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Effect of Diluent Amount on Properties of Porous NiAl

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Keywords

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Abstract

Porous NiAl parts were formed by using Ni and Al elemental powders, preformed NiAl as diluent and NaCl particles as space holder (SH). The aim of utilizing preformed NiAl (30%-40%) as a diluent was to preserve the shape of the products. The amounts of the SH NaCl particles in NiAl were 25-50-75 vol.% and their sizes were in 300-500 µm range. Porous NiAl samples were prepared by volume combustion synthesis (VCS). The adiabatic temperatures of the Ni+Al mixtures having 30 and 40% diluent NiAl were calculated as 1638.9 and 1460.8°C, respectively. Formation of NiAl phase was verified by XRD analyses. In the green pellets, the total porosity amount was higher than the added NaCl amount. Also it was slightly higher in the product pellets than in the green pellets before VCS. Compressive strength and microhardness values of the samples which contained 30% diluent NiAl were higher than the samples which contained 40% diluent. Average compressive strength values of the products that were obtained by 25% NaCl and 30 and 40% diluent NiAl additions were 112.0±29.5 and 66.0±20.5 MPa, respectively.

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1. INTRODUCTION

Porous metals are used in structural applications of various industries, including the automotive and aerospace industries, shipbuilding, sports equipment and biomedical industries (Banhart, 2001). The spacer or space holder (SH) method attracted interest because of its ability to regulate the dimensions, shape, and connection of porosity (Arifvianto & Zhou, 2014). In this method, removable particles are used for creating pores of controlled size and amount. In literature, urea and NaCl particles are most widely utilized space holders. After shaping the metal structure, they can be removed by dissolving in water or urea can also be removed by thermal decomposition (Banhart, 2001). The sintering method is one of the methods used in the production of porous materials including porous ceramics and metals. During sintering the characteristics of the pores may be regulated by physically adjusting the morphology, size and amount of the space holder among the starting powder particles (Jiang et al., 2021).

Volume combustion synthesis (VCS) is a convenient method for obtaining articles made of intermetallic compounds. It utilizes the exothermic energy of the reactions; therefore, it is an economical process. In this method, a powder mixture is ignited by heating throughout its whole volume and the reaction takes place all at once. Self-propagating high-temperature synthesis (SHS) on the other hand, involves ignition of the reactant pellet from one side, mostly by heating with a resistance wire. Systems which have higher adiabatic temperatures than 1800K were suggested to be self-propagating (Su et al., 2014). However, in VCS, sysnthesis of systems which have lower adiabatic temperature can be possible. VCS has been used for preparation of compounds such as FeAl, NiAl and NiTi, etc (Varma et al., 1998). 3 significant temperature

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points in the combustion synthesis that affect the reaction process and properties of the end product are: 1) ignition temperature (T_i) is expressed as the stage at which the SHS initiates, 2) adiabatic temperature (T_{ad}) is the highest combustion temperature reached in adiabatic circumstances, 3) the actual combustion temperature (T_c) is the highest temperature obtained in non-adiabatic conditions (Moore & Feng, 1995).

In one of the studies in literature which employed both space holder and combustion synthesis technique, Wang et al. (2016) have produced porous TiAl-based intermetallics by VCS. They used NaCl as the space holder (SH) agent. They found that TiAl was developed as the major phase in materials sintered at 1100°C, and the open porosity increased up to 84% with the addition of 80% NaCl to Ti-50Al. Cai et al. (2021) prepared porous NiAl intermetallics by thermal explosion by using space holder NaCl. They showed that there was no cracking or deformation in the sample when the space-retaining NaCl content in NiAl reached 30% by volume. They reported that the porosity was increased to 63% when 50% NaCl was added by volume. In the study of Shu et al. (2019) porous Ni3Al, NiAl and NiAl3 were obtained by reactive sintering at 650°C for 10800 s. The products were reported to exhibit good energy absorbing characteristics. Li et al. (2019) have synthesized porous Ni_3Al, NiAl and $NiAl_3$ intermetallics by thermal explosion process. The open porosity of Ni_3Al, NiAl and $NiAl_3$ were obtained as 27.4%, 40.3% and 41.5%, respectively. They stated that especially porous NiAl showed excellent oxidation resistance. Yang et al. (2019) investigated compression fatigue properties of open-celled Al foams produced by the spacer method with different porosities. Fatigue strengths of 50%, 60% and 70% porous Al foams were obtained as 12.77 MPa, 7.61 MPa and 3.02 MPa, respectively. It was observed that there was a linear decrease with increasing porosity.

