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Production of Al-Co-Ni Ternary Alloys by the SHS Method for Use in Nickel Based Superalloys Manufacturing

Abstract: In this study, Al-Co-Ni ternary alloys were synthesized, in order to obtain low-cost starting material for Ni-based superalloy production, by a self-propagating high temperature synthesis (SHS) both under normal gravity conditions ($a = 9.81$ m/s^2) and under high gravity conditions (up to 1000 g-force) by using a centrifugal machine. The mixture of Co_3O_4-NiO powder were reduced by Al powder for the production of SHS alloys with the estimated compositions of 5–10 mass% Al, 20–65 mass% Co, 25–75 mass% Ni. The effect of green mixture compositions and centrifugal overload on combustion temperature, alloy/slag separations, chemical composition and microstructure of final alloys were investigated. The chemical analysis results showed that production of SHS alloys were achieved by having up to 86.12% of Co and 92.32% of Ni recoveries. The highest metal recovery value was obtained in SHS alloy with the estimated composition of 10%Al-65%Co-25%Ni by the addition of 20% Al_2O_3 into the green mixture. The metal/slag separation efficiency increased by increasing the centrifugal overload.

Keywords: Ni-based superalloys, Al-Co-Ni ternary system, SHS, centrifugal overload

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1 Introduction

The superalloys are used at elevated temperature applications due to their resistance to environmental attack, fatigue, creep and corrosion. Ni-based superalloys are the most common superalloys used in the industrial applications. Ni-based superalloys contain several alloying elements such as Fe, Co, Cr, Al, Mo, Ti, etc. and each of them has a special role for enhancing alloy properties. Addition of Co makes an increase in solid-state strength and sulfidation resistance, whereas addition of Al increases oxidation resistance and precipitation hardening [1–4].

The Al-Co-Ni ternary alloy system is one of the most commonly used alloy systems both in industrial usage and in scientific studies since 1989 due to their structural and mechanical properties. The investigations on the structural properties were mainly focused on B2 type ordered crystal structure, quasicrystalline phase, decagonal phase and their modifications. In the previous studies, the ternary alloys were produced by melting the mixtures of high purity metal powders in a close system furnace under an inert atmosphere such as argon, and their mechanical and structural properties were also examined [5–9].

The earliest study about self-propagating high-temperature synthesis (SHS) technique which was a simple, low cost and low energy required process has been published by Merzhanov, Borovinskaya and Shkiro in 1967, described as the production of refractory inorganic materials from powder mixtures of a metal with nonmetal such as C, B or etc., by thermal explosion with combustion wave occurred during the exothermic reaction. During the last 50 years, all known structural ceramics, metal alloys, composite materials and intermetallic compounds were produced by SHS techniques both under normal atmospheric condition and under controlled atmosphere (inert, vacuum, high or low pressure, artificial gravity conditions). SHS production method is a mode of combustion process and first step of the process can be started with initiation of the powder mixtures by using different techniques (such as flux ignition, laser ignition, heated gas, heating coil, furnace, etc.) to produce a combustion wave. The synthesizing process begins when the combustion wave reaches heat release zone in nonequilibrium structure or the synthesis zone in the equilibrium structure. During SHS reactions, reaction temperature can reach up to 4000 °C, propagation velocity of the combustion wave can be between 0.1–15.0 cm/s and heating rate can be up to 10^6 °C/s. Products are obtained after solidification and final structuration stages [10–21].

Some of the SHS processes can be affected by gravity, and some of them such as NiO/Al/Ni powder mixture system are highly sensitive to gravity force depending on the reaction mechanism in the combustion wave. Sensitivity to gravity force is high in the system where there is one or more melted reactants and even higher with melted products. The first published study about the effect of high artificial gravity on a gasless combustion process was reported in 1968 by Serkov, Maksimov and Merzhanov where an iron-aluminum thermite system was investigated under the effect of centrifugal force accelerated up to 900 g-force (1 g-force a = 9.81 m/s^2). The effects of artificial gravity force obtained by centrifugal force on the heating rate, propagation velocity of the combustion wave, chemical and phase compositions, macro- and microstructures of the products have been investigated [22–27].

