PAPER • **OPEN ACCESS**

The influence of direct powder rolling parameters on the properties of aluminium strip

To cite this article: K Kunene *et al* 2021 *IOP Conf. Ser.: Mater. Sci. Eng.* **1147** 012017

The influence of direct powder rolling parameters on the properties of aluminium strip

K Kunene[1], C P Bemont*[1], L A Cornish[2], A Dittes[3], T Grund[3], P Sinclair[1] and T Lampke[3]

[1] University of KwaZulu-Natal, Discipline of Mechanical Engineering, Durban, 4001, South Africa
[2] DSI-NRF Centre of Excellence in Strong Materials; School of Chemical and Metallurgical Engineering, University of the Witwatersrand, Johannesburg, South Africa
[3] Chemnitz University of Technology, Materials and Surface Engineering Group, 09125 Chemnitz, Germany

*e-mail: bemontc@ukzn.ac.za

Abstract. Direct powder rolling (DPR) can be used to create metallic sheet at a reduced cost compared to traditional methods. The process is particularly relevant for metals that are beneficiated to a powder form. The DPR process, however, is not yet optimised and the material properties corresponding to DPR sheet do not compare well with sheet produced from rolled metallic billet. This study explores the effect of DPR input parameters on DPR aluminium sheet material properties. Roll speed, roll gap, sintering time and sintering temperature are shown to have an impact on the density, microstructure, hardness and ductility of DPR aluminium strip. It is shown that a low roll speed and a small roll gap produced the most dense strip. A roll gap of 0.7 mm and roll speed of 0.366 rpm produced green strip with a density of 90% of cast aluminium while a roll gap of 0.8 mm and roll speed of 0.73 rpm produced green strip with a density of 79.6% of cast aluminium. In addition, a Vickers hardness analysis and a three-point bending test show that increasing the sintering temperature of the green DPR strip will produce softer aluminium strips with higher ductility, as expected.

1. Introduction

Powder metallurgy has been identified as a process capable of reducing beneficiation costs of metal products by removing some of its process complexities and reducing the required energy input. Direct powder rolling (DPR) is a powder metallurgical beneficiation process used to create sheet metal by compacting metallic powders using a rolling mill and further densifying the product by sintering. This process, however, is yet to be optimised and sheet metal produced using this method has lower density, strength, ductility and formability when compared to sheets produced conventionally despite having good tensile elongation [1]. Optimising this process can therefore have a major impact on the metal working industry by making formally exotic materials readily available for applications that would have previously used cheaper material such as steel. This is especially of interest for titanium, which is among Earth's most abundant metals although is not often used in industry due to the high costs associated with its beneficiation [2].

This study is focused on the investigation of the effects of varying the input parameters of aluminium DPR only. These input parameters are to be manipulated in order to improve the product's mechanical properties such as density, formability and strength. It is believed that the findings from this research can be adapted to be used effectively for other materials such as titanium. Aluminium's relatively low cost, low ultimate tensile strength and low melting point require less time and resources to perform multiple DPR tests making it an ideal material for DPR research. DPR process parameters known to impact the properties are powder morphology, roll size, roll speed, roll surface roughness, roll gap, sintering time and sintering temperature, although how these parameters can be optimised to create competitive properties is not well understood [3]. This study explores the effects that roll speed, roll gap, sintering time and sintering temperature have on the density, hardness and bending strength of DPR strips.

Roll speed refers to the rotational speed of the rolling mill rolls. It has been found that at high rolling speeds, the strip produced is of inferior quality, particularly of poor density when compared to strips produced at lower speeds. The reduction in density due to roll speed can be attributed to the shorter time in which each particle of powder mass is subjected to pressure. Additionally, as the roll speed increases, the velocity of the air escaping the powder does not increase proportionally, resulting in lower density [4]. It has also been implied that an increase in roll speed will affect the position of the nip angle negatively, however, no experimental evidence exists that can support this. It has been found that when powder is slowly rolled at a given roll gap, the density and thickness of green (not sintered) strip remain constant up to a certain speed named the transition speed. Above this speed these properties decrease as the roll speed increases due to the factors listed above [5, 6].

For a given powder and roll combination, a decrease in roll gap increases the roll separation force, thus increasing the strip density. This occurs because at high pressure, more particle deformation is present and there is an increase in contact points within the powder mass [7]. Increasing the roll gap increases the DPR sheet thickness, but the density of the sheet decreases resulting in only a moderate increase in mass flowrate. This produces low density and consequently low ductility and low strength green sheets [4, 8]. The density will continue to increase with a decrease in roll gap until the roll gap approaches a point where the roll gap is close to the powder particle size [9].