The amount of diluent is critical for the mechanical properties of the products in VCS. This is because the adiabatic temperature of the reaction decreases with the increase in the amount of diluent. When the diluent amount is too high, mechanical properties of the products becomes inferior due to inefficient sintering. On the other hand, when the diluent amount is too low, too much liquid phase may form in the product as a result of the high adiabatic temperature. This leads to partial melting, shape distortion of the product and also reduction in porosity if porous products are aimed.

In the present study, NiAl porous materials were produced using the space holder method. Pores were formed with controlled size and amount by using 25, 50 and 75 vol.% NaCl particles in NiAl as space holder. In order to control the adiabatic temperature during VCS, preformed NiAl was utilized as diluent. Therefore, the effect of 30 and 40% diluent NiAl addition on macrostructure of the formed porous articles were investigated and mechanical properties were determined.

2. EXPERIMENTAL PROCEDURE

Porous NiAl samples were produced according to Reaction (1) by using 25, 50, 75 vol.% NaCl (Merck, >99.5) as space holder particles. NaCl particles were sieved in a sieve set, in order to separate the particles in 300-500 μm range. These particles were utilized as space holder. Preformed NiAl was added to nickel (Merck, 99+%, <10 μm) and aluminum (Alfa Aesar, 99.5%, <10 μm) powders as diluent, as shown in Reaction (1).

$$(x)\mathrm{Ni} + (x)\mathrm{Al} + (y)\mathrm{NiAl} = (x+y)\mathrm{NiAl} \quad (1)$$

2.1. Preparation of Preformed NiAl Powder

Ni, Al and MgO (Merck, 99.5%, <1 μm) were weighed and mixed. MgO was used as diluent. After the prepared powder mixture was taken into a steel mold with a diameter of 10 mm, a pressure of 100 MPa was applied. The compressed sample was removed from the mold and volume combustion synthesis was conducted in an induction furnace. The formed product was crushed in a mortar and pestle. In order to dissolve the MgO in the mixture, it was kept in HCl+pure water for about 1 hour. Then, this mixture was filtered and the residue was dried in an oven at 65°C for 1 day. As a result of these processes, diluent NiAl was obtained. XRD analyses verified that the product was single phase pure NiAl powder.

2.2. Sample Preparation

Ni, Al powders, preformed NiAl and NaCl were weighed on an analytical balance and mixed homogeneously in the mixing bowl. The sample height for each piece was calculated as 9 mm. The cylindrical samples obtained were prepared in an 8 mm diameter steel mold by applying 100 MPa pressure in a cold press. The produced part was kept in pure water for about one day in order to dissolve the NaCl particles. Afterwards, the samples were dried in an oven for about a day. After drying, the parts were subjected to combustion synthesis reaction by heating in a tube furnace up to 700°C at a rate of 10°C/min. in Ar atmosphere. Codes of the prepared samples, amount of diluent NiAl and space holder NaCl are presented in Table 1.

Prepared sample codes, amount of diluent NiAl and space holder NaCl are presented in Table 1. For example, 30DN25SN is code used for the sample that contained 30% Diluent NiAl and 25% Space holder NaCl.

***Table 1**. Amount of diluent NiAl and space holder NaCl in the prepared samples and their codes*

Diluent NiAl amount (%)	**NaCl Amount (vol.%)**		
	25	50	75
30	30DN25SN	30DN50SN	30DN75SN
40	40DN25SN	40DN50SN	40DN75SN

Adiabatic temperatures (T_{ad}) of the reactant mixtures were calculated by the HSC Chemistry software according to Reaction (1). Adiabatic temperature is the maximum temperature that the products will reach as a result of an exothermic reaction, when there is no heat loss in the system. It can be calculated by Equation (1). ΔH_{298}, ΔH_m, Tm and C_p in Equation (1) are the reaction enthalpy, and latent heat of fusion, melting temperature and heat capacity of the product phase, respectively. T_{Ad} is the adiabatic temperature.

