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Araştırma Makalesi / Research Article

Effect of Cold Compaction Pressure on Porous NiAl Articles Produced by Using Space Holder Urea via VCS

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ABSTRACT: Porous NiAl samples were obtained by using 20, 40, 60 vol.% space holder urea particles. The urea particle size was in the range of 150-300 μm. Pressures of 50, 100, 200 and 300 MPa were applied by cold press to the mixtures formed by nickel, aluminum, preformed NiAl powders and urea particles. The dimensions, macrostructure, microstructure and compressive strength of the parts produced with different cold compaction pressures were analyzed after volume combustion synthesis (VCS). The aim of the study is to determine the cold compaction pressure which provides the closest dimensional changes after VCS, in samples having different urea amounts. These samples will be used for forming a multi-stack article in a further study. Since a high difference in dimensional change of different layers during VCS will lead to cracking and separation, similar dimensional change is required for different layers in a multi-stack sample design. The samples which were cold pressed with 300 MPa pressure sometimes contained cracks after they were taken out of the die after cold pressing or after VCS. Therefore, they could not be subjected to characterization. The closest dimensional change and higher compressive strength values after VCS were obtained in samples having different urea contents, when they were cold pressed at 200 MPa. The highest mean compressive strength (218.8±29.5 MPa) was attained in the sample which was prepared with 20% urea particles and which was cold compacted with 200 MPa.

Keywords: NiAl, Combustion synthesis, Space holder method, Porous intermetallic

1. INTRODUCTION

Porous materials are produced using spacer method, gas expansion method and chemical reaction techniques (Michailidis and Stergioudi, 2011; Kanetake and Kobashi, 2006). They have been widely used in fields such as medical, biological, energy saving, chemical industry due to their organizable permeability, thermal shock resistance, controlled pore size properties (Qin et al., 2016).

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NiAl intermetallic has attracted attention as a light weight material that can be used at high temperatures due to its high temperature strength and low specific gravity (Dey, 2003).

It is known that the porosity and pore size of the material decrease with increasing pressure in the production of porous materials through powder metallurgy methods (Gülsoy and German, 2008; Dessai et al., 2013). Gao et al. (2015) produced the FeAl intermetallic using the reactive synthesis method by applying cold compaction pressures between 60 and 360 MPa. The effect of pressure on the pore structure of the obtained porous FeAl intermetallics was investigated. The manufactured parts were sintered at a sintering a temperature of 1200 °C for 60 minutes. Due to the Kirkendall effect (Gao et al., 2015) between Fe and Al elements, a large number of pores were produced along the diffusion direction of the Al element as Al atoms diffuse faster than Fe atoms. In addition, since the yield strength of pure Al is lower than that of Fe, Al particles undergo plastic deformation and take an elongated shape along the Fe particles. It was observed that the maximum pore size decreased by 33.7% when the pressure was increased from 60 MPa to 360 MPa. It has been reported (Gao et al., 2015) that by increasing the pressure, the pores between the particles and the diffusion distance can be reduced, and as a result, the degree of solid and liquid reaction may change. It was concluded (Gao et al., 2015) that the open porosity, maximum pore size and permeability of the porous FeAl intermetallics produced by reactive synthesis can be reduced with increasing pressure. No information was provided on the mechanical properties of the obtained samples.

It is known that the strength of porous intermetallics decreases with increasing porosity (Jiang et al., 2021). In the study of Jie et al. (2011), Ni+Al+urea mixtures were cold pressed at 200 MPa for compaction. The reaction was carried out in SHS mode. Pore size and amount of 0.4-2.0 mm and 58-85%, have been reported, respectively. The sample with 58% porosity exhibited a compressive strength of 27 MPa. Strength values decreased with increasing amount of porosity. Huang et al. (2020) used elemental Mo, Si and B powders and NH_4HCO_3 particles in the size range of 48 µm to 230 µm to form pores. A pressure of 300 MPa was applied to the green compacts. In the study, the size of the small pores was 0.1–10 µm, and the size of the large pores was obtained as 10–300 µm. It was observed that the compressive strength decreased rapidly from 329 MPa to 87.3 MPa at 40 vol.% porosity, as compared to 0 vol.%.

