry (AAS, Perkin Elmer Analyst 800) techniques. Brinell hardness values of the samples were measured via (QNESS, Q250C). Optical microscope micrographs were taken (Yamer).

4. Results and discussion

The metallothermic reduction of a metal occurs when the metal used as the reducing agent has higher oxygen affinity than the metal to be reduced. This evaluation can be done by examining the Ellingham Diagram of oxides. In the Ellingham Diagram, it is seen that calcium, aluminum and magnesium are at the bottom of the diagram, respectively. According to this data, all metal oxides can be reduced by these three elements. Energy generation is increased when oxygen affinity difference between the metal oxide and the reducing metal gets bigger. The increase of the energy facilitates the reaction to take place. Metals' oxygen affinity depend on the formation energy (ΔH) of the compounds they make with oxygen. Formation reaction enthalpy values of the main reactions which were used in this study were determined by HSC Chemistry 6.12 and results are given in Table 4.

Table 4 shows that the aluminium is suitable to reduce the metal contents of iron oxide and cobalt oxide. Adiabatic temperature and specific heat are two parameters that determines whether a metallothermic reaction takes place. The adiabatic temperature of the reaction must be above 1527 °C for the reaction to continue spontaneously. Specific heat is the energy value generated per gram of products. Specific heat value must be between 2250 J/g and 4500 J/g to have a controlled reaction and avoid scattering losses originating from explosion. If the specific heat value is below 2250 J/g, metal-slag separation, due to the density difference, can't take place. If the specific heat value is greater than 4500 J/g, a high amount of metal loss occurs [23–26].

Fig. 2. Propagation of metallothermic production experiments.

Table 3
Compositions of raw material mixtures which were used in metallothermic reduction experiments.

Experiment	Mill Scale, g	Al, g	Co_3O_4, g	Al Stoichiometry, %
1	100	26.57	37.65	95
2	100	27.97	37.65	100
3	100	29.36	37.65	105
4	100	30.76	37.65	110
5	100	32.16	37.65	115

Table 4
Standard formation enthalpy values of reactions which were utilized in experimental studies.

Reaction	ΔH^0_{rxn}, kJ
$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3$	−852.7
$3FeO + 2Al \rightarrow 3Fe + Al_2O_3$	−873.9
3Co3O4 + 8Al → 9Co + $4Al_2O_3$	−3972.7

Table 5
Adiabatic temperature and specific heat values of reactions which were utilized in experimental studies.

Reaction	T_{ad}, °C	Specific Heat, j/g
$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3$	2851.3	−3990.1
$3FeO + 2Al \rightarrow 3Fe + Al_2O_3$	2659.5	−3254.7
$3Co_3O_4 + 8Al \rightarrow 9Co + 4Al_2O_3$	2923.2	−4233.1

When Table 5 is evaluated, it is seen that all adiabatic temperature values are greater than 1527 °C (1800 K) and all specific heat values are between 2250 J/g and 4500 J/g. Accordingly, it was determined that these reactions can proceed spontaneously without problems.

After adiabatic temperatures and specific heat values were determined, phases that are probable to form with changing aluminium stoichiometry were modeled. At this stage, the equilibrium mode of FactSage and Equilibrium Compositions Module of HSC software which works with the Gibbs free energy minimization were utilized. These software show stable phases that will probably form at the simulated pressure and temperature values. The result of the simulation that shows the reduction of mill scale by aluminium is given in Fig. 3; the result of the simulation shows the reduction of mill scale and cobalt by aluminium is given in Fig. 4.

According to Fig. 3, iron can be obtained at every step of adiabatic system, it is also seen that aluminium which passed to the slag formed again in a new structure. This shows that energy values of the reaction are changed with increasing aluminium addition and this situation can have a positive or negative effect on the efficiency of the reaction.

After reduction conditions of mill scale were modeled with FactSage 6.4, effects of Co_3O_4 addition in the charge were modeled by HSC 6.12 database. According to Fig. 4, Fe converted from oxide form to metallic form with increasing Al addition. $FeAl_2O_4$ phase, which formed at low Al stoichiometry, did not form at high aluminium stoichiometries. When the formation of cobalt under these conditions is examined, it is seen that cobalt formation decreased with increasing aluminium stoichiometry, but a sufficient amount of cobalt was formed at every composition.