However being studied for 25 years research about Al-Co-Ni ternary alloy system by SHS method is insufficient. On the other hand binary alloy systems of these alloys especially Ni-Al systems were commonly studied via SHS methods using either metallic powder or metal oxide powder mixtures under both normal atmospheric and high artificial gravity conditions [17, 18, 22–26, 28–30].

In this study, cobalt and nickel rich Al-Co-Ni ternary alloys with different compositions were produced by aluminothermic SHS process starting from metal oxide powder mixtures. The effects of initial mixture composition and artificial gravity forces on the metallic recoveries, final alloy compositions, and phase separations were also examined.

2 Experimental

The raw materials used in the SHS experiments were Co_3O_4 (99.70% purity with 38 μm average particle size), NiO (99.00% purity with 45 μm average particle size), Al (99.5% purity and 200 μm average particle size) and Al_2O_3 (99.98% purity with 2 μm average particle size) were supplied from Alfa Aesar. The estimated alloy compositions and the amounts of the raw materials were given in Table 1. The estimated Al composition in the final mixture was fixed as 10 mass% for batch-type reaction under atmospheric gravity, and as 5 mass% for centrifugal experiment under high artificial gravity conditions.

The amount of components in initial mixtures prepared from dried powders were calculated to produce the estimated alloy compositions. The powder mixtures (150 g) were charged into a crucible and compacted after they were mixed thoroughly for 15 minutes in a turbula mixer. A copper crucible was used in batch-type SHS reactions under normal gravity conditions while C/SiC composite crucible was used in centrifugal systems under high gravity conditions. The schematic views of the experimental setups for the centrifugal system was given in Figure 1.

Copper crucible was designed as two pieces; a base plate ($d_{in} = \varnothing$ 40 mm $d_{out} = \varnothing$ 50 mm) and a cylindrical part (h = 140 mm, $d_{in} = \varnothing$ 40 mm $d_{out} = \varnothing$ 50 mm) fitted with each other (Fig. 1, a-5). The rotation velocity of the rotor (Fig. 1, b-4) provides centrifugal acceleration for desired artificial gravity force from 1 to 1000 g-force. An equal amount of counter-weight was placed at the other side of the rotor in order to make the gravity force vector parallel to the direction of the combustion wave propagation. A resistance wire was placed at the top of the crucible and the reaction realized by passing current through the wire. After initiation the resistance wire was taken

Table 1: Estimated alloy compositions and amounts of raw materials used in SHS reactions

RUN	Estimated alloy composition (mass%)			Weight of raw materials (g)		
	Al	Co	Ni	Al	Co_3O_4	NiO
1	10	65	25	40.23	80.75	29.02
2	10	40	50	39.07	51.17	59.76
3	10	20	70	38.09	26.21	85.70
4	5	20	75	34.12	25.73	90.15

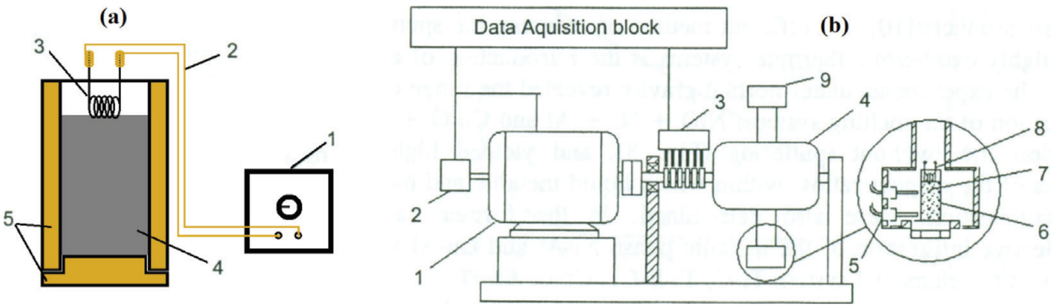

Fig. 1: The schematic views of SHS experimental setup. a) batch-type system: (1) power supply, (2) Cu cable, (3) igniter, (4) green mixture, (5) Cu crucible; b) centrifugal system: (1) electrical motor, (2) tachometer, (3) collector, (4) rotor, (5) photodiodes, (6) C/SiC crucible, (7) green mixture, (8) igniter, (9) counter-weight

out from the system and a highly exothermic reaction became self-sustaining and propagated throughout the SHS mixture. The obtained SHS products were discharged from the crucible after cooling.