In this study, experiments were carried out to determine the cold compaction pressure for the production of porous NiAl parts. For this purpose, parts were produced by applying cold compaction pressure of 50, 100, 200 and 300 MPa by using nickel, aluminum, preformed NiAl (30 vol.%) powder and urea particles as a space holder. The aim of the study is to determine the cold compaction pressure which provides the closest dimensional changes after VCS, in samples having different urea amounts.

2. MATERIALS AND METHODS

The effect of cold compaction pressure on the dimensional change and strength of the porous NiAl parts produced via VCS was investigated. The aim of the study was to determine the cold compaction pressure which provided the closest dimensional changes after VCS, in samples having different urea amounts. Close dimensional change is necessary to design a multi-stack sample having layers containing 20, 40 and 60 % porosity. Because a large difference in the dimensions change of different layers during VCS of a multi-stack sample would lead to cracking and separation of layers.

2.1 Materials Used

In this study, Nickel (Merck, <10 µm, 99%), aluminum (Alpha Aesar, <10 µm, 99.5%), preformed NiAl (Sarıyer and Çamurlu, 2022) powders and 150-300 micron sized urea (NH_4HCO_3

Merck) particles were used. It was observed that nickel powder consistsed of spherical agglomerates of around 10 micrometers in diameter. Aluminum powder consisted of irregular shaped non-faceted particles of 20 micrometer size. In order to preserve the shape of the produced samples in the volume combustion synthesis (VCS) process, 30 vol.% preformed NiAl powder was used as the thermal diluent in the samples. Preformed NiAl powder was obtained in this study through the reaction of Ni and Al powders. It was observed that preformed NiAl particles generally consisted of 5-10 micron sized particles.

2.2 Sample Preparation

Porous NiAl parts were prepared according to Equation (1). Nickel, aluminum, preformed NiAl and space holder urea particles were weighed on an analytical balance and mixed homogeneously in the mixing bowl. Preformed NiAl powder was used as thermal diluent since Equation (1) is highly exotermic. During VCS, the sample was seen to partially melt as a result of the high combustion temperature attained when thermal diluent is not utilized.

$$(a)\ \mathrm{Ni} + (a)\ \mathrm{Al} + (b)\ \mathrm{NiAl}\ (\text{preformed}) = (a+b)\ \mathrm{NiAl} \quad (1)$$

The powder mixture was transferred into a 10 mm diameter steel die was prepared in a cold press by applying three different pressures (50, 100, 200 and 300 MPa). Samples were coded according to their cold compaction pressure and their urea space holder particle content. For example, the sample code 50M20U refers to the sample which was cold compacted by using 50 MPa pressure and which contains 20% space holder urea particles. In order to dissolve the urea particles which were added to create pores, the parts were kept in distilled water for one day. They were dried at 65 °C for one day. Then, they were heated to 700 °C, at a heating rate of 10 °C/min in a tube furnace under argon atmosphere and volume combustion synthesis was accomplished.

