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Research Article

INVESTIGATION OF MICROSTRUCTURE AND MICROHARDNESS PROPERTIES OF CO AND CR REINFORCED NIAL INTERMETALLIC COMPOUND PRODUCED BY SHS METHOD

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In this study, the porous samples were manufactured through self-propagating high-temperature synthesis (SHS) by adding 10 % Co and Cr to 50 % Ni – 50 % Al powders. 10 % Co and Cr added 50 % Ni – 50 % Al samples were mixed for 12 hours using a mixer with a rotational speed of 300 rpm and compacted under 100 MPa pressure. The obtained samples were then synthesized by moving them to an argon atmosphere and combusted with the aid of a tungsten electrode. The obtained samples after the combustion reaction were characterized in terms of their microstructure using scanning electron microscope - the backscattered electrons (SEM-BSE). The elemental and phase analysis were carried out using EDS and XRD, respectively. The Energy-dispersive spectroscopy (EDS) results showed that the dominant element was Ni and the presence of Co with Cr was less than that of Al. As a result of XRD analyses, it was determined that NiAl was formed as the dominant phase in the samples and also Ni3Al and NiAl3 phases were formed in low amounts. Both Cr and Co addition increased hardness of NiAl alloy. However, Cr has a greater effect on the increase of hardness values than Co.

Key words: SHS, NiAlCo, NiAlCr, Microstructure, Microhardness

1. Introduction

Properties such as resistance to temperature, corrosion resistance at high temperatures and resistance to oxidation, which are among the desired properties in equipment operating at high temperatures are presence in the NiAl intermetallic material [1-3]. Therefore, its produce for mass production and put into use has an important place in industrial sense [4-6]. Reaction synthesis method, which is a low cost, easy to install and use method, which does not require external energy and thus does not cause environmental damage, has started to attract great attention in obtaining NiAl and Ni3Al intermetallic compounds [7-9].

Problems such as brittleness, improvement of mechanical properties, limiting the use of NiAl intermetallic alloy cover a significant part of the researches for the development of these materials. The most important of these problems is the brittleness of these materials and thus the restriction of

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Received: 9 November 2019; Accepted: 28 December 2019 Doi: https://doi.org/10.36222/ejt.657185

forming [2]. In the researches, Cr, which is indicated to increase toughness and ductility, and Co, which is indicated to increase porosity and hardness, were investigated by adding to NiAl, and the hardness values of the produced samples were investigated [2,5,10, 11]. The aim of this study is to investigate the effect of Co and Cr element added to NiAl powder compound on microstructure and microhardness properties of NiAl. At the same time, microstructure image by SEM-BSE, elemental analysis by EDS and phase components were determined by XRD analysis.

2. Materials and Method

Ni, Al, Co and Cr metal powders used in alloy production were obtained from a commercial company. Information on these metal powders is given in Table 1.

Table 1: Properties of metal powders used in laboratory

Materials	Purity (%)	Powder Size (mesh)	Melting Temperature (°C)	Specific Weight (gr/cm^3)
Aluminum	99,99	-325	660	2,700
Nickel	99,99	-325	1453	8,908
Cobalt	99,99	-325	1495	8,920
Chromium	99,99	-325	1875	7,190

In this study, every step of the laboratory studies was carried out in Argon atmosphere. In the glove box, a mixture of Ni and Al element powders prepared in the atomic ratios given in Table 2 and 10% Co and Cr powders were added. The prepared powder mixtures were mixed in the mixing mill at 300 rpm for 12 hours [12]. Table 2 shows the atomic and mass ratios and theoretical densities of Ni, Al, Co and Cr powder mixtures.