The phase compositions of the SHS products were characterized by X-ray diffractometer (PANalytical PW3040/60, Cu Kα radiation) equipment with X'Pert HighScore+ software and ICDD, ICSD databases. The morphologies of the products were characterized by scanning electron microscope (SEM, JOEL JCXA-733) with EDS (Energy Dispersive Spectrometer). Wet chemical analysis were realized by using Atomic Absorption Spectrometer (AAS, Perkin Elmer Analyst 800).

3 Results and discussion

3.1 Thermodynamic simulation results

Before the SHS experiments, a thermochemical simulation was performed to estimate the possible product compositions obtained with different initial ratios of Co_3O_4, NiO and Al. Calculations were made by using the advanced "Equilib" module of FactSage 6.3 and estimated final products under 1 atm pressure at the adiabatic temperature were given in Figure 2.

In Figure 2, Al composition was fixed as 10 mass%, and amounts of products were given due to the estimated

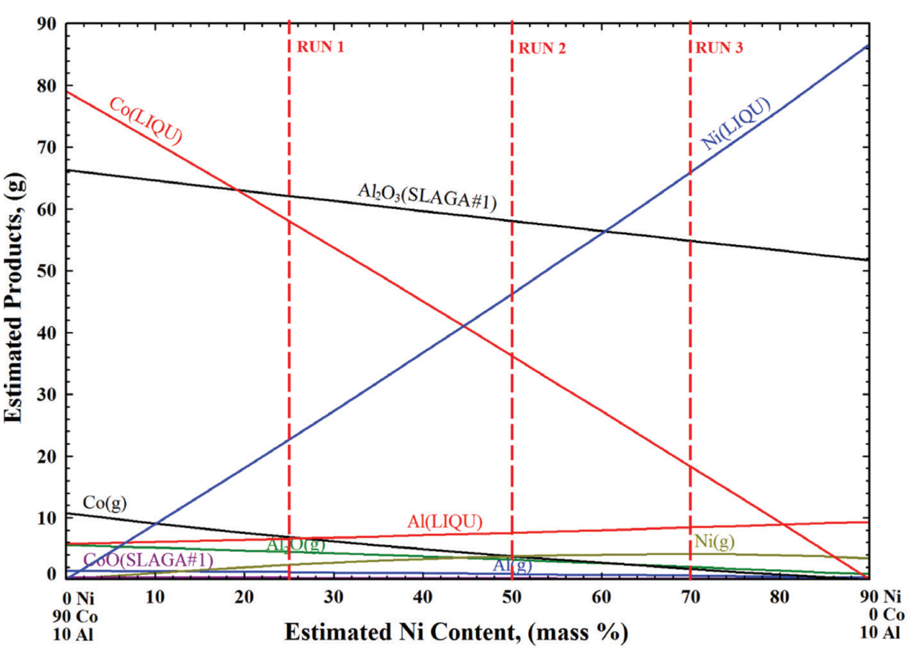

Fig. 2: Estimated final products of SHS reactions with changing in estimated Ni contents in the final alloy while Al content fixed as 10 mass% [31]

Ni mass% in the final alloy compositions. Due to the high adiabatic temperature, Co and Ni losses can be expected as volatile Co and Ni gases. Al losses are also expected as volatile Al_2O, and Al gases. Therefore, there would be an expected lack of Al mass percentage in the final alloy due to the volatile Al compounds. Theoretically, while the estimated Co and Ni contents could be achieved without any additives, Al content in the final product became less due to the volatilization. Calculated compositions of Al was 7.50 mass% in RUN1; 8.38 mass% in RUN2; and 9.08 mass% in RUN3, respectively [31].

The adiabatic temperatures of SHS reactions and total amounts of theoretical volatilized products with different amounts of Al_2O_3 additions were given in Figure 3. It was clear that, between 3–11% of the initial mixtures volatilization of materials would be obtained due to the high adiabatic reaction temperature in SHS reactions without any Al_2O_3 addition.