2.3 Characterization

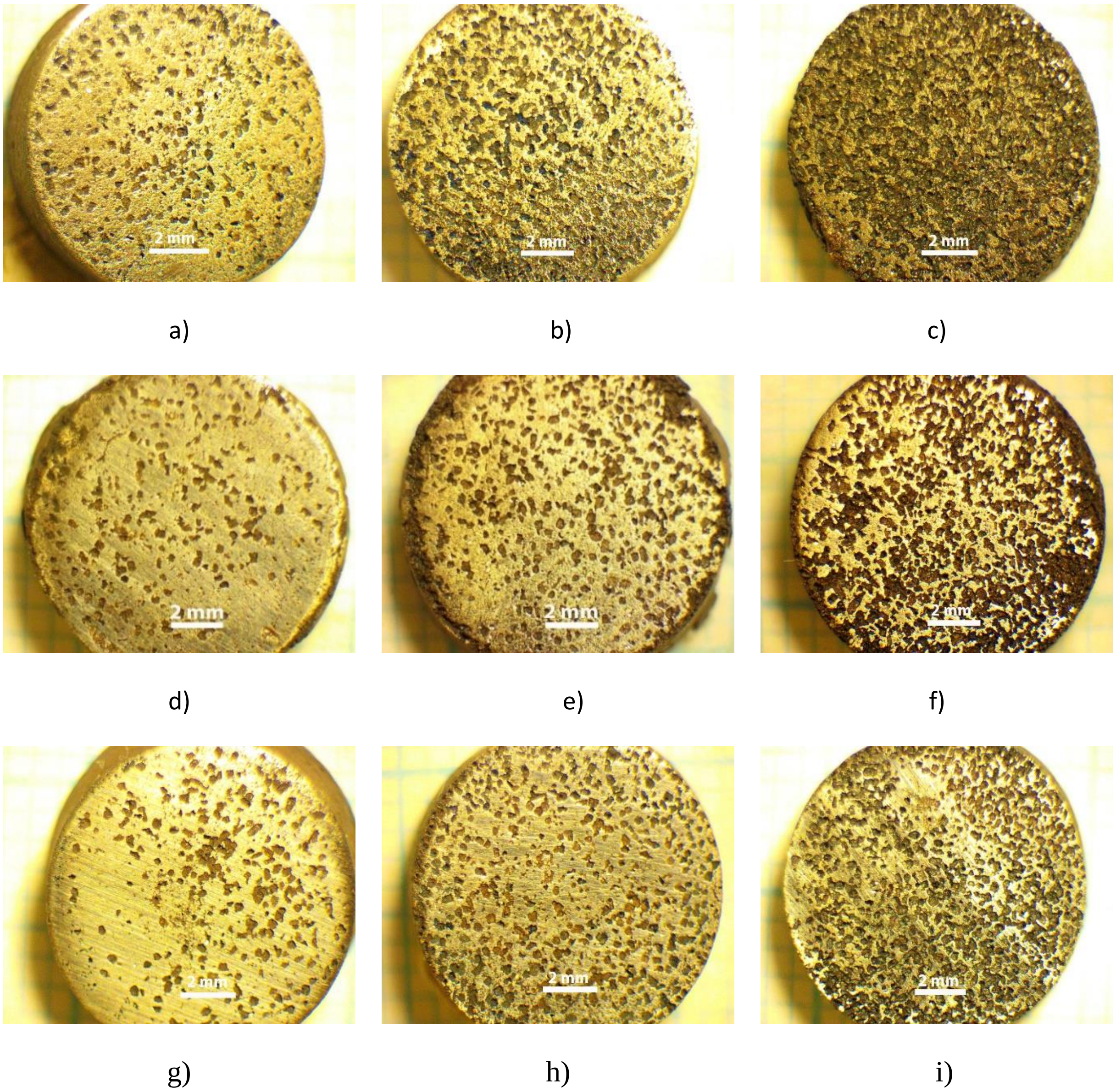
The parts obtained after volume combustion synthesis were subjected to compression test with a Shimadzu Autograph Universal Tension-Compression Device (50 kN) according to ISO 13314 standard. The compresive strength values of the materials produced with three different cold compaction pressures were measured. The produced porous NiAl parts were ground and polished on 600, 1200 and 3000 grit sandpaper. Then, the macro and micro structures of the samples were examined by stereo and optical microscope, respectively. The porosity values were calculated by using the dimensions and weight of the samples.