Table 2: Atomic and theoretical densities of powder mixtures

Mixture Number	Powder Mixture	Atomic Ratio	Weight Ratio	Theoretical Density
1	NİAl	%50Ni- %50Al	%76,74 Ni %23,26 Al	5,804 g/cm^3
2	NiAlCo(10)	%45Ni - %45Al - %10Co	%65,56 Ni %19,88 Al %14,56 Co	6,11 g/cm^3
3	NiAlCr(10)	%45Ni - %45Al - %10Cr	%67,45 Ni %20,45 Al %12,10 Cr	5,94 g/cm^3

The powder mixtures obtained at the end of this process were compressed in a cylindrical mold by cold press with a pressure of 100 MPa. Pre-and post-ignition (raw and synthesized) pictures of compacted 10% alloy doped NiAl raw samples are given in Figure 1.

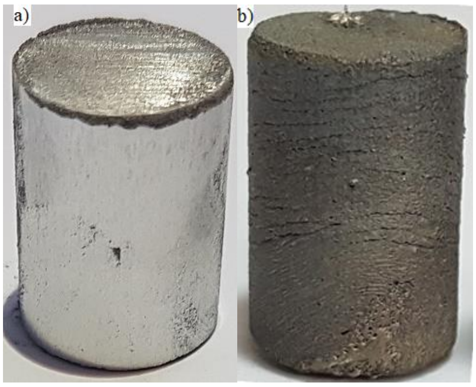


Figure 1: a) Raw sample b) Synthesized sample

The device for preheating and igniting the pressed samples in argon atmosphere was designed and produced. Ignition unit consists of preheating chamber, ignition center and chamber, temperature control knob and argon gas inlet-outlet sections. The scheme of the production by SHS method is given in Figure 2.

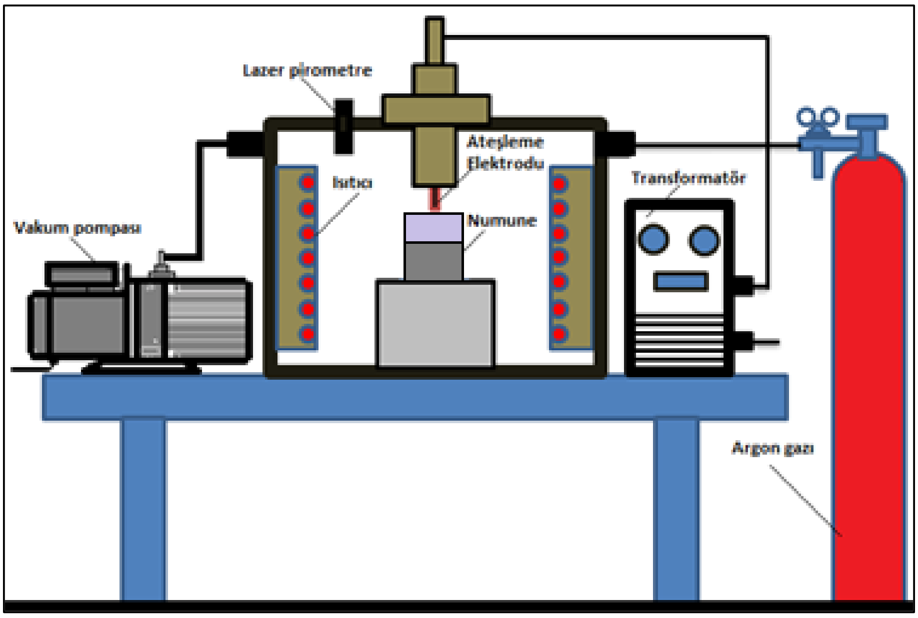


Figure 2: The scheme of ignition unit[13]

The external heat, which would allow the self-synthesis of the compacted samples, was given by ignition in the firing chamber under argon atmosphere. Samples were cut on the cutting disc for metallographic analysis after synthesis.