The green strip produced from DPR is porous with only the pressure induced cold welds holding the strip together. Sintering is a heat treatment process, lowering the surface energy of the powder particles, allowing the cold welds to form necks [10]. This improves the physical and mechanical properties of green compacted metal powders. Mechanisms such as vapour transport and surface diffusion, transport material from the surface to form the neck. These mechanisms do not change the distance between the centres of the particles, thus they contribute little to densification, however, a small increase in density is observed for an increase in temperature [8]. An increase in sintering time will also increase the density of aluminium strip until grain growth begins resulting in a decrease in strip density [10].

Additionally, sintering relieves the strain hardening occurring during cold working and significantly increases the ductility, reducing the hardness of the strip [11]. In order to achieve higher physical and mechanical properties of the powder metallurgy product, the sintered strip can be cold rolled multiple times, annealing the product between compactions to relieve cold working stresses applied during rolling [8].

2. Experimental apparatus and methodology

2.1. Powder material properties

The aluminium powder used to create aluminium strips in this research was purchased from Insimbi Alloys Suppliers Pty (Ltd) in South Africa. The powder has a particle size distribution of 75 MIC-90.04% and its angular morphology is depicted in the scanning electron microscopy (SEM) image in Figure 1. The powder is 98.7% pure aluminium by weight.

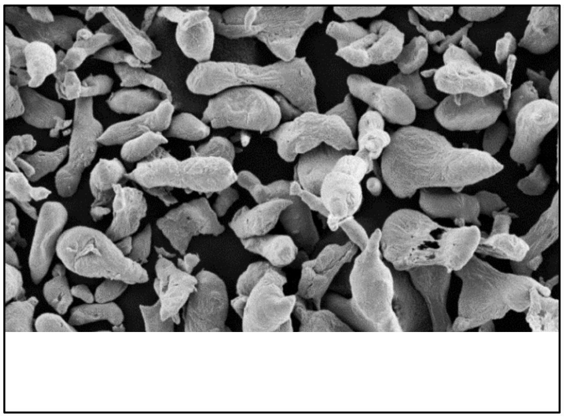

Figure 1. SEM-SE image showing the morphology of the aluminium powder.

2.2. Direct powder rolling parameters

A vertical rolling mill consisting of a powder feeder and 200 mm diameter rolls was used to directly powder roll the aluminium into continuous green strips. The surface roughness of the rolls was not measured but remained constant throughout experimentation. The rolling mill has adjustable roll speed and roll gap, allowing study of the effects of varying these two rolling parameters in DPR. The continuous strips were trimmed into test specimens, seen in Figure 2, using a metal file.

Green DPR strips were produced initially using a roll speed of 0.366 rpm and roll gaps of 0.7 mm, 0.75 mm and 0.8 mm. The roll speed was then doubled to 0.73 rpm and strips were produced for the same roll gaps. Strips produced with roll gaps less than 0.7 mm were not analysed due to severe cracking occurring when rolled below this limit. Green strips rolled above a 0.8 mm roll gap had very little density and crumbled when handled.

Figure 2. Trimmed green aluminium strips produced by DPR process.

2.3. Sintering parameters

Sintering was performed by heating the green aluminium strips using a Brother XD-1600A inert gas muffle furnace in three heating phases. The first phase involved heating the furnace at a constant rate of 10 K per minute until the peak temperature was reached. The temperature was then held constant at its peak for a predetermined time period. The third phase involved allowing the furnace to cool to room temperature at 10 K per minute.

In order to reduce the amount of oxidation occurring during sintering, all sintering occurred in an argon gas environment. This environment was achieved by pulling a vacuum of 0.05 MPa from the furnace before refilling it with 99.9 % pure argon gas. This process was conducted a total of five times before the gas was allowed to run at three litres per minute continuously. The three peak temperatures investigated along with their sintering hold times can be observed in Table 1.

Table 1. Sintering process parameters.

Sintering peak temperature (°C)	Phase 1 Heating time (min)	Phase 2 Peak temperature time (min)	Phase 3 Cooling time (min)	Total sintering time (min)
500	50	60	50	160
550	55	45	55	155
580	58	30	58	146

2.4. Post sintering densification parameters

Once sintered, the properties of the aluminium strips were further manipulated by re-rolling the strips and annealing the products. All re-rolling was performed at 0.366 rpm on strips initially rolled at 0.366 rpm. Re-rolling was conducted at a roll gap 0.5 mm less than each strip experienced during DPR, densifying the strips by further cold working them. Note that 0.5 mm was the maximum roll gap change that did not result in excessive cracking of the strips. Re-rolled strips were then annealed to further increase their density and release the internal stresses that occurred due to cold working during re-rolling. Annealing times and temperatures matched those experienced during sintering for each strip and therefore follow the procedures outlined in Table 1. Strips that have been re-rolled and annealed will henceforth be labelled, densified strips.