$$\Delta \mathrm{H}_{298} = \int_{298}^{\mathrm{T_m}} \mathrm{C_p dT} + \Delta \mathrm{H_m} + \int_{\mathrm{T_m}}^{\mathrm{T_{Ad}}} \mathrm{C_p dT} \tag{2}$$

2.3. Sample Characterization

Before and after the removal of NaCl particles, the pellets were weighed, and their dimensions were measured. The density of the green pellet after NaCl removal was calculated by dividing the weight of the pellet by the volume of the pellet. The relative % density of the green pellet was calculated by dividing the density of the pellet by the theoretical density of NiAl (5.9 g/cm^3) and multiplying by 100. % total porosity was calculated by subtracting the relative % density of the pellet from 100. The amount of porosity in the cell walls of the green pellet was calculated by subtracting the NaCl amount from % total porosity of the pellet.

$$\text{Density of the green pellet } = \frac{\text{weight of the pellet}}{\text{volume of the pellet}} \tag{3}$$

$$\text{\% Relative density } = \left(\frac{\text{density of the pellet}}{\text{theoretical density of NiAl}}\right) * 100 \tag{4}$$

$$\text{\% total porosity } = 100 - \text{Relative \% density} \tag{5}$$

$$\text{Porosity in the cell walls of the green pellet} = \text{\% total porosity} - \text{ NaCl amount of the pellet} \tag{6}$$

XRD analyses were performed on selected samples. A Rigaku Smartlab unit was used with 0.02^o steps and 2^o/min speed. Compression test was applied to the produced samples with a Shimadzu Autograph Universal Tensile-Compression Device (50 kN) after volume combustion synthesis, in accordance with the ISO 13314 standard. The strength values of the materials were measured, and the results were presented in a stress-strain diagram. For macrostructural examinations, metallographic preparation methods were employed. Porous NiAl parts formed after volume combustion synthesis were ground on 600, 1200 and 3000 grit sandpaper and polished. After polishing, the macro structure of the samples was examined with a stereo microscope. In addition, an optical microscope was utilized for microstructural examinations (Nikon Eclipse LV150). Microhardness measurements were conducted via a vickers microhardness tester by applying 200g load.

3. RESULTS AND DISCUSSION

3.1. Adiabatic Temperature Calculations

The adiabatic temperatures of the reactions were calculated by the HSC Chemistry (Roine, 2002) software. The calculated adiabatic temperatures of Reaction (1) is presented in Figure 1, up to 50% preformed NiAl addition. It was seen that the adiabatic temperature remains constant at 1638.9°C up to 30% preformed NiAl addition. 1638.9°C is the melting temperature of NiAl. When there is no diluent in the system, a certain fraction of NiAl in the products melts. The high amount of liquid phase that is formed in the system when there is no diluent NiAl results in distortion of the product pellet. The shape of the pellet and the pores can be preserved by the addition of diluent NiAl in VCS.

The fraction of liquid phase decreases with the increase in the diluent NiAl addition to the starting mixture, since the preformed NiAl absorbs some of the evolved heat. This continues up to 30% preformed NiAl addition. After this point, the evolved heat is not sufficient to cause melting of the products and there will be no liquid phase formation in the products. The adiabatic temperature starts to decrease with preformed NiAl addition after 30%. T_{ad} is 1460.8°C and 1253.2°C when 40 and 50% preformed NiAl was added, respectively. According to these results, 30% preformed NiAl addition was selected in order to decrease the adiabatic temperature and the amount of liquid phase in the system, for preventing the distortion of the product pellet. In VCS the reaction initiates at a higher temperature than the room temperature. Therefore, the attained temperature of the pellet may be higher than the calculated adiabatic temperature. Thus, the effect of addition of 40% diluent NiAl was also investigated.