Estimated composition was obtained by the introduction of additives (like Al_2O_3) into the green mixture to reduce the adiabatic reaction temperatures and thus amount of total volatilized materials. There were no any loss observed due to the volatilized materials when 20 mass% of Al_2O_3 was added into the green mixture. Thus, 30 g of Al_2O_3 powder (20 mass%) was added into the green mixtures for the subsequent experiments.

3.2 Batch-type SHS results (normal gravity conditions)

The weight, total metal recovery and scattered ratios of the products obtained in normal gravity conditions with 20 mass% Al_2O_3 added were given in Table 2. It was seen that, the weights of alloys and total metal recovery values slightly increased by increasing in NiO/Co_3O_4 ratio in the green mixtures (Ni/Co ratio in the final alloy). This increase was achieved by the decreases in both adiabatic reaction temperature (Figure 2).

The effect of Al_2O_3 addition was investigated by the addition of, 0–10–20 mass% of Al_2O_3 into the green mixture, and the results of the reactions were given in Table 3. Higher adiabatic reaction temperature causes not only the

Table 2: Weight of products, total metal recoveries and scattered ratios of SHS reactions by the addition of 20 mass% Al_2O_3 into the green mixtures

	RUN1	RUN2	RUN3
Alloy weight (g)	83.2	85.7	87.8
Slag weight (g)	95.2	91.0	85.0
Total metal recovery (%)	91.95	91.86	92.52
Scattered ratio (%)	0.90	1.85	4.03

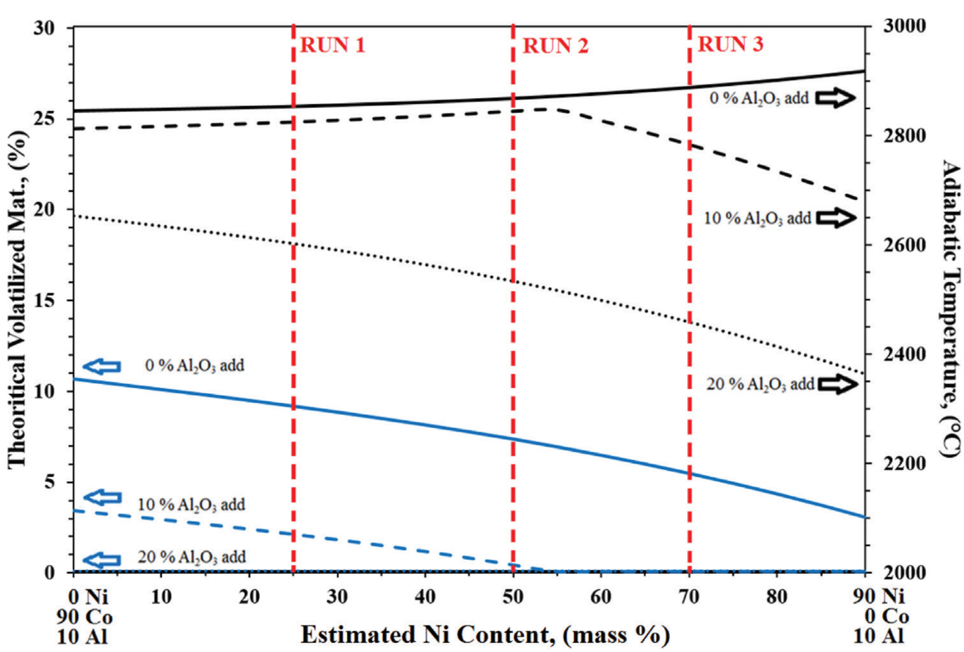

Fig. 3: Simulation results of the adiabatic temperature and estimated volatilized products changes with changing in Ni contents of final alloys while Al content fixed as 10 mass% [31]

Table 3: Effect of different Al_2O_3 additions on weight of products, total metal recoveries and scattered ratios of SHS reactions for estimated composition of 10Al-40Co-50Ni mass% (RUN2)