3. Experimental analysis and results

3.1. Density analysis

The manipulation of DPR and sintering parameters largely focuses on increasing the density of the final product, consequently improving its mechanical properties [7]. The aim is to achieve a final density that is close to that of wrought aluminium at 2.7 g/cm^3.

Table 2 depicts the impacts that a change of roll gap and a change of roll speed have on the density of green DPR strips. It is observed that a smaller roll gap produces strip with a higher density. It is also clear that the slower roll speed of 0.366 rpm results in a higher strip density than 0.73 rpm. This means that a roll speed of 0.73 is above Tundermann and Singer's transition speed and thus, too high for this powder and roll combination [5]. It is unknown whether a 0.366 rpm roll speed is above or below this transition speed. More experimentation needs to be completed in order to find the transition roll speed for time efficient DPR of the highest density for this powder and roll combination.

Table 2. Green aluminium strip density.

Roll gap (mm)	0.366 rpm roll speed Density (g/cm^3)	0.366 rpm roll speed (% of cast Al.)	0.73 rpm roll speed Density (g/cm^3)	0.73 rpm roll speed (% of cast Al.)
0.7	2.43	90	2.32	85.9
0.75	2.36	87.4	2.25	83.3
0.8	2.28	84.4	2.15	79.6

Following initial direct powder rolling, sintering of the green strips further increased their density. Table 3 represents the densities for direct powder rolled aluminium, rolled at 0.366 rpm, when green and when sintered at 500 °C, 550 °C and 580 °C respectively. The sintering process is in accordance with the procedure outlined in Table 1. Percentage change 1 uses the density of wrought aluminium, 2.7 g/cm^3, as the denominator while Percentage change 2 uses the measured green density as the denominator.

The results from these tables show that density is not greatly improved during sintering with a maximum density change from green of only 0.41% which was achieved by sintering 0.7 mm roll gap strips to 580 °C. The tables show that higher green densities result in a higher density change during sintering. In addition, a higher density is achieved by sintering at a higher temperature. Strips sintered to 550 °C and 580 °C, however, show very similar average density change values. A conclusion drawn from this is that high densities can be achieved by sintering strips at lower temperatures for a longer time.

Table 3. Sintered aluminium strip density.

Roll gap (mm)	Green density (g/cm^3)	Sintered density (g/cm^3)	Density increase 1 (% of wrought)	Density increase 2 (% of green)
Sintered (500 °C) aluminium strip density:				
0.7	2.40	2.406	0.222	0.250
0.75	2.37	2.373	0.111	0.127
0.8	2.28	2.282	0.074	0.088
Average			0.136	0.155
Sintered (550 °C) aluminium strip density:				
0.7	2.45	2.458	0.296	0.327
0.75	2.37	2.376	0.222	0.253
0.8	2.3	2.306	0.222	0.261
Average			0.247	0.280
Sintered (580 °C) aluminium strip density:				
0.7	2.44	2.45	0.370	0.410
0.75	2.38	2.386	0.222	0.252
0.8	2.28	2.284	0.148	0.175
Average			0.247	0.279

Further density analyses were conducted on densified strips after which they were rolled and sintered, were re-rolled and annealed according to the procedure outlined in Section 0. Table 4 presents density data for strips of varying roll and re-rolling gaps when green and when densified at 500 °C, 550 °C and 580 °C respectively.

The density results for densified DPR strips no longer show a significant increase with an increase in temperature, with the average density increases all showing very similar results. This implies that the maximum increase in density that can be achieved during sintering has been reached and the leading source of density increase during densification is due to re-rolling. Further re-rolling steps should be explored to achieve even denser strips. Strips previously rolled at a 0.75 mm roll gap and re-rolled at a 0.7 mm roll gap consistently produced the highest total increase in density.

Table 4. Sintered, re-rolled and annealed aluminium strip density.