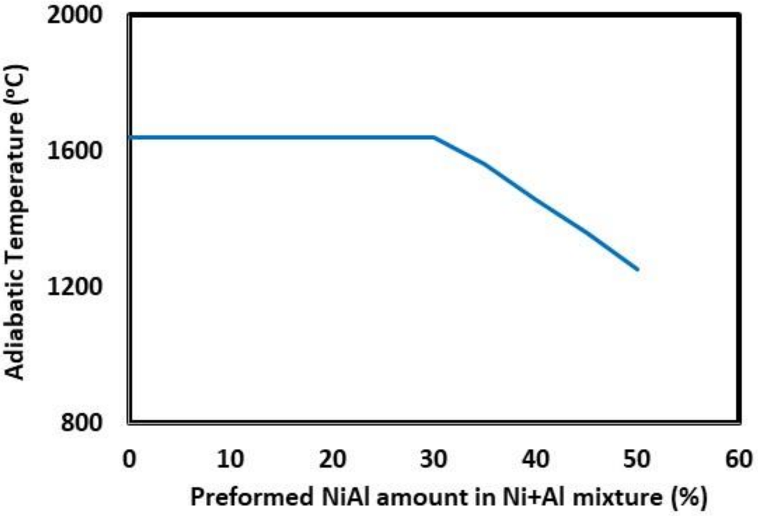


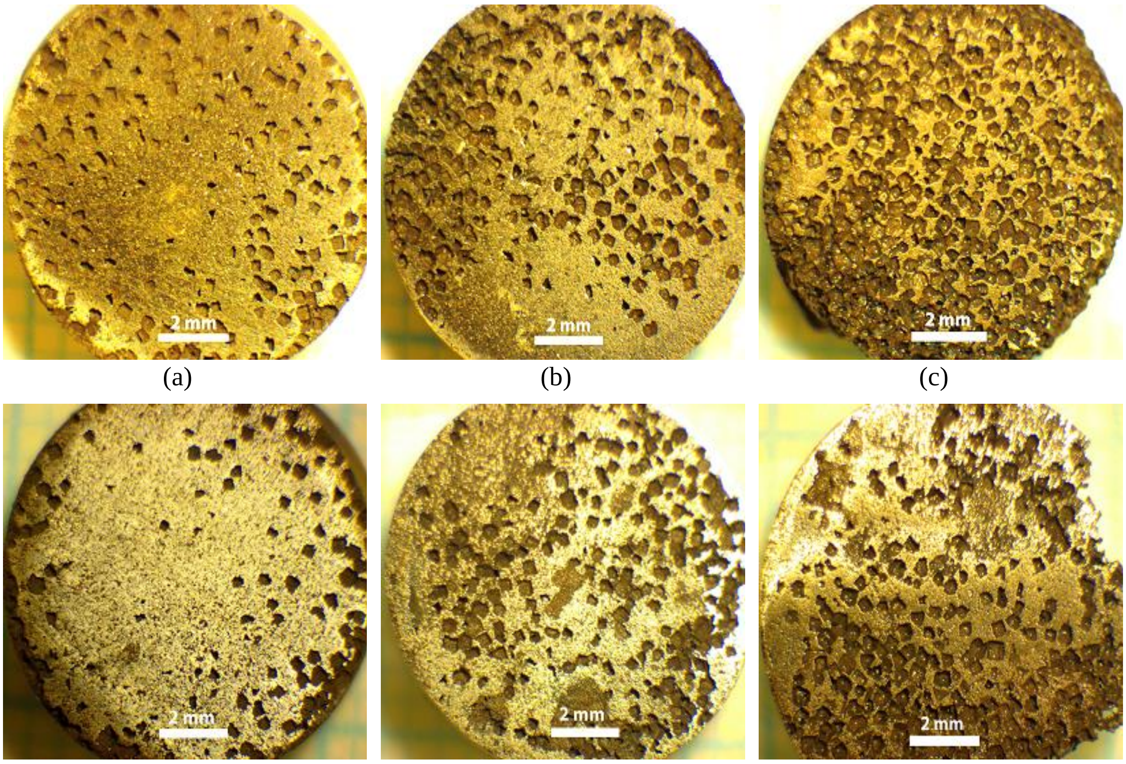
Figure 1. *Calculated adiabatic temperatures of the Ni+Al mixtures as a function of diluent preformed NiAl addition*

Selected samples in the prepared sample groups were subjected to XRD analyses. It was found that pure NiAl phase was formed in the samples by using both 30 and 40% diluent NiAl addition.