In the experiments carried out to synthesize FeCo, mill scale was used as iron source, Co_3O_4 was used as cobalt source and alu-

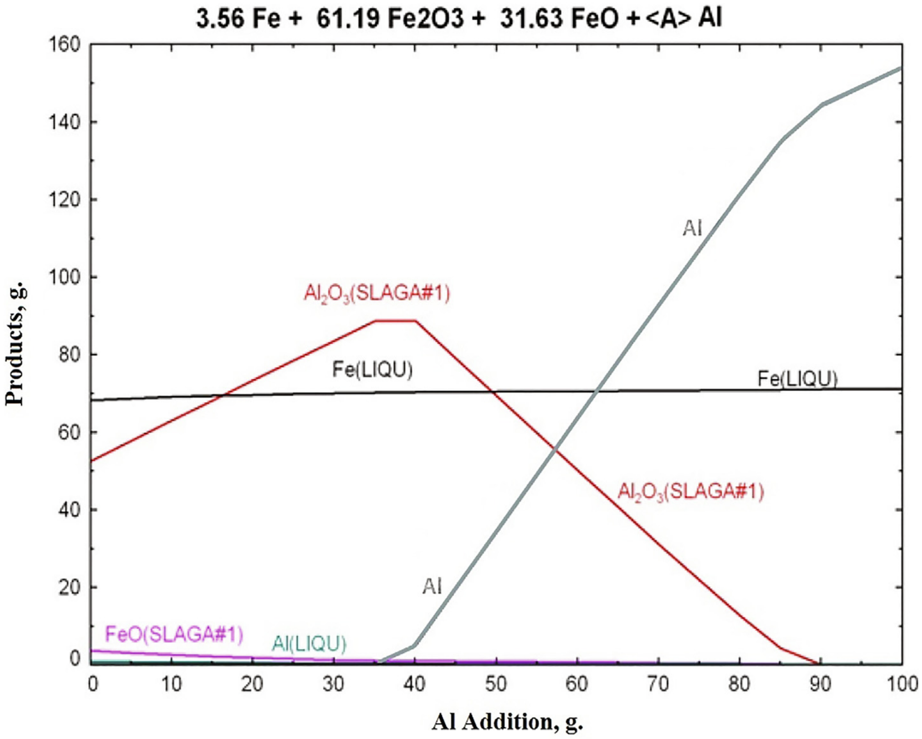

Fig. 3. Effect of Al stoichiometry change on the formation of possible phases in mill scale reduction.

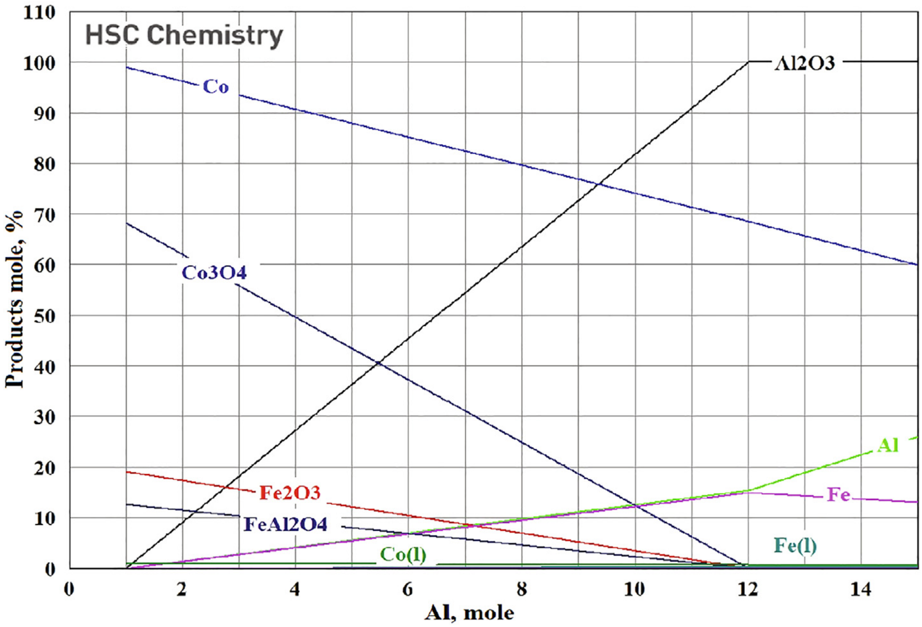

Fig. 4. Effect of Al stoichiometry change on the formation of possible phases in mill scale and cobalt reduction.

Table 6
XRF analyses of metal and slag parts of samples produced using different aluminum stoichiometries.