	0 mass%	10 mass%	20 mass%
Alloy weight (g)	39.5	66.6	85.7
Slag weight (g)	42.7	65.1	91.0
Total metal recovery (%)	44.96	72.00	91.86
Scattered ratio (%)	45.23	20.19	1.85

increase of volatilized materials but also more scattered materials. Without any Al_2O_3 addition, nearly half of the initial mixture was lost as scattered and volatilized materials due to the highly exothermic reaction. The highest recovery was obtained by the addition of 20 mass% of Al_2O_3. Other positive effect of Al_2O_3 addition was to increase the duration of the chemical reactions. Thus controlled reactions resulted in the decrease in scattering.

The chemical analysis of SHS alloys and slags were given in Table 4. Produced alloy compositions were corresponded with the estimated alloy compositions, and total metallic Co, Ni loss into the slags were measured below 3.5 mass%. It would be possible to produce SHS alloys with the desired compositions over 90% efficiency by preparing the accurate initial mixtures. The chemical compositions of SHS alloys by mass were 11.10% Al, 62.60% Co, 25.37% Ni, 0.24% Cr, 0.16% Cu, 0.41% Fe, 0.12% Si in RUN1; 12.37% Al, 39.03% Co, 47.61% Ni, 0.27% Cr, 0.18% Cu, 0.43% Fe, 0.12% Si in RUN2; 10.34% Al, 19.34% Co, 69.38% Ni, 0.27% Cr, 0.09% Cu, 0.45% Fe, 0.13% Si in RUN3, respectively. Total impurity content measured less than 1.0 mass were originated from the melting of the Ni-Cr resistance wire and Cu cable during the process while Fe and Si were originated from NiO and Al as raw materials.

The distribution ratios of metals among the alloy, slag and scattered part were calculated by using Eq. (1) while the distribution of metals in alloys was given in Figure 4.

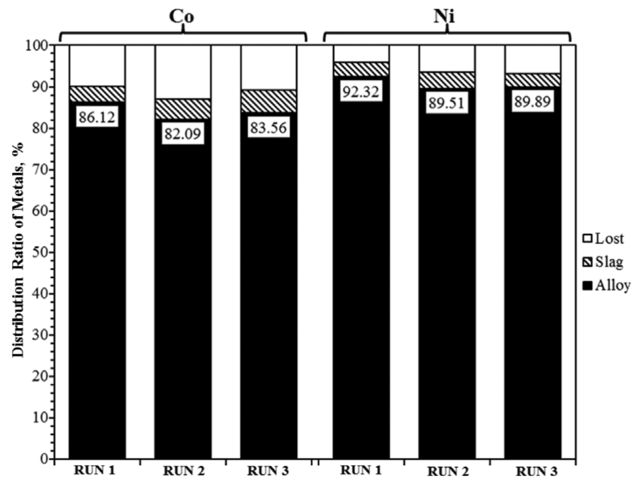

Fig. 4: The distribution ratios of Co and Ni among alloy, slag and scattered parts obtained by 20% Al_2O_3 addition to batch type SHS reactions under normal gravity conditions

$$[D_{Me}] \text{ or } (D_{Me}) = \frac{[\%\,\text{Me}] \times \text{weight of alloy} \quad \text{or} \quad (\%\,\text{Me}) \times \text{weight of alloy}}{\text{wt.\% Me in initial mix} \times \text{weight of initial mix}} \tag{1}$$

where

$[D_{Me}]$: Distribution ratios of metal in alloy
(D_{Me}): Distribution ratios of metal in slag

As seen in Figure 4, higher Ni recovery values were measured comparing to Co recovery values; because total reduction of NiO into metallic Ni was more favorable than total reduction of Co_3O_4 into metallic Co and also the heat of vaporization of Ni (379 kJ/mol) was a bit higher than Co (377 kJ/mol). The highest Co and Ni metal recoveries were obtained as 86.12% and 92.32% in RUN1 (estimated composition as 10% Al, 65% Co, 25% Ni) where the lowest scattered ratio was obtained with 20% Al_2O_3 addition by weight, respectively.