3.2. Macrostructure

Addition of both 30 and 40% diluent NiAl powder addition to the starting mixture resulted in preserving the shape of the cylindrical pellet. There was no sign of melting in the pellets. On the other hand, when no diluent NiAl was used, the shape of product pellet was distorted.

The macrostructures of the product pellets are presented in Figure 2. These images were obtained from the cross-sections of the pellets. It can be seen in these images that the created pores mostly replicated the initial shape of the NaCl particles. The porosity increased with increasing amount of NaCl content of the initial mixture. When 75% NaCl was used, the formed pores tended to combine and unify. It can be concluded that 75% is above the limit of proper space holder content.



(a) (b) (c)

(d) (e) (f)

Figure 2. *Macrostructures of the product pellets*
a) *30DN25SN,* ***b)*** *30DN50SN,* ***c)*** *30DN75SN,* ***d)*** *40DN25SN,* ***e)*** *40DN50SN,* ***f)*** *40DN75SN*

Average total porosity of the green pellets having 30 and 40% diluent NiAl before VCS are presented in Table 2 and Table 3, respectively. The amount of total porosity in the green pellets before VCS was very similar in both series, as expected. It was seen that the total porosity amount was more than the added space holder NaCl amount in both series. This is due to the presence of porosity in the cell walls which contained the Ni+Al particles. This difference decreased with the increase in the NaCl content, or decrease in the amount of Ni+Al. The cells in the green pellet are the voids which were formed by the removal of the NaCl particles by dissolution in water.

The amount of porosity in the cell walls before VCS decreased with the increase in the space holder NaCl amount as shown in Table 2 and 3. It can be suggested that compressibility increased with the increase in the amount of NaCl particles.

Table 2. *SH NaCl contents and average total porosity of the pellets having 30% preformed NiAl, before and after VCS*

	Before VCS				**After VCS**	
SH NaCl Content (%)	**Av. Total Porosity (%)**	**Standart Dev.**	**% Pore in Cell walls**	**Standart Dev.**	**Av. Total Porosity (%)**	**Standart Dev.**
25	48.8	2.4	31.8	3.2	50.1	3.0
50	62.3	1.6	24.7	3.2	64.2	2.0
75	78.3	1.3	13.4	5.2	78.3	0.1

Table 3. *SH NaCl contents and average total porosity of the pellets having 40% preformed NiAl, before and after VCS*

	Before VCS				**After VCS**	
SH NaCl Content (%)	**Av. Total Porosity (%)**	**Standart Dev.**	**% Pore in Cell walls**	**Standart Dev.**	**Av. Total Porosity (%)**	**Standart Dev.**
25	49.1	2.3	32.1	3.1	54.3	3.1
50	62.6	1.6	25.2	3.2	65.6	1.8
75	77.9	2.3	11.4	9.2	77.9	1.2

Average pore amount in the products obtained from 30 and 40% NiAl diluent added mixtures after VCS, as a function of NaCl content are presented in Tables 2 and 3, and in Figure 3. In both series, the total porosity increased a few percent after VCS as compared to green pellets. For comparison, amount of NaCl space holder amount is indicated in Figure 2 as the gray straight line. Increase in the pore amount is expected in VCS due to gas release as a result of the temperature increase, Kirkendall effect during diffusion and density difference of the reactants and the products (Jiang et al., 2021).