Roll and re-roll gap (mm)	Green density (g/cm^3)	Densified density (g/cm^3)	Density increase 1 (% of wrought)	Density increase 2 (% of green)
Sintered (500 °C), re-rolled and annealed aluminium strip density:				
0.7 and 0.65	2.452	2.468	0.593	0.653
0.75 and 0.7	2.394	2.412	0.667	0.752
0.8 and 0.75	2.304	2.318	0.519	0.608
Average			0.593	0.671
Sintered (550 °C), re-rolled and annealed aluminium strip density:				
0.7 and 0.65	2.412	2.426	0.519	0.580
0.75 and 0.7	2.334	2.35	0.593	0.686
0.8 and 0.75	2.294	2.308	0.519	0.610
Average			0.543	0.625
Sintered (580 °C), re-rolled and annealed aluminium strip density:				
0.7 and 0.65	2.424	2.44	0.593	0.660
0.75 and 0.7	2.352	2.372	0.741	0.850
0.8 and 0.75	2.298	2.312	0.519	0.609
Average			0.617	0.707

3.2. Optical image analysis

A Nikon Eclipse MA200 was used to view the impact that sintering temperature has on the microstructures of aluminium strips. The strips represented were rolled at 0.366 rpm with a roll gap of 0.75 mm although the results were consistent for all three roll gaps. Figure 3 shows a green aluminium DPR strip, Figure 4, a strip sintered to 500 °C, Figure 5, a strip sintered to 550 °C and Figure 6, a strip sintered to 580 °C. Figure 7 shows an optical image of a strip that has been re-rerolled with a roll gap of 0.7 mm roll gap and annealed at 580 °C. The images show a small decrease in pore size with an increase in sintering temperature and no grain growth when compared to the green strip.

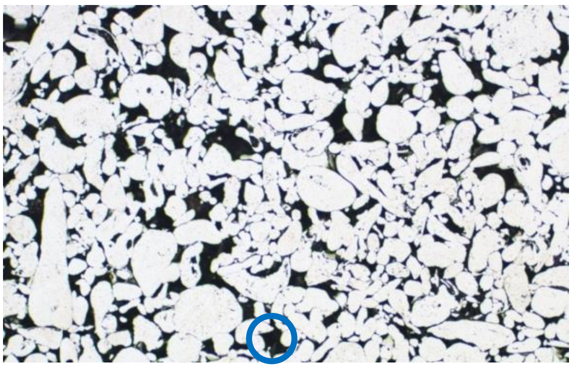

Figure 3. Optical image of a green DPR aluminium strip (pores depicted black, one is exemplarily marked by blue circle).

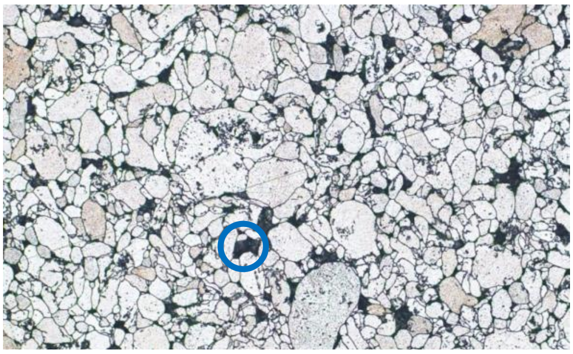

Figure 4. Optical image of DPR aluminium strip sintered at 500 °C (pores depicted black, one exemplarily marked by blue circle).

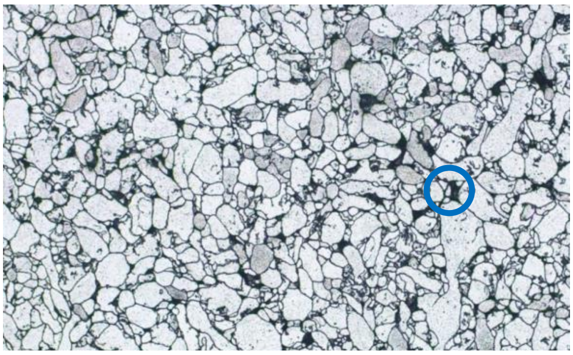

Figure 5. Optical image of DPR aluminium strip sintered at 550 °C (pores depicted black, one is exemplarily marked by blue circle).

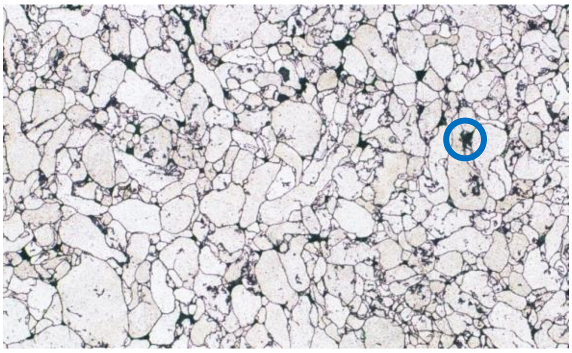

Figure 6. Optical image of DPR aluminium strip sintered at 580 °C (pores depicted black, one is exemplarily marked by blue circle).