Al Sto. wt%	Metal, g				Slag, g				
	Fe	Co	Al	Cr	Fe	Co	Al	Cr	Bal.
95	51.74	29.49	17.62	0.14	10.10	4.77	10.82	0.04	52.54
100	55.22	38.75	0.29	0.12	3.92	1.31	11.87	0.03	74.74
105	56.57	40.52	0.68	0.13	9.78	4.77	4.99	0.06	57.27
110	55.38	37.10	0.55	0.21	3.01	0.75	5.27	0.01	83.31
115	54.25	37.79	0.55	0.22	4.01	1.64	8.05	LOD	74.79

minium was used as reductant, and effects of change in aluminium stoichiometry on FeCo recovery rate were investigated. In this study, it was aimed to produce an alloy that includes 24% Co and 76% Fe. Samples which were containing 95-100-105-110-115% stoichiometric aluminium were prepared. Each of these samples was 100 g. Samples were mixed and then they were dried at 105 °C for 60 min. Dried mixture was charged to the ceramic crucible where reaction was carried out by using a variac. Metallic sample and slag obtained as result of the experiment was analyzed by XRF technique and consistent results were observed. Chemical analysis results of metal samples and slags are given in Table 6. Metal recovery yields were calculated with Eq. (5) based on the data obtained from XRF analyses. Metal recovery yields are given in Fig. 5.

$$\text{MetalRecovery} = 100 \times \frac{\text{Theweightofmetalaftermetallothermicprocess(g)}}{\text{Theweightofaspectmetalaftermetallothermicprocess(g)}} \quad (5)$$

It is seen in Fig. 5 that Fe and Co recovery ratios increased with the increase of aluminium stoichiometry from 95% to 105%. According to thermodynamic data, adiabatic temperature and specific heat of the reaction decreased with aluminium stoichiometry increase and that affected the reduction yield negatively. The highest Fe and Co recovery ratios were obtained at %105 aluminium stoichiometry and these values were 81.25% and 93.78%, respectively. As it can be seen in Table 6, iron and cobalt amount in the metal increased until aluminium stoichiometry increased to 105%. With the further increase of aluminum stoichiometry, the amount of iron and cobalt in the metal decreased. Thermodynamic modeling of Fe and Co formation quantities is given in Fig. 4. It is seen in the figure that Fe and Co formation decreased and metallic Al is formed with increasing addition of Al. Al_2O_3 which is formed in the slag is reduced by aluminium, and conse-

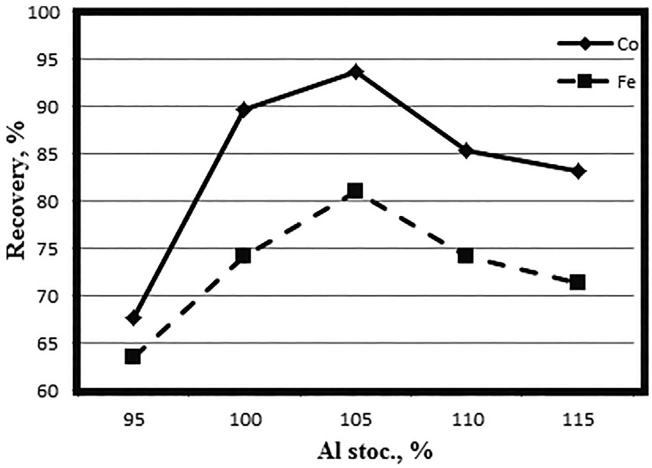

Fig. 5. Metal recovery yields of Fe and Co with the increase in the aluminium stoichiometry.

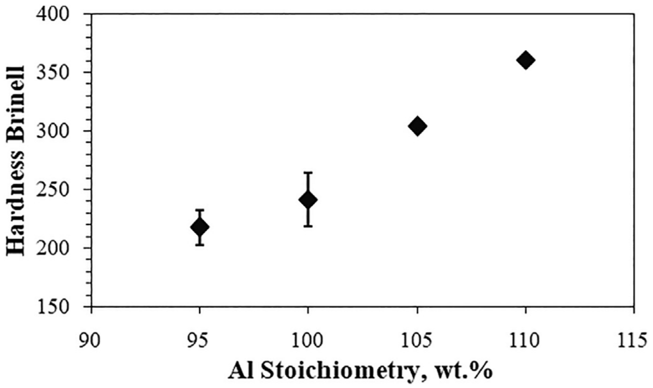

Fig. 6. The change of hardness values of obtained FeCo phases with the increase in the aluminium stoichiometry.

Table 7
The change of Brinell hardness values of obtained FeCo phases with the increase in the aluminium stoichiometry.

	Al Stoichiometry, wt%			
	95	100	105	110
Hardness (HB)	217.25	241.25	303.50	360.25
Standard deviation	15.31	22.52	3.79	1.89

quently the reaction energy decreased. This explains the decrease in Fe and Co efficiency when aluminum stoichiometry exceeds 105%.

When Table 6 is examined, it is seen that Fe and Co can be reduced with aluminothermic reduction, but target alloy (Permendur 24) composition could not be reached in the experimental studies. It is thought that sufficient energy cannot be produced when aluminum is used as a reductant. It is anticipated that the target alloy can be achieved by using magnesium or magnesium together with aluminum as the reductant.