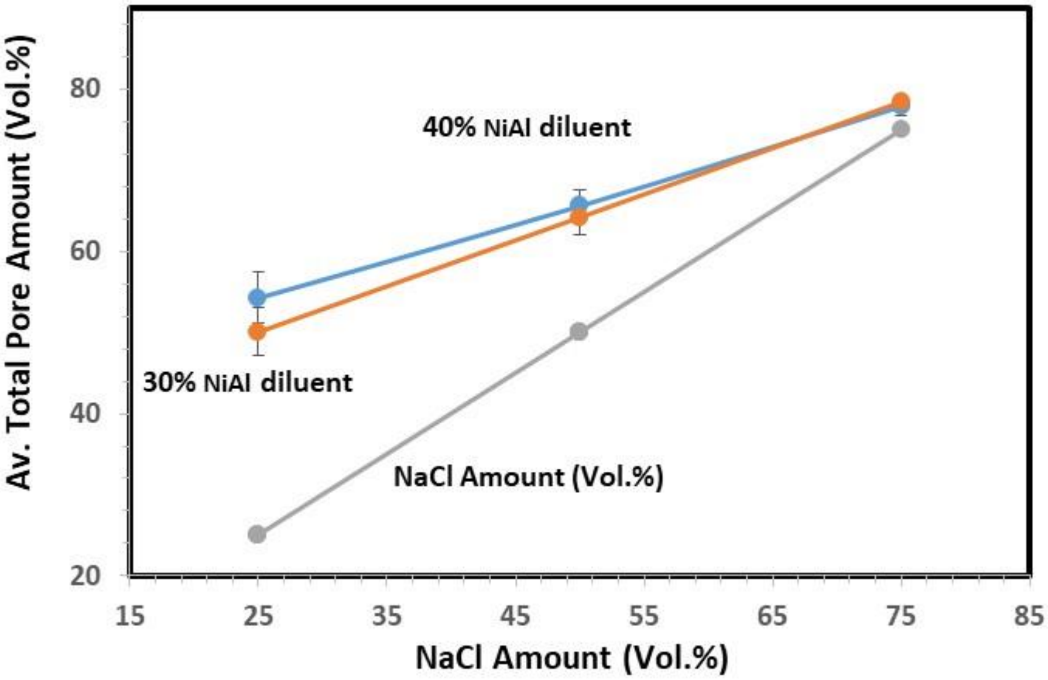
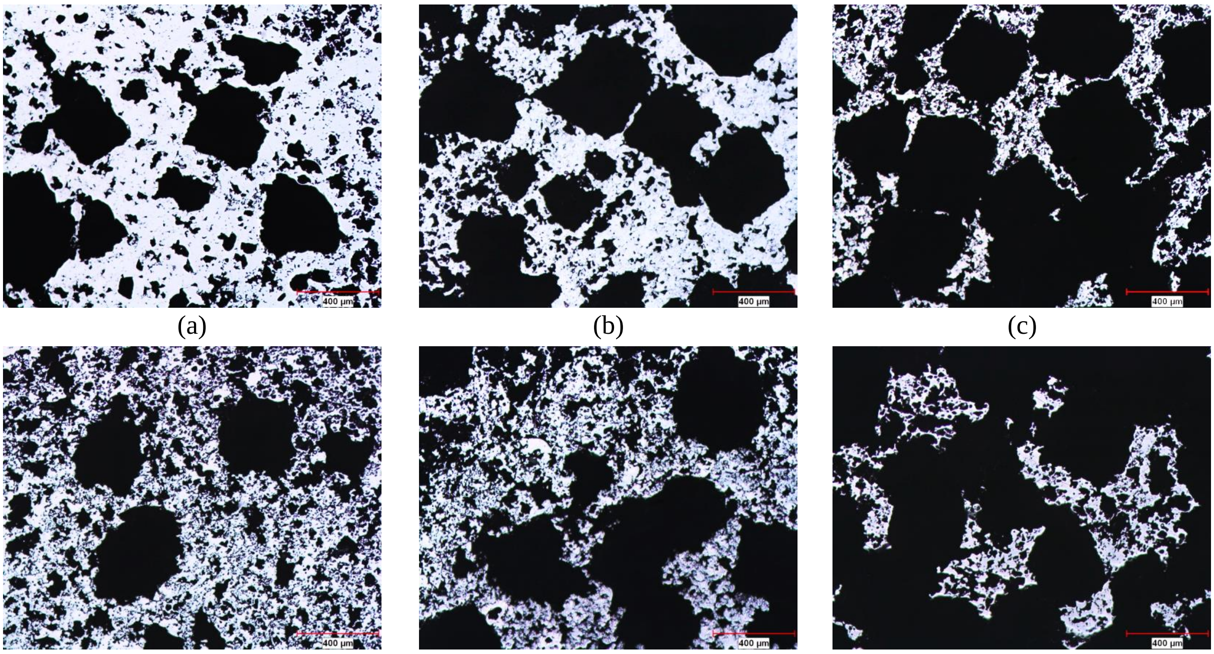


Figure 3. *Average pore amount in the products obtained from 30 and 40% NiAl diluent added mixtures after VCS, as a function of NaCl content*

3.3. Microstructure

The microstructural images of the samples are presented in Figure 4. In these images, the white regions are the cell walls and black regions are the pores. The large primary pores (cells) of about 400 μm size were formed by the space holder NaCl particles. The sizes of these large pores match well with the size of the utilized NaCl particles. It can be seen that the cell walls also contained some porosity. These secondary pores in the cell walls are believed to form due to the Kirkendall effect, porosity of the green pellet and volume difference between the reactant and product phases. It was inferred from microstructure examinations that the amount of the secondary pores in the cell walls increased with increasing diluent NiAl amount.