3. RESULTS AND DISCUSSION

3.1 Macrostructure

The macro images of the samples which were obtained by stereo microscope are presented in Figure 1. These images show the top circular side of the cylindrical samples, which were produced by using 20, 40 and 60% urea and by applying 50, 100 and 200 MPa cold compaction pressure. It was determined that the pores were generally homogeneously distributed and the volumetric pore ratio increased with increasing urea content. Parts in all systems were seen to retain their shapes. It was understood that the pore shape and dimensions were also preserved. It was found that the 30%

thermal diluent NiAl powder in the initial mixture was beneficial in this context. In a recent study which was conducted by Sarıyer and Çamurlu (2022), it was reported that addition of 30 % preformed NiAl was effective in preventing the melting of formed NiAl during VCS. Additionally, MgO powder was used as an inert thermal diluent in the synthesis of NiAl powder (Sarıyer and Çamurlu, 2024).



a) b) c)

d) e) f)

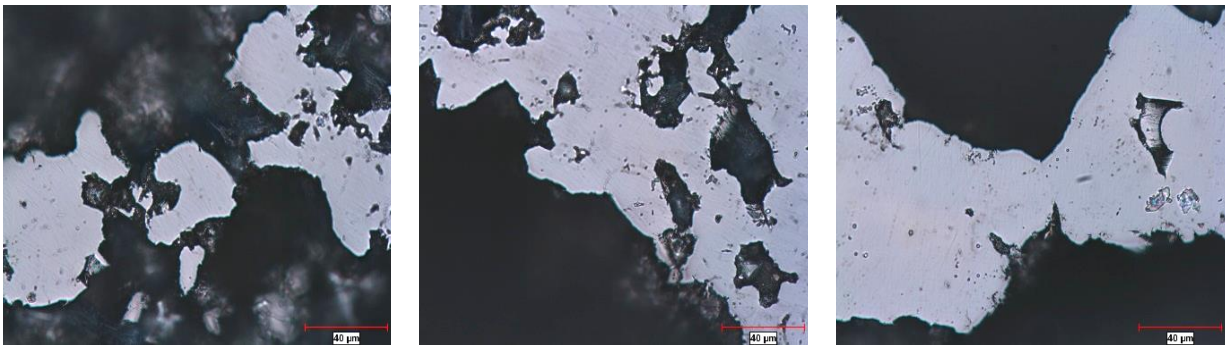
g) h) i)

Figure 1. Macrostructure (stereo microscope) images of samples produced with different cold compaction pressure a) 50M20U , b) 50M40U, c) 50M60U, d)100M20U, e)100M40U, f)100M60U g) 200M20U, h) 200M40U, i) 200M60U

3.2 Microstructure

The microstructure images of the parts, which were cold compacted with 50, 100 and 200 MPa pressure and produced by using 40% space holder urea, are presented in Figure 2 (a-c), respectively. The large and small dark regions in these micrographs are pores and the light gray parts are the NiAl cell (or pore) walls around these pores. Two kinds of porosity were observed in the microstructure. The large pores, which have the same dimensions of the urea particles are referred to as primary pores and the small ones which are on the cell walls are secondary pores ((Shu et al. 2019; Jiang 2021). The primary pores are formed by the spaces left by space-forming urea particles. The secondary pores were reported to form due to the presence of i) porosity in the green sample, ii) density difference between the reactant and product phases and also iii) due to Kirkendal effect (Jiao et al. 2021; Salamon and Mehrer, 2005).

In the microstructure examinations, it was observed that pore walls (or the cell walls) were less connected to each other and there were discontinuities in the post-VCS structure of the parts which were pressed at 50 MPa pressure. It was found that the cell walls of the parts pressed with 100 and 200 MPa were more continuous and less porous. This is believed to be due to the lower green density of the pellets when they are compacted with lower pressures. It can be seen in Table 1 that the amount of porosity before VCS was the highes when 50 MPa compaction pressure was applied. On the other hand, porosity of the green samples was seen to decrease with increasing compaction pressure (Table1). The discontinuous structure of the cell walls which was observed in the microstructure of the 50M samples can be explaned by the lower green density of these pellets. Similar resuls were observed in another system (FeAl) by Gao et al. (2015). It was proposed that the pores between the particles and the diffusion distance decreased by increasing the compaction pressure, thus lower porosity could be attained after VCS.



a) b) c)

Figure 2. Microstructure (optical microscope) images of the produced samples a) 50M40U, b) 100M40U c) 200M40U (500x)