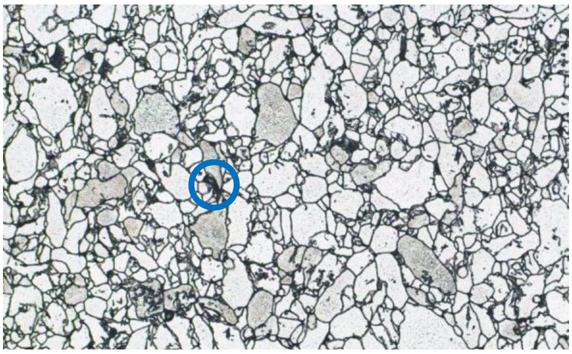

Figure 7. Optical image of DPR aluminium strip sintered at 580 °C, rerolled at 0.7 mm roll gap and re-sintered at 580 °C (pores depicted black, one exemplarily marked by blue circle).

3.3. SEM analysis

The effect that roll gap has on the microstructure on the surface of sintered DPR strips was analysed using a Zeiss Ultra plus FE scanning electron microscope. DPR aluminium strips were rolled at three different roll gaps, all sintered at 580 °C and characterized by SEM. Figure 8 shows a strip rolled at a roll gap of 0.7 mm, Figure 9 a roll gap of 0.75 mm and Figure 10 a roll gap of 0.8 mm. Smaller pores and larger pressure welds coupled with neck formation is evident in the images of the strips rolled at smaller roll gaps.

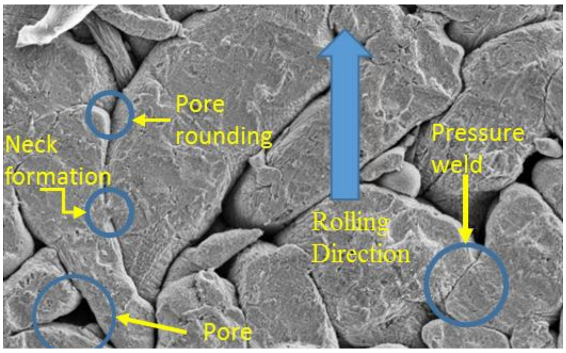

Figure 8. SEM-SE image of an aluminium strip surface after being rolled at 0.7 mm and sintered at 580 °C.

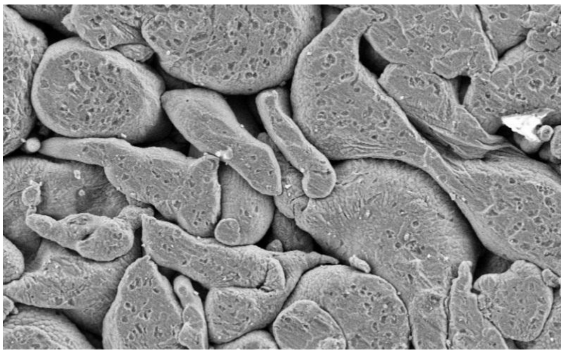

Figure 9. SEM-SE image of an aluminium strip surface after being rolled at 0.75 mm and sintered at 580 °C.

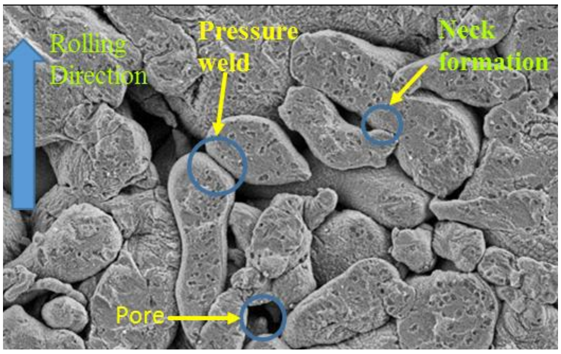

Figure 10. SEM-SE image of an aluminium strip surface after being rolled at 0.8 mm and sintered at 580 °C.

Densified strips that had been re-rolled and annealed were then analysed using the SEM. The following figures show strips that have been direct powder rolled, sintered and densified according to the procedure in Section 0. Figure 11 shows a densified strip originally rolled at a roll gap of 0.7 mm, Figure 12 a strip originally rolled at a 0.75 mm roll gap and Figure 13, a strip originally rolled at a 0.8 mm roll gap. All strips were sintered and annealed at 580 °C according to Table 1. These images show a clearer reduction in pore size than non-densified strips with densified strips originally rolled at 0.7 mm roll gap showing almost no surface pores. The pressure welds and necks have grown so much that in some cases, particles can no longer be identified as separate.