(a) (b) (c)

(d) (e) (f)

Figure 4. *Optical microscope images of the product pellets*
a) *30DN25SN,* ***b)*** *30DN50SN,* ***c)*** *30DN75SN,* ***d)*** *40DN25SN,* ***e)*** *40DN50SN,* ***f)*** *40DN75SN (magn. 50X)*

3.4. Mechanical Tests

Compression strength values of the samples formed by the addition of 30 and 40% diluent NiAl and 25, 50 and 75% space holder NaCl are presented in Figure 5 and Table 4. Samples with 30% NiAl diluent addition had higher compressive yield strength values than samples with 40% NiAl diluent addition. This may be a result of higher amount of liquid phase and thus better sintering of the samples that contained 30% diluent NiAl. With the increase in the amount of diluent NiAl from 30 to 40%, less amount of liquid phase is expected to form in the sample during VCS, as a result of the increased dilution effect of the higher amount of preformed NiAl.

The samples obtained from a mixture of 30 and 40% diluent NiAl and 25% spacer NaCl added showed average compressive strength values of 112.0±29.5 and 66.0±20.5 MPa, respectively. The high standard deviation may be attributed to the brittle nature of NiAl. With the increase in the amount of NaCl, there was a gradual decrease in the compressive strength of the porous NiAl samples. The compressive strength of the samples obtained from the mixtures with the addition of 30 and 40% diluent NiAl and 75% NaCl exhibited a compressive strength of 8.0±1.3 and 6.4±0.4 MPa, respectively.

Table 4. *Compressive yield strength values of the products obtained from 30 and 40% NiAl diluent added mixtures, as a function of NaCl content*

	30% Diluent NiAl			**40% Diluent NiAl**		
SH NaCl Content (%)	**Av. Comp. Yield Str. (MPa)**	**Standart Dev.**	**Strain at max. stress (%)**	**Av. Comp. Yield Str. (MPa)**	**Standart Dev.**	**Strain at max. stress (%)**
25	112.0	29.5	5.4	66.0	20.5	5.0
50	38.3	5.4	6.1	25.4	2.5	1.9
75	8.0	1.3	10.1	6.4	0.4	2.4