3.3 Change in Porsity and Dimensions During VCS

Mean total porosity before and after VCS, and changes in diameter and height of the parts produced at different pressures (50, 100 and 200 MPa) are presented in Table 1 and Figure 3. It can be seen that the amount of porosity increased with increasing urea content in all the series, before and after VCS, as expected. Before VCS, it was observed that the total porosity amount in the green samples was higher than the amount of urea added. The difference is thought to be due to the fact that the Ni+Al parts (cell walls) of the green pellets are not fully dense and they contain pores (termed as the secondary pores) after cold compression. This difference decreases with increasing rate of space holder urea. For example, the porosity of the green sample which contained 20 % urea was 46.2%, whereas that of the sample which contained 60 % urea was 67.3, when they were compressed at 50MPa.

Before VCS, it was observed that the amount of pores in the green parts decreased with the increase in the cold compaction pressure. This effect was less pronounced in the green samples which contained 60% urea particles. These green samples (100M60U and 200M60U) exhibited similar porosity (about 62%) after compacting with 100 and 200 MPa pressure.

After VCS, an increase was observed in the average porosity of all samples. For example, the average porosity of the green pellet produced at 100 MPa cold compaction pressure and with 60 vol.% urea addition (100M60U) was 62.1% before VCS, while it was 66.9% after VCS. The increase in the secondary pores is believed to be responsible for the overall porosity increase after VCS. Secondary pores are believed to form in the cell walls primariliy due to two reasons: i) density difference between the reactant and product phases and also ii) due to Kirkendal effect. The density of Ni is 8.90 g/cm^3, and that of Al is 2.70 g/cm^3 whereas that of NiAl is 5.90 g/cm^3. Aluminum particles, having a large volume, occupy most of the volume of the reactant mixture. When the VCS reaction is completed, the formed phase with less volume (higher density) causesa shrinkage inside the pellet. This results in the formation of the pores due to density difference (Jiang 2021). The formation of the secondary pores is believed to take place due to Kirkendal effect. According to this phenomena, the difference in the diffusion coefficient of the reactant phases results in an insufficient exchange of atom among these phases. The species with the higher diffusion coefficient accumulates at a higher rate in the other species, whereas the species with the lower diffusion coefficient creates voids and pores inside the other species (Jiao et al. 2021; Salamon and Mehrer, 2005). Gao et al. (2015) reported that majority of the pores FeAl were seen to form at the locations which were originally occupied by Al particles.

Table 1. The percentages of change in porosity and dimensions of porous parts produced at different pressures (50 MPa, 100MPa, 200MPa) using 20%, 40% and 60% space holder urea (150-300 micron) (PBV: Porosity Before VCS, SD: Standard Deviation, CH: Change in Height CD: Change in Diameter, PAV: Porosity After VCS)

Sample Code	PBV (%)	SD (%)	CH (%)	SD (%)	CD (%)	SD (%)	PAV (%)	SD (%)
50M20U	46.2	0.1	-4.79	0.93	-3.33	0.47	47.5	0
50M40U	55.4	0.3	-3.60	0.03	-1.63	0.03	58.4	0.3
50M60U	67.3	0.1	-1.91	0.67	-2.44	1.75	69.8	1.3
SD			1.45		0.85			
100M20U	41.1	0.4	-4.40	0.08	-1.94	0.29	44.5	0.4
100M40U	51.9	0.1	-3.59	0.06	-1.46	0.73	55.1	0.2
100M60U	62.1	0.3	-1.22	0.21	-0.57	0.52	66.9	1.5
SD			1.65		0.69			
200M20U	34.3	0.2	-1.00	0.83	-0.25	1.56	44.2	2.1
200M40U	47.4	0.2	-1.38	2.34	-0.70	2.04	56.2	2.9
200M60U	62.0	0	-0.69	1.40	-1.08	0.69	66.0	0.3
SD			0.35		0.42			