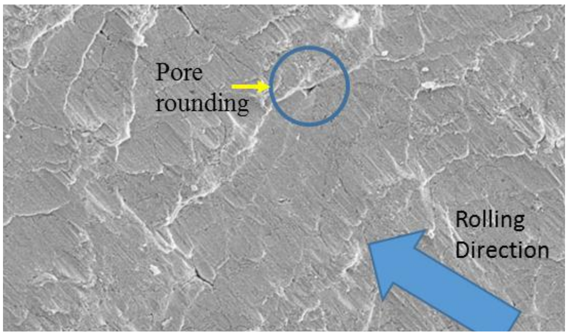

Figure 11. SEM-SE image of an aluminium strip surface after being rolled at 0.7 mm and sintered at 580 °C, then re-rolled at 0.65 mm roll gap then re-sintered at 580 °C.

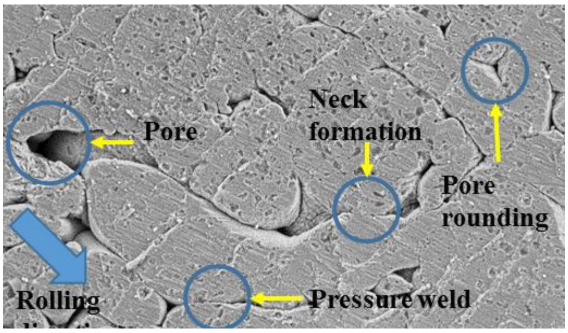

Figure 12. SEM-SE image of an aluminium strip surface after being rolled at 0.75 mm and sintered at 580 °C, then re-rolled at 0.7 mm roll gap then re-sintered at 580 °C.

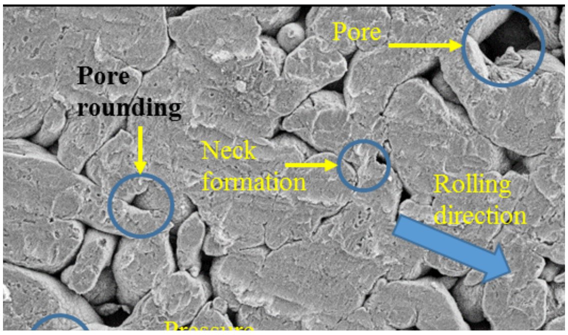

Figure 13. SEM-SE image of an aluminium strip surface after being rolled at 0.8 mm and sintered at 580 °C, then re-rolled at 0.75 mm roll gap then re-sintered at 580 °C.

3.4. Micro hardness analysis

The Vickers micro hardness of the aluminium samples was investigated by an applied force of 1 kgf using an Emco Test Durascan. Prior to indentation, the samples were mounted in bakelite resin with their rolled surface exposed and polished. The top three rows of data in Table 5 show the Vickers hardness for aluminium DPR strips rolled at 0.366 rpm with varying roll gaps and sintering temperatures. The bottom three rows of Table 5 show the Vickers hardness for aluminium DPR strips that have been sintered, and densified according to the procedure explained in Section 0. Each value in the table was achieved by averaging two readings taking from different places on each of three strips making a total of six independent readings per value.

In aluminium DPR, high hardness values are not desired because increased hardness is associated with decreased ductility. High ductility is a desired quality in aluminium DPR allowing higher levels of formability of the aluminium sheet.

Table 5 outlines that similar to density, hardness increases with decreasing roll gap. This is due to the higher pressure the rolls exert on the aluminium powder resulting in a higher level of cold working of the material. The table shows that this hardening is relieved during sintering and higher sintering temperatures resulting in a greater reduction of hardness. The values of hardness show that there is a limit to the reduction of hardness that can be achieved during annealing with this asymptotic value being close to 33 HV.

Table 5. Micro hardness of DPR aluminium strips.

Roll gap (mm)	Green (HV)	Sintered 500 °C (HV)	Sintered 550 °C (HV)	Sintered 580 °C (HV)
0.7	57 ± 3.10	52 ± 2.45	48 ± 1.2	44 ± 1.05
0.75	49 ± 2.03	40 ± 1.64	36 ± 2.51	34 ± 2.80
0.8	44 ± 2.30	38 ± 3.22	35 ± 2.34	33 ± 3.05
0.7 and 0.65		40 ± 2.36	37 ± 2.45	35 ± 2.74
0.75 and 0.7		40 ± 2.30	38 ± 3.00	37 ± 3.10
0.8 and 0.75		39 ± 2.21	36 ± 1.59	35 1.73

3.5. Bending analysis

A common disadvantage of powder metallurgical products is their poor strength when a bending load is applied. It is therefore of importance to prioritise maximising strip strength in bending in order to produce sheet that can easily be formed. In agreement with DPR literature, the specimens being tested displayed low resistance to bending and were thus analysed adapted from 3-point bending analysis standards using a bearing distance of 120 mm. The results obtained can be used for comparative analysis within this study only. The maximum deflection of the specimens was measured while 50-gram weights were incrementally loaded until failure of the specimen.