Table 4: Chemical analysis results of obtained SHS alloys under normal gravity conditions by the addition of 20 mass% Al_2O_3 into the green mixtures

RUN	Estimated alloy comp. (mass%)			Alloy (mass%)				Slag (mass%)	
	Al	Co	Ni	Al	Co	Ni	Impurities	Co	Ni
1	10.0	65.0	25.0	11.10	62.60	25.37	0.93	2.15	0.73
2	10.0	40.0	50.0	12.37	39.03	47.61	0.99	1.83	1.68
3	10.0	20.0	70.0	10.34	19.34	69.38	0.94	1.14	2.23

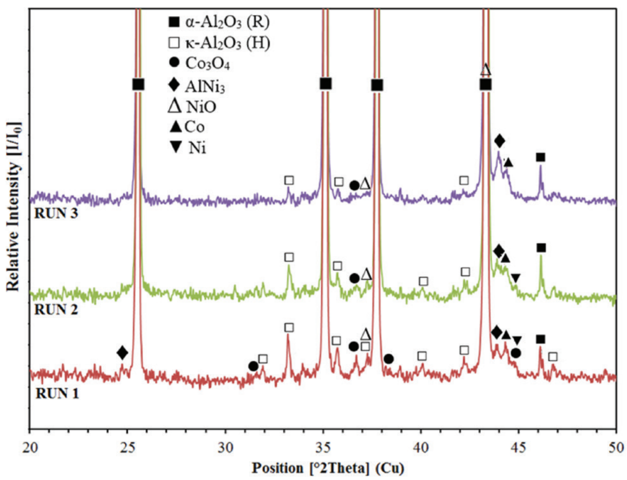

Fig. 5: The comparative XRD results of slags obtained by 20% Al_2O_3 addition to batch type SHS reactions under normal gravity conditions

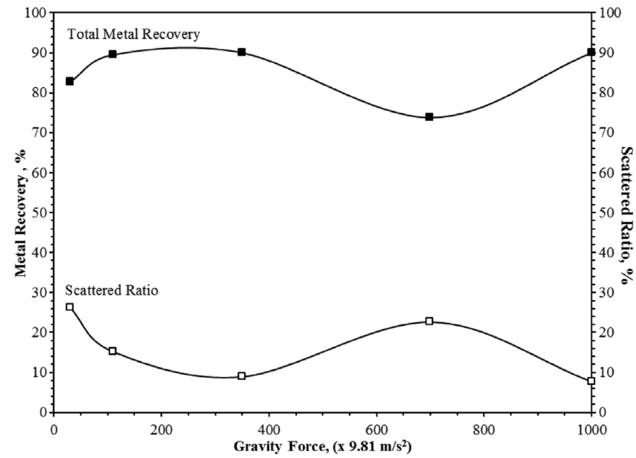

Fig. 6: Effect of gravitational force on total metal recovery and scattered ratio of SHS alloy for estimated composition of 5Al-20Co-75Ni mass%

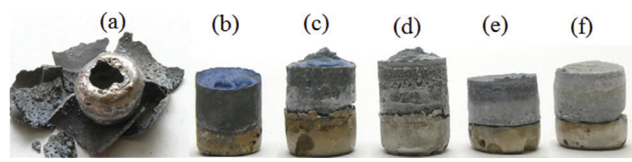

Fig. 7: View of the obtained metal and oxide phases for estimated composition of 5Al-20Co-75Ni mass% under a) 1 g-force, b) 30 g-force, c) 110 g-force, d) 350 g-force, e) 700 g-force, f) 1000 g-force of artificial gravity conditions

The comparative XRD results of the slags were given in Figure 5. Al_2O_3 was found as the major phase in the slags. Due to the rapid solidification, Co and Ni losses into slags were found as both metallic (as $AlNi_3$, Ni, Co) and oxide (NiO, $CoO.Co_2O_3$, and $CoO.Al_2O_3$) forms. As maximum intensity values of XRD peaks for $CoO.Co_2O_3$ (36.724°) and $CoO.Al_2O_3$ (36.740°) were similar, d-spacing values of the phases were considered to identify the possible phases. Furthermore, as relative intensity of the detected peak was only 0.70%. It was difficult to decide which phase was feasible. It is assumed that $CoO.Co_2O_3$ phase might be more feasible than $CoO.Al_2O_3$, because d-spacing value of the highest intensity peak [3,1,1] of $CoO.Co_2O_3$ (2.445A°) more closed to the detected peak (2.448A°) than d-spacing value of the highest intensity peak of $CoO.Co_2O_3$ (2.444A°).