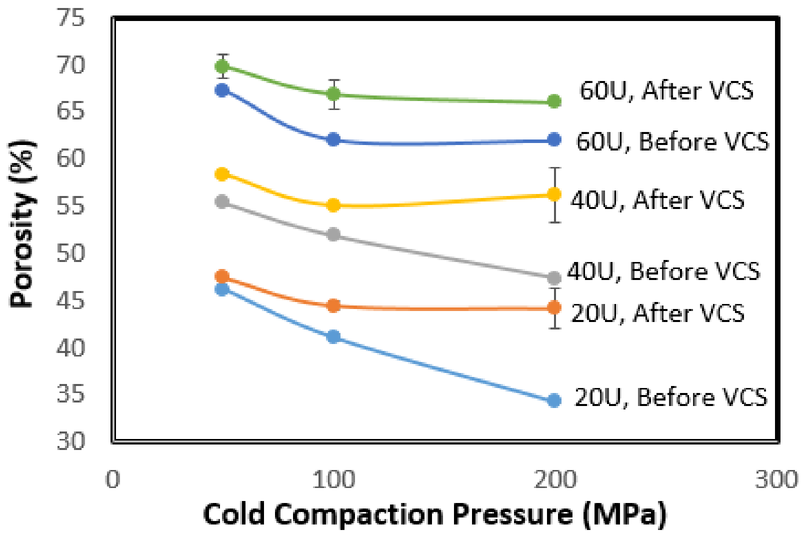
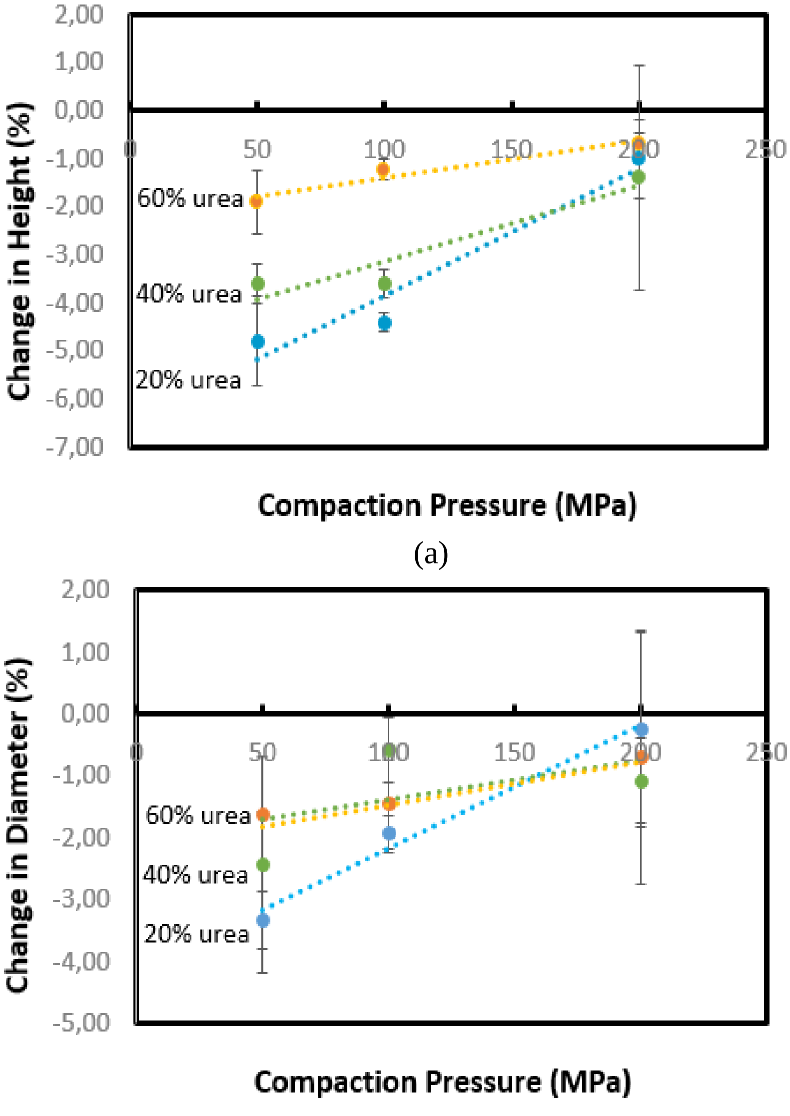