The bending tests were all conducted using aluminium strip that was rolled at 0.366 rpm. The effect that roll gap and sintering temperature have on the maximum deflection are of interest in order to maximise strip ductility. Figure 14 is a bar graph showing the maximum deflection that each strip experienced before failure and Figure 15 shows the maximum loads each strip experienced before failure. In contrast to strip density, strip ductility is largely affected by sintering while roll gap has a lesser effect on ductility. A high sintering temperature resulted in the maximum deflection of strip sintered at 580 °C to more than double the deflection of a green strip.

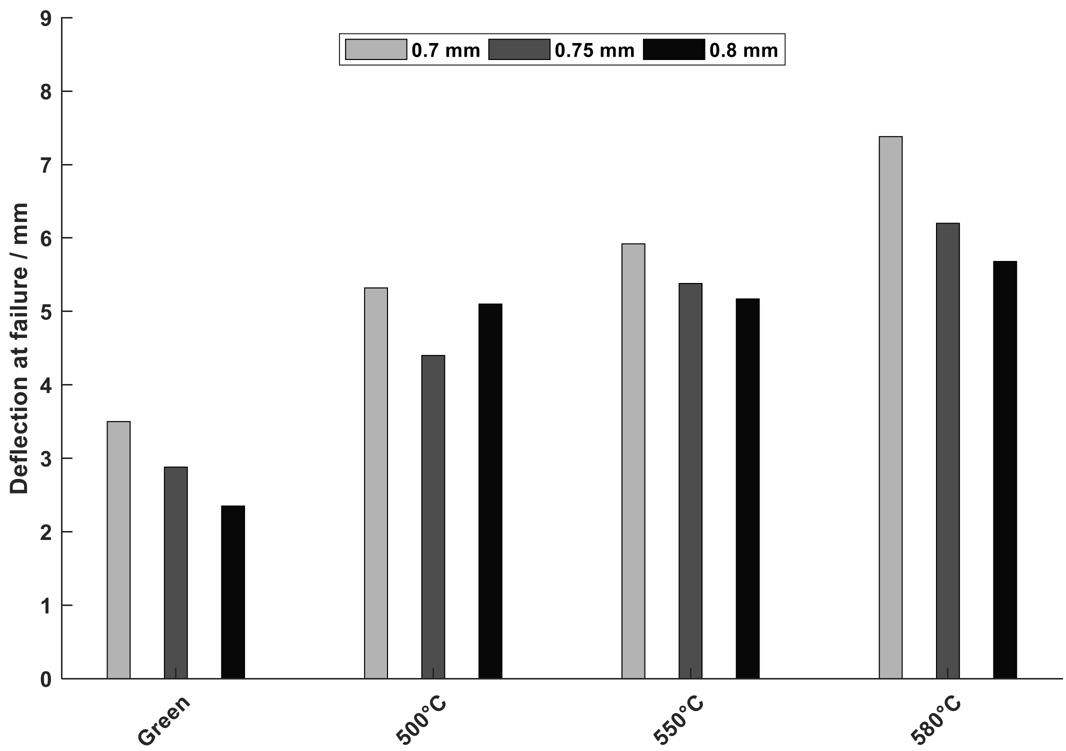

Figure 14. Bar graph representing aluminium strip maximum deflection at failure.