The cut samples were polished using 240, 400, 600, 800, 1000 and 1200 mesh sanders. After pre-polishing, the samples were polished with 1 μm size diamond paste solution for final polishing. For the microstructure analysis of the polished samples, etch of 33 wt% HF, 33 wt% HNO_3, 33% water solution was used. Leica brand optical microscope, Jeol brand SEM-BSE and EDS elemental analysis devices were used for microstructure analysis of samples after etching. The phase compositions were determined using Cu Kα radiation and the sample in horizontal position (2θ angles: 10 to 120 ° C) using Rigaku MiniFlex. Shimadzu brand microhardness tester with 20 g ($HV_{0.02}$) loading was used to obtain microhardness values.

3. Results and Discussion

In this study, samples were obtained by adding Co and Cr to NiAl powder mixture. Microstructures and microhardness values of these samples were determined. In this method, which is similar to powder metallurgy production methods, porosity decreases as a result of increasing contact of powder particles with pressing [14]. It is thought that the rapid synthesis reaction may increase the pore ratio since there is no time for shrinkage of the samples. In addition, addition of Co and Cr alloy elements changed the porosity rates. As a result of the calculations, the porosity ratio of unalloyed NiAl was determined as 27.17%. However, the NiAl intermetallic material with 10% Co added was 28.25% due to the effect of Co element. However, the addition of 10% Cr reduced this porosity by 9.83% and was found to be 25%. It is found that Co and Cr additive increases microhardness values at different rates.[12] The addition of Co and Cr triggers the formation of NiAl3 and Ni3Al phases. Formation of NiAl3 and Ni3Al changes hardness because the hardest point of Ni3Al is almost 500 HV and NiAl3 harder than Ni3Al about 200 HV.

The lowest point of hardness of NiAl as a function of stoichiometry is when Ni/Al is 1 and it is 330 HV [12].The Cr additive reduced the porosity relatively, and the Co additive was found to increase the pore ratio further. These data and the data in the literature support each other. [11,15-19]. Microhardness measurements taken from Cr and Co reinforced samples are given in Figure 3. According to the hardness measurement results, the highest hardness value obtained in the Cr alloyed samples as 578 HV and 523 HV for Co alloyed samples while hardness of NiAl is between 279-339 HV according to studies.[12The mean hardness of Cr alloyed samples was 77.6 HV harder than the mean hardness of Co alloyed samples. In addition, the value range of the measurements taken in Cr reinforced samples has a narrower range than Co reinforced samples. The reason for this is thought to be that the phases in the structure of Cr reinforced samples are less and the number of phases in Co reinforced samples is higher. In addition, the porosity of the material is one of the factors affecting this situation.