Figure 3. % changes in the porosity before and after VCS of samples produced at 50, 100 and 200 MPa cold compaction pressure using 20, 40 and 60 vol.% space holder urea

The changes in height and diameter of parts during VCS are presented in Figure 4 (a) and (b) as a function of cold compaction pressure. When the size changes of the parts produced by applying different cold compaction pressure are examined, it is understood that the change in height is below 4.8% and the change in diameter is below 3.3% in all systems. These largest dimensional change values occurred in the parts pressed with 50 MPa. In both graphs, it is seen that with increasing cold compaction pressure, large negative values approach to zero, indicating that the dimensional change decreases when the cold compaction pressure increases. During VCS, as a result of the increase in temperature, partial sintering of the product pellet occurs. Sintering involves diffusion, mass transport and decrease in the porosity. In powder metallurgy, during sintering the external dimensions of the sample decreases as a result of the decrease in the internal porosity (German, 2005). Therefore, when the porosity of the green sample is high, the shrinkage or the change in the dimensions of the sample is higher. The green samples which were produced by lower compaction pressure were seen to have higher amount of porosity before VCS (Table 1). Therefore, higher amount of shrinkage in the samples which were produced by lower compaction pressure is in accord with this explanation. Amount of shrinkage decreases when high compaction pressure is applied.

The change in height and diameter values of the parts containing different ratios of space holder urea which were compacted with 200 MPa are closer to each other than the parts which were compacted with 50 and 100 MPa. This situation is also understood from the standard deviation values of change in dimensions given for each compaction pressure value in Table 1. When the change in height and diameter are examined, the smallest absolute values (0.35 and 0.42, respectively) for the parts containing different ratios of urea were found in the samples compacted with 200 MPa.

A large difference in the dimensionsional change of different layers during VCS of a multi-stack sample would lead to cracking and separation of layers. Similar dimensional change is necessary to design a multi-stack sample having layers containing 20, 40 and 60 % porosity. It was seen from the dimensional changes that promising results were obtained for design of multi-stack samples.



(a)

(b)

Figure 4. % changes in a) height and b) diameter during VCS of samples produced at 50, 100 and 200 MPa cold compaction pressure using 20, 40 and 60 vol.% space holder urea

3.4 Mechanical Properties

In Table 2 and Figure 5, strength values of samples cold compacted at different pressures (50, 100, 200 MPa) using 20, 40 and 60% space holder urea (150-300 microns) after VCS are presented. The samples which were cold pressed with 300 MPa pressure sometimes contained cracks after they were taken out of the die after cold pressing or after VCS. Therefore, they could not be subjected to characterization. This was attributed to the high springback effect that formed when high compaction pressure was applied (German, 2005).

The strength was observed to decrease with the increase in the amount of urea in each applied pressure group. The decrease in the strength was attributed to the increase in the amount of porosity. The density of a material is a chief trait to foresee its mechanical properties and permeability. As pointed out by Smith and Brown (1985) that density decreases (and porosity increases), mechanical properties decline and also permeability increases. It was stated that the mechanical traits of porous substances, like ductility, strength and fatigue life basically rely on the porosity and manufacturing method (Lenel, 1980; Eisenmann and Morgan, 2015).

It was observed in Table 1 that the amount of porosity was higher in the samples after VCS, when they were compacted with lower compaction pressure. Strength of materials was reported to decrease with increasing porosity due to the decrease in the load bearing cross section of the material when porosity is present. In addition, pores can act as stress concentrating regions under tensile forces, which also results in a decrease in the strength of the material (German, 2005). When the samples containing the same amount of space holder urea were cold compacted at different pressures, it was observed that the strength increased with increasing compacting pressure (Figure 5). This is due to the fact that higher compaction pressure results in lower amount of porosity after VCS, as can be seen in Table 1. It can be seen in Figure 5 that the strength values of the parts produced with 200 MPa cold compaction pressure are the highest. This result is in accord with the explanation that higher compaction pressure results in lower amount of porosity, which leads to higher strength.