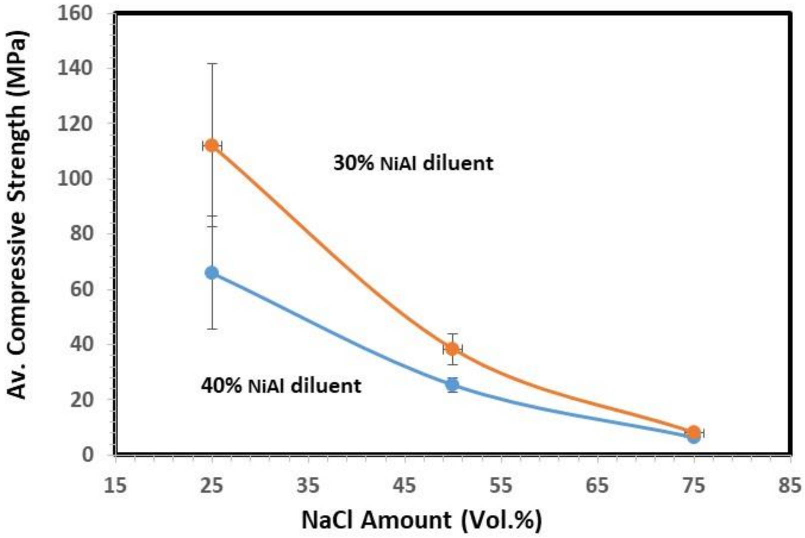
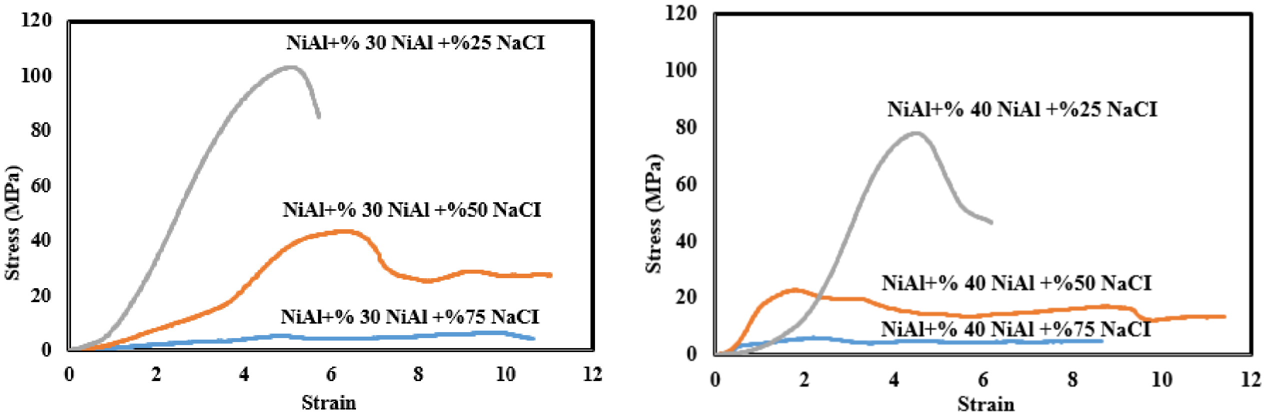


Figure 5. *Average compressive yield strength values of the products as a function of NaCl content in 30 and 40% diluent NiAl added samples*

Compressive stress-strain plots of the samples formed by the addition of 25, 50 and 75% space holder NaCl and 30 and 40% diluent NiAl are presented in Figure 6a and 6b, respectively. It can be observed that 30% diluent NiAl added samples exhibited higher compressive strength values for all NaCl amounts. There was a gradual decrease in the stress values, especially in the 50 and 75% NaCl added samples. This may be a result of the collapse of the cell walls in a sequence.

The microhardness measurements were conducted on samples which contained 25% NaCl. The thickness of the cell walls of the samples which contained higher amount of NaCl was not suitable for microhardness measurements. In addition, they contained high amount of secondary porosity in the cell walls. Microhardness of the samples which contained 30% diluent NiAl was higher than that of the samples which contained 40% diluent NiAl. This was attributed to the lower amount of secondary porosity in the samples which contained 30% diluent NiAl. The microhardness values of the samples which were produced by using 25% NaCl, and 30 and 40 diluent NiAl were 243±34, 188±25 $HV_{0.2}$, respectively. The microhardness of non-porous NiAl was previously reported as 394±37 $HV_{0.2}$, (Camurlu & Maglia, 2009) which is in accordance with the microhardness values obtained in the present study for porous samples.



(a) (b)

Figure 6. *Compressive stress-strain plots of samples which were formed by* ***a)*** *30% and* ***b)*** *40% diluent NiAl and 25, 50 and 75% space holder NaCl addition*

4. CONCLUSION

Porous NiAl parts were prepared by using NaCl space holder particles and via volume combustion synthesis technique, from Ni and Al powders. Addition of both 30 and 40% diluent preformed NiAl powder to the starting mixture resulted in preserving the shape of the cylindrical pellet. The adiabatic temperatures of the mixtures having 30 and 40% diluent NiAl were calculated as 1638.9 and 1460.8°C, respectively. The total porosity amount was seen to be more than the added space holder NaCl amount in both series. There was slight increase in total porosity amount in both series as a result of VCS reaction. This was attributed to the density difference of reactants and products, initial porosity in the reactants and Kirkendall effect during diffusion. Secondary porosity in the cell walls increased with increasing amount of diluent NiAl. Compressive strength and microhardness values were higher when 30% diluent NiAl was utilized. This was attributed to better sintering of the samples. Strength values decreased as a result of increase in the pore amount.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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