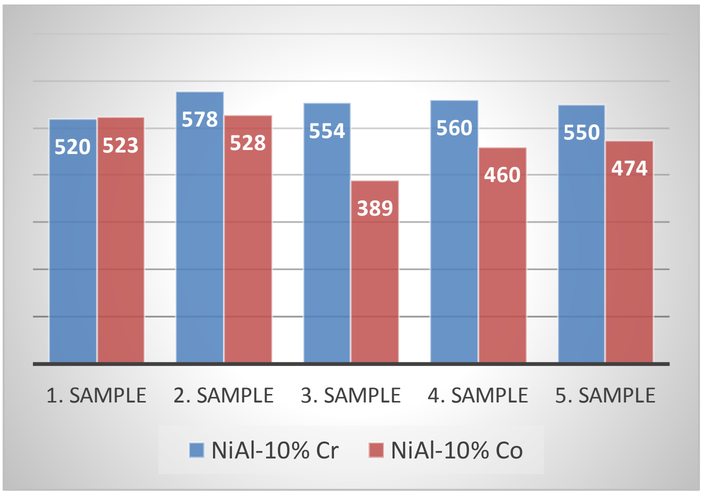
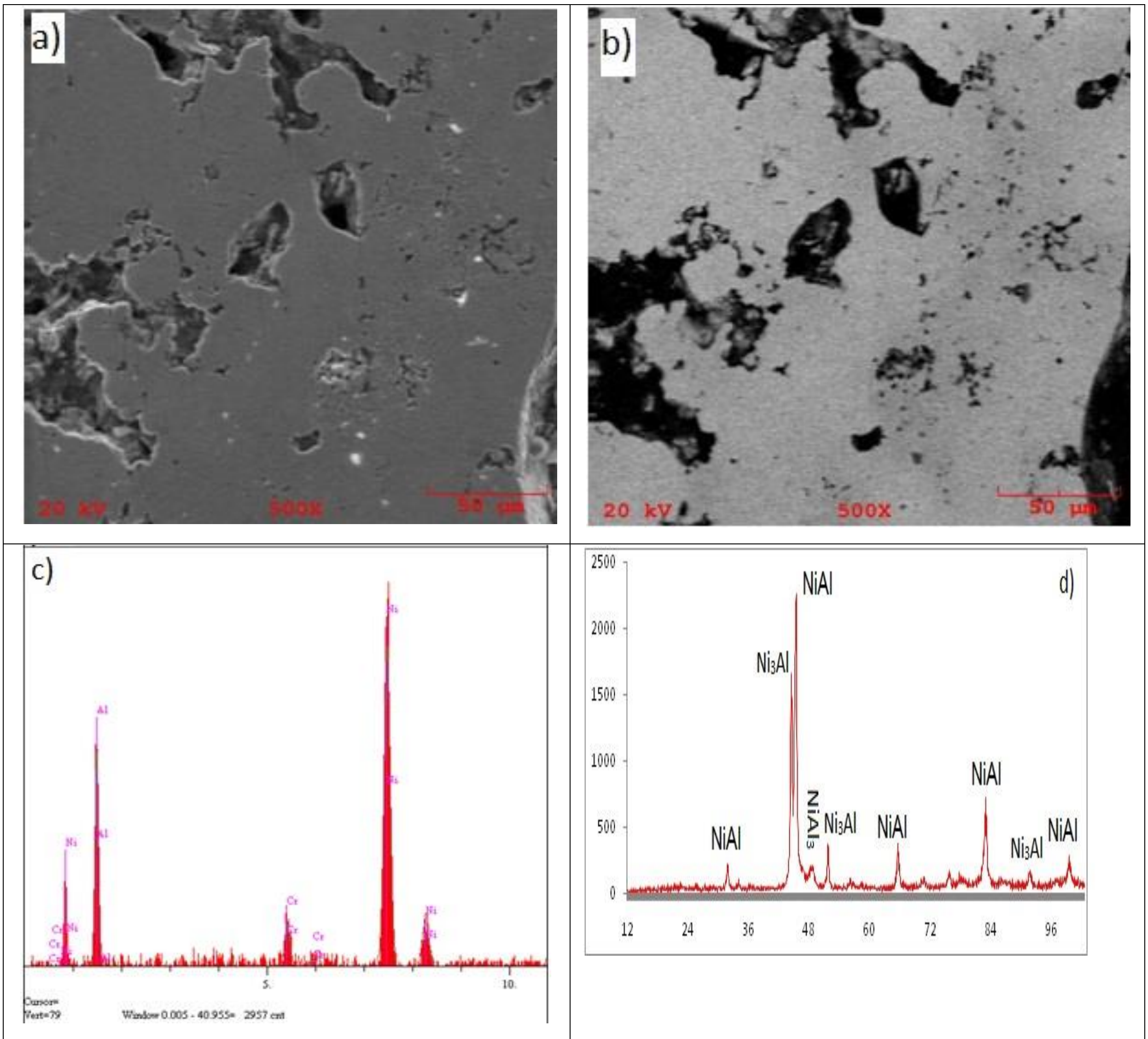
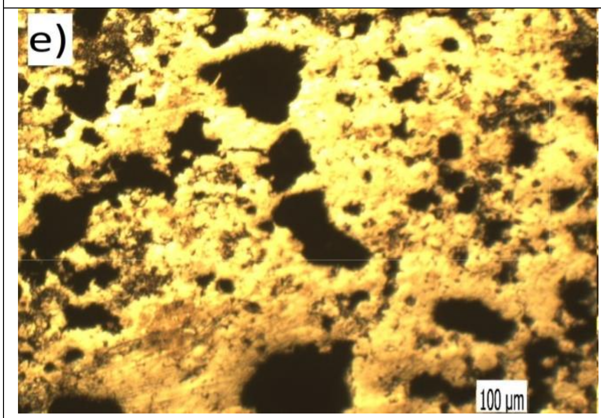