3.3 High gravity SHS results

SHS alloys with estimated alloy compositions of 5 mass% Al, 20 mass% Co, 75 mass% Ni (RUN4) were carried out under artificial gravity conditions. Total metal recovery and scattered ratio values changing with different artificial gravity forces realized in the centrifugal system were given in in Figure 6. The metal recovery increased with increasing in gravity force, but there was a decrease under 700 g-forces due to the increase in the scattered material. The highest metal recovery with 90.0% and the lowest scattered ratio with 7.7% were obtained under applying 1000 g-forces artificial gravity. Although there was no Al_2O_3 addition into the green mixture, the lowest ratio of the total scattered material was measured as 7.7%. The effects of Al_2O_3 addition into the green mixture when the under artificial gravity conditions were applied will be the subject of other work.

The visual appearance of the products obtained at different artificial gravitational forces was given in Figure 7. It can be seen that the separation of metal and oxide phase was increased with increase in gravity forces. The obtained products under 350, 700 and 1000 g-forces were shown readily separated metal and oxide layers. Furthermore increase in artificial gravity force resulted in the formation of both metallic and oxide phases denser.

The comparative XRD analysis of obtained slags was given in Figure 8. As the artificial gravity force was increased, metallic ($AlNi_3$, Co, Ni) leakage into the slag were decreased and only small amount of Co and NiO phases were detected under 1000 g-force. One of the high temperature stable spinel, $CoAl_2O_4$, formed instead of Co_3O_4 when artificial gravity force was increased. Change in slag composition for different artificial gravity forces will be the subject of other study.

The back scattered electron (BSE) images of SHS alloys obtained under different artificial gravitational forces were given in Figure 9, while SEM/EDS analysis results for selected areas were given in Table 5.

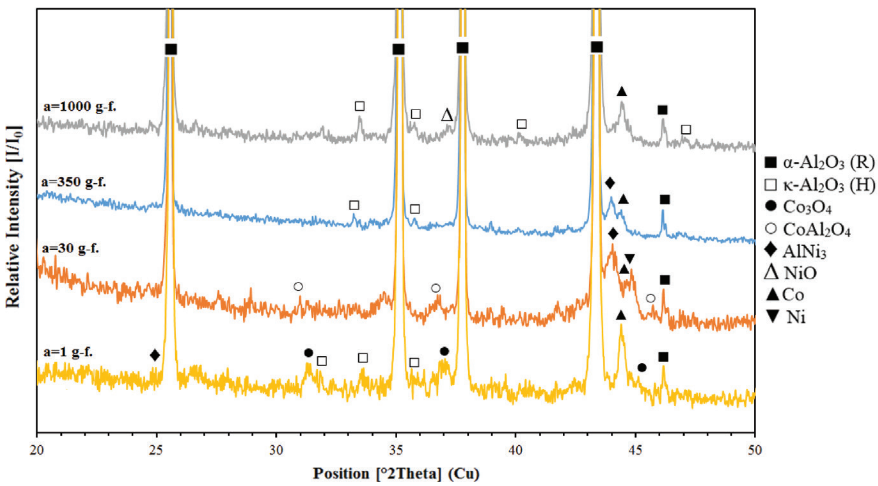

Fig. 8: The comparative XRD results of slags obtained for estimated composition of 5Al-20Co-75Ni mass% under high artificial gravity conditions