Obtained FeCo alloys (from 95% Al stoichiometry to 110%) in the experiments were polished through metallographic sample preparation techniques. 320 grits to 2500 grits polishing papers and 3 μm diamond-containing polishing solution were used respectively. The sample obtained in the experiment with 115% Al stoichiometry was not used for further tests because the reduction system already reached the equilibrium at 105% Al stoichiometry. Therefore, it was predicted that metallographic investigation of samples up to 110% Al stoichiometry would be enough. Brinell hardness values of the samples were measured (QNESS, Q250C) and the results were given in Fig. 6 and in Table 7 with standard deviation values. Hardness values continuously increased from 217.25 HB (95% Al sto.) to 360.25 HB (110% Al sto.). All samples were etched by using 5 vol% nital solution for 5 s and their optical microscope micrographs were taken (Yamer). Micrographs at 200x magnification were shared in Fig. 7. As seen in the figure, 95% stoichiometric Al ratio was insufficient to reduce reactants. So, there were Al-based precipitates in the micrograph (A). In the other Al stoichiometric ratios, reactions were completely realized and, with the increase in the Al stoichiometry, Fe-Co-Al precipitates occurred as a secondary phase. Therefore, it was predicted that the increase in the secondary phase formation increased the hardness values.

5. Conclusion

In this study which was carried out with the aim of the production of FeCo, mill scale and Co_3O_4 powders were used as raw materials and, Al powders were used to reduce the metal contents of those raw materials. The effects of the use of aluminium in different ratios on total metal recovery yield were investigated. FactSage 6.4 and HSC 6.12 databases were used in thermochemical simulations. Heat and energy values which are generated by the reactions were modeled by using those software. When obtained data was evaluated, it was seen that these reactions occur spontaneously in a controlled manner. In metallothermic experiments, mixtures having aluminum stoichiometries ranging from 95% to 115% were prepared and, the effects of changing aluminium stoichiometry on

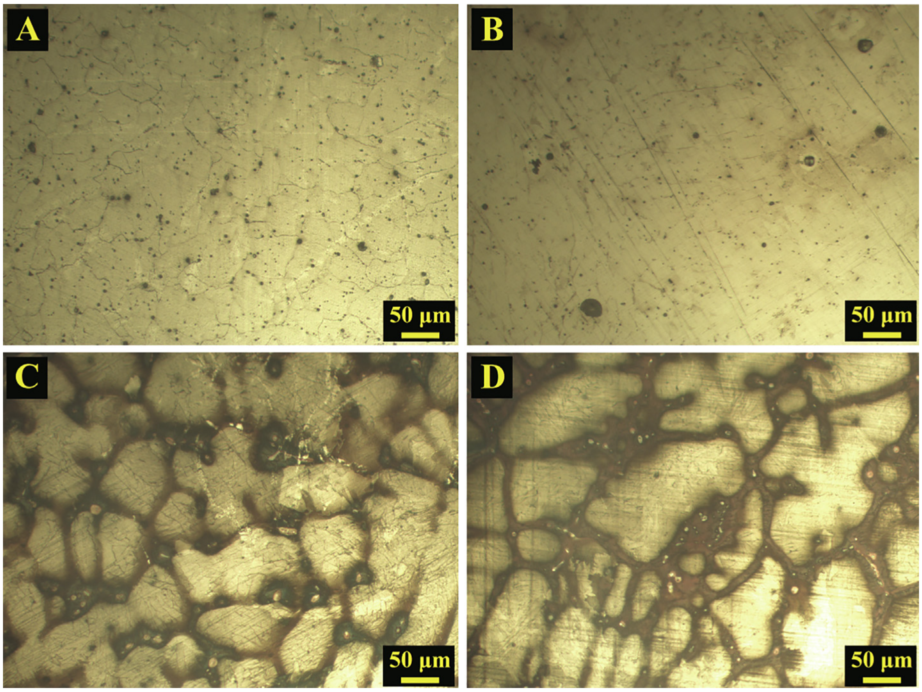

Fig. 7. Optical microscopy micrographs of obtained FeCo phases at 200x magnification (A: 95% Al sto., B: 100% Al sto., C: 105% Al sto., D: 110% Al sto.).

metal recovery yield and product compositions were investigated. As a result of the experimental studies, it was seen that highest metal recovery yields were obtained as 81.25% for iron and 93.78% for cobalt with the mixture including 105% stoichiometric aluminium. Also, it was seen that hardness values increased from 217.25 HB to 360.25 HB with increasing aluminium stoichiometry, because of the Fe-Co-Al precipitates occurred as a secondary phase.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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