Figure 3: Co and Cr reinforced NiAl intermetallic compounds microhardness values

SEM, BSE and optical microscope images and XRD, EDS graphs of Cr and Co reinforced NiAl alloys are given in Figure 4 and Figure 5. The porous structure of the produced materials can be seen in SEM and optical microscope views . The composition ratios of the samples in the EDS results confirm the alloying element ratios in the produced samples.

In the XRD analysis, NiAl and Ni_3Al phases and low $NiAl_3$ phases were detected in Cr alloyed NiAl sample. On the other hand, more NiAl phase and low Ni_3Al and $NiAl_3$ phases were detected in the Co alloyed NiAl sample

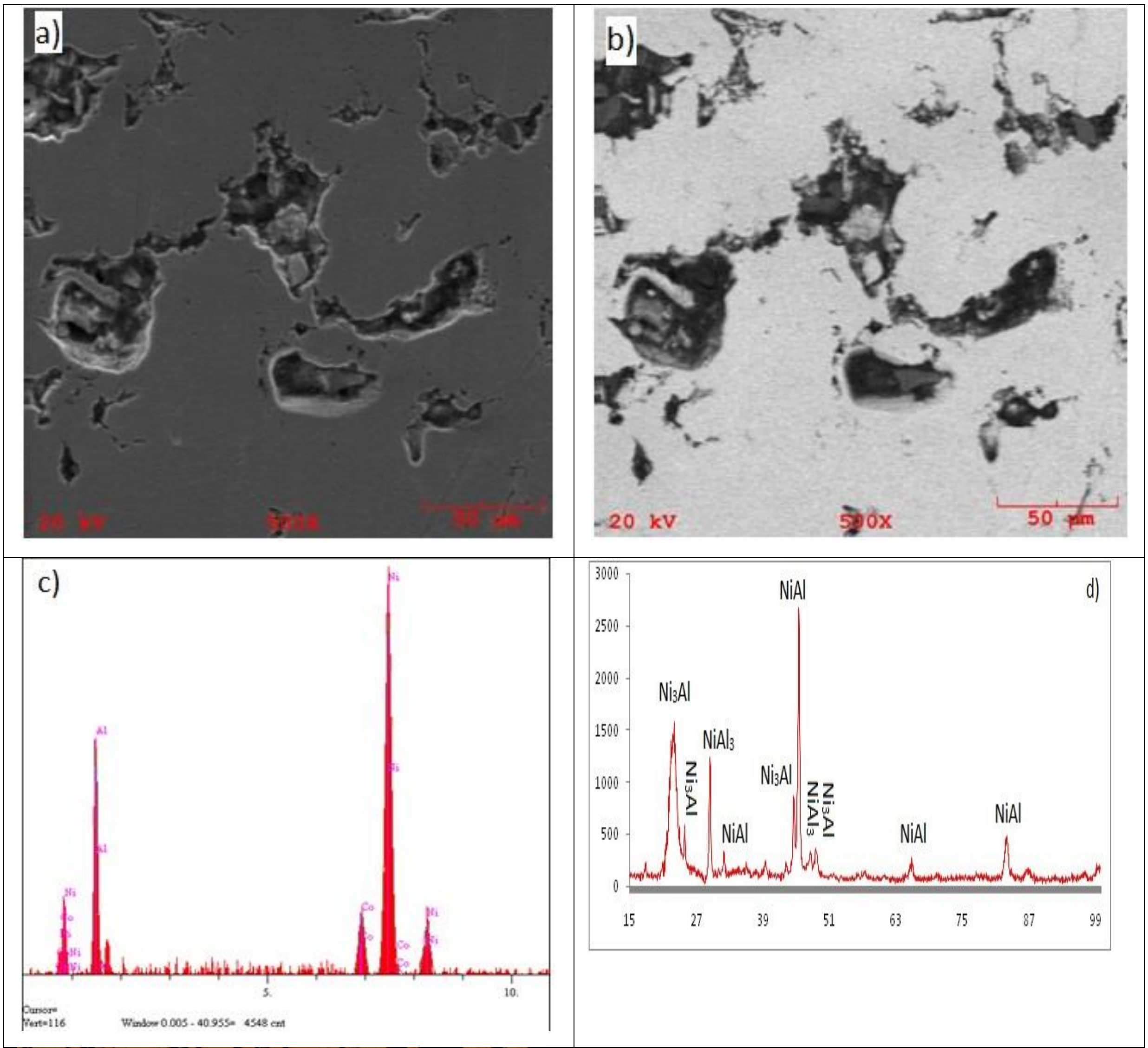


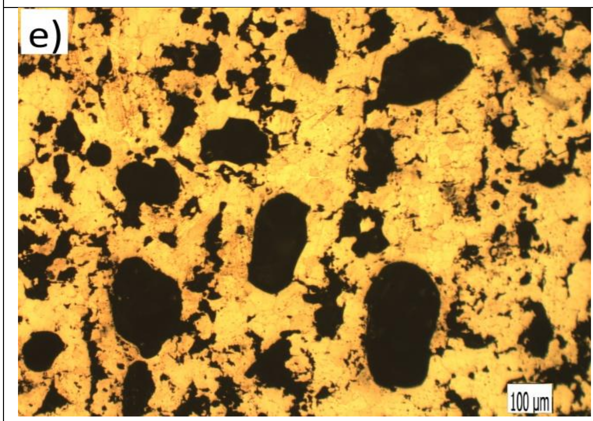


f)

Element	Line	Intensity (c/s)	% Atomic
Al	Ka	21,15	% 15,3
Co	Ka	8,83	% 9,8
Ni	Ka	55,49	% 74,9
			% 100

Figure 4: a) SEM, b) BSE, c) EDS graph, d) XRD, e) optical microscope image, f) EDS composition ratios of the Cr reinforced NiAl alloys





f)

Element	Line	Intensity (c/s)	% Atomic
Al	Ka	21,15	% 15,3
Co	Ka	8,83	% 9,8
Ni	Ka	55,49	% 74,9
			% 100

Figure 5: a) SEM, b) BSE, c) EDS graph, d) XRD, e) optical microscope image, f) EDS composition ratios of the Co reinforced NiAl alloys

4. Conclusions

In this study, by using SHS method, 50% Ni-50% Al powders were reinforced with 10% Co and 10% Cr and sample production was carried out and microstructure and microhardness analysis of samples were performed. According to the data obtained;

- It was found that Co and Cr additive increases the microhardness values of NiAl at different rates. The highest hardness of Cr alloy specimens was 578 HV, while Co alloy specimens had 523 HV. The mean hardness of Cr alloy samples was 77.6 HV harder than the mean hardness of Co alloyed samples.
- Based on the optical microscope and SEM-BSE images, the samples produced were found to have high porosity. This porosity is thought to be an important factor in microhardness results.
- The most common NiAl and Ni_3Al phases and low $NiAl_3$ phases were determined in the Cr alloyed NiAl sample. Co alloyed NiAl sample has more NiAl phase and low Ni_3Al and $NiAl_3$ phases.

Acknowledgements

This study was supported by YTU BAP Coordination Unit as project FYL-2018-3354. We would like to thank YTU BAP Coordinator for their support.

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