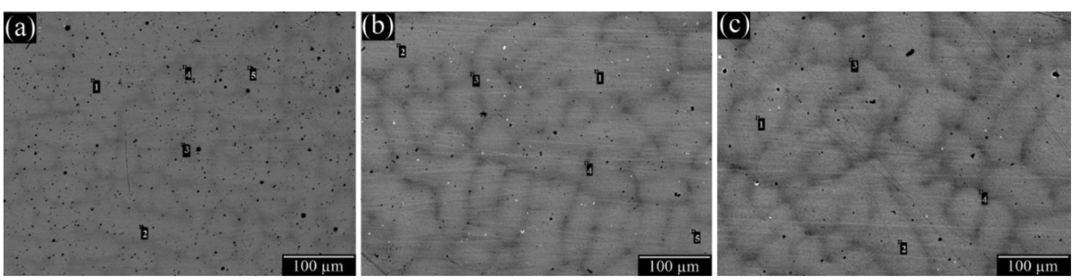

Fig. 9: Back scattered electron images of SHS alloys with 5Al-20Co-75Ni estimated composition under a) 1 g-force, b) 110 g-force, c) 1000 g-force of artificial gravity conditions

Table 5. SEM/EDS analysis results of obtained SHS alloys under high artificial gravity conditions for estimated composition of 5Al-20Co-75Ni mass%

Gravity force	Area	Alloy composition (mass%)					
		C	O	Al	Si	Co	Ni
a = 1 g	1	1.94	0.00	5.88	0.53	17.93	73.72
	2	2.49	0.00	5.80	0.45	17.59	73.67
	3	8.79	0.63	6.05	1.24	15.05	68.25
	4	3.96	0.00	6.27	1.21	15.34	73.20
	5	5.46	46.34	41.38	0.00	1.39	5.43
a = 110 g	1	2.12	0.00	5.29	1.07	17.53	73.99
	2	2.71	0.00	5.33	1.02	17.87	73.07
	3	2.90	0.00	5.39	5.39	12.11	74.21
	4	2.14	0.00	5.48	5.50	11.49	75.39
	5	18.70	29.22	1.41	27.01	4.52	19.13
a = 1000 g	1	2.48	0.00	4.81	2.08	16.69	73.94
	2	2.87	0.00	4.63	1.27	17.79	73.43
	3	3.16	0.00	4.68	3.72	14.41	74.02
	4	3.29	0.00	4.53	4.34	13.57	74.27

The visibility of the grain boundaries increased with increasing in applying artificial gravitational forces whereas, the formation density of the impurities reduced with increasing in applying artificial gravitational forces. Thus, the effect of artificial gravity force increased both product capacity and its quality. Ni compositions were seemed to be homogenous between the selected areas. It can also be seen that, the solubility of C and Si into the alloy was increased with increasing in gravity forces. The effect of artificial gravity was observed deeply in the chemical conversion for C and mostly Si that lead to increase of Si content in the final alloy. The source of the C and Si into the metal was utilized C/SiC composite crucible. High amount of C and Si contents in some SHS alloys resulted in lower Co and Ni contents than expected.

4 Conclusion

On the basis of the presented results of the SHS process to synthesize Al-Co-Ni ternary alloys by using batch-type and centrifugal systems, the following conclusions can be made:

Thermodynamic studies showed that the reduction of NiO to Ni could be realized more easily than the reduction of Co_3O_4 to Co. Al_2O_3 addition into the green mixture reduced both the adiabatic reaction temperature and volatilized materials while increasing the duration of the chemical reactions. In the batch-type SHS experiments containing 20 mass% Al_2O_3 in the green mixture, the maximum metal recoveries were obtained as 86.12% for Co and 92.32% for Ni, whereas SHS alloys were synthesized with the composition of 11.10 mass% Al, 62.60 mass% Co, 25.37 mass% Ni. Results of the chemical analysis showed that, it is possible to obtain different alloy compositions which are suitable for using as master alloy in the manufacturing of Ni-based superalloy, by the determination of initial green mixture composition in SHS processes.

In the centrifugal SHS experiments, the maximum metal recovery was obtained as 90.00% under 1000 g-force artificial gravity, while the minimum scattered material was achieved as 7.70% without using any additives. The metal/slag separation increased by increasing the gravitational force which resulted in the formation of denser products. The products under 350, 700 and 1000 g-forces were shown readily separated from the slag. It can be possible to increase the metallic recoveries even higher by using some additives into the green mixture to reduce the ratio of scattered materials under artificial gravity conditions.

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