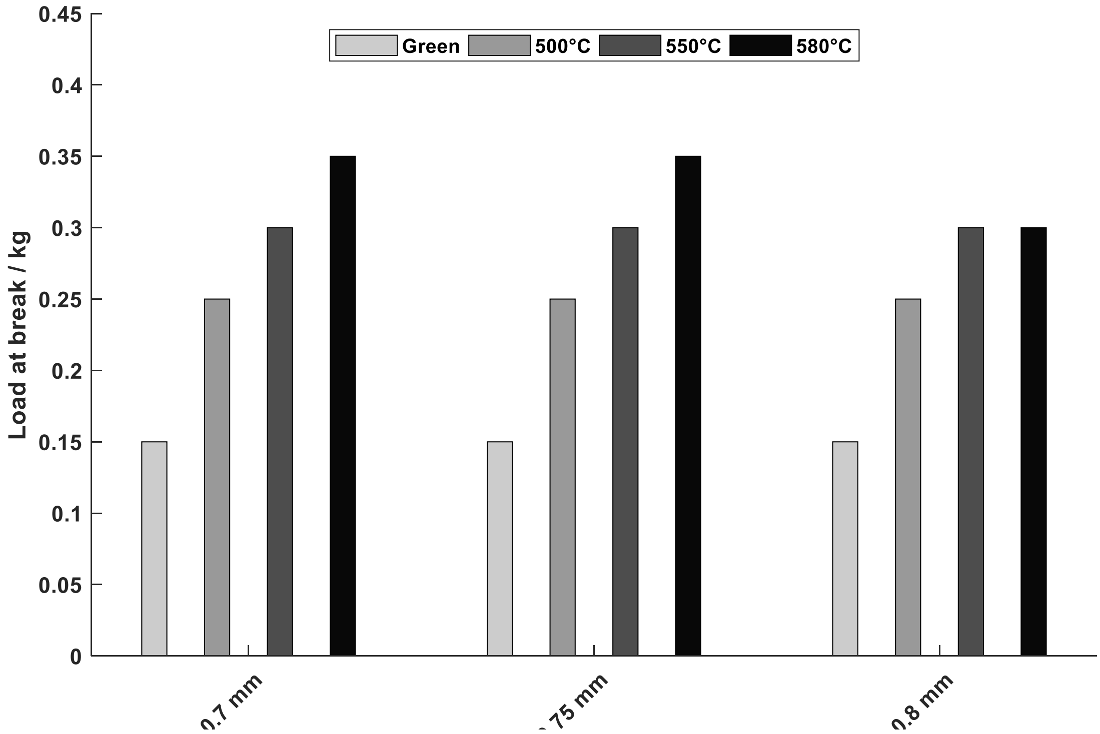

Figure 15. Bar graph representing aluminium strip maximum load at failure.

4. Conclusions

In this study, aluminium DPR strips were successfully created with varying roll speed, roll gap, sintering temperature and sintering time. The effects that each parameter had on the density, microstructure, hardness and ductility were explored. It can be concluded that the density is largely dependent on the rolling conditions with smaller roll gaps and slower roll speeds produces denser strip. Although only a small difference was recorded, higher sintering temperatures produced strip with higher density. The density can be further improved by re-rolling and annealing the strip. Furthermore, larger pressure welds are evident in strips rolled at smaller roll gaps. Re-rolling and annealing the strips produces much larger pressure welds and necks. The hardness of green strip increases with a decrease in roll gap. The cold working resulting in these pressure welds are relieved by sintering. Higher sintering temperatures had a larger influence in relieving this strain hardening. Ductility analyses in the form of a three-point bend test proved that the largest influence on ductility is the sintering conditions. High sintering temperatures produced significantly higher strip ductility.

References

[1] Peter W H, Muth T, Chen W, Yamamoto Y, Jolly B, Stone N A, Cantin G M D, Barnes J, Paliwal M, Smith R, Capone J, Liby A, Williams J and Blue C 2012 Titanium sheet fabricated from powder for industrial application *Journal of Materials* **64** 566-571

[2] Cantin G M D, Kean P L, Stone N A, Wilson R, Gibson M A, Yousuff M, Ritchie D and Rajakumar R 2011 Innovative consolidation of titanium and titanium alloy powders by direct rolling *Powder Metallurgy* **54** 188-192

[3] Johanson J R 1965 A rolling theory for granular solids *Journal of applied mechanics* **32** 842-848

[4] Evans P E and Smith G C 1959 The compaction of metal powders by rolling. II-An examination of the compaction process *Powder Metallurgy* **2** 26-44

[5] Tundermann J H and Singer A R E 1968 The flow of iron powder during roll compaction *Powder Metallurgy* **11** 261–294
[6] Dube R K 1990 Metal strip via roll compaction and related powder metallurgy routes *International Materials Reviews* **35** 253-292
[7] Delavari M, Salarvand A, Rahi A and Shahri F 2011 The effect of powder metallurgy process parameters on mechanical properties of micro and nano-iron powder *International Journal of Engineering, Science and Technology* **3** 86-94
[8] Worn D K and Perks R P 1959 Production of pure nickel strip by the direct powder rolling process *Powder Metallurgy* **2** 45-71
[9] Bindhumadhavan G, Seville J P K, Adams M J, Greenwood R W and Fitzpatrick S 2005 Roll compaction of a pharmaceutical excipient: Experimental validation of rolling theory for granular solids *Chemical Engineering Science* **60** 3891-3897
[10] Gürbüz M, Şenel M C and