Table 2. Strength values of samples cold pressed at different pressures (50, 100 and 200 MPa) and VCS applied using 30 vol.% preformed NiAl powder and 20, 40, 60% space holder urea

Cold Press Pressure (MPa)	Sample Code	Strength	Standard deviation
50	50M0U	216.76	94.43
	50M20U	109.90	16.89
	50M40U	56.90	3.14
	50M60U	18.67	0.27
100	100M0U	311.49	22.44
	100M20U	181.77	1.60
	100M40U	84.64	0.73
	100M60U	33.36	5.03
200	200M0U	290.54	75.98
	200M20U	218.86	29.47
	200M40U	91.27	2.04
	200M60U	34.06	1.43

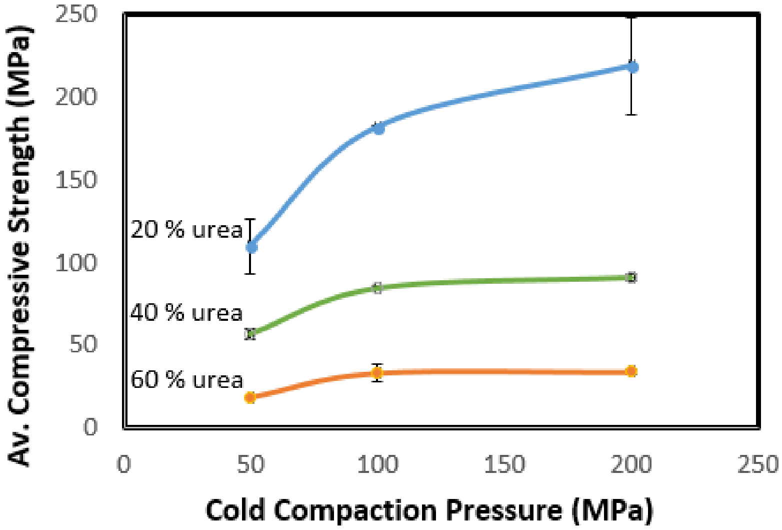


Figure 5. Strength values of the samples (after VCS) which contained 20, 40 and 60% space holder urea, as a function of compacting pressure

4. CONCLUSION

The results obtained in this study are summarised below:

- Porous NiAl samples were obtained by using 20, 40, 60 vol.% space holder urea particles and pressures of 50, 100, 200 and 300 MPa were applied by cold press to the mixtures formed by nickel, aluminum, preformed NiAl powders and urea particles. The samples which were cold pressed with 300 MPa could not be characterized since they contained cracks. This was attributed to the high springback effect that formed when high compaction pressure was applied.
- The dimensions, macrostructure, microstructure and compressive strength of the parts produced with different cold compaction pressures were analyzed after volume combustion synthesis (VCS). The compressive strength values of the porous NiAl samples after VCS were the highest when the samples were cold pressed with 200 MPa as compared to lower cold compaction pressures. This was attributed to the lower porosity content of these samples, as compared to the ones obtained with lower compaction pressures.
- The aim of the study is to determine the cold compaction pressure which provides the closest dimensional changes after VCS, in samples having different urea amounts. Minimum and the closest dimensional change values after VCS were obtained with in samples having different urea contents, when they were cold pressed at 200 MPa. This was attributed to the lower shrinkage of these samples during VCS, as a result of their lower porosity amount. Lower porosity was achieved with high compaction pressure. Therefore, it was concluded that 200 MPa pressure was optimum for cold pressing a multi-stack sample having layers of different urea content.

5. ACKNOWLEDGEMENTS

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6. CONFLICT OF INTEREST

Author(s) approve that to the best of their knowledge, there is not any conflict of interest or common interest with an institution/organization or a person that may affect the review process of the paper.

7. AUTHOR CONTRIBUTION

Gülizar Sarıyer contributed in experimental preparation and characterization of the samples. Hasan Erdem Çamurlu contributed in design of the experiments, and analysis and interpretation of the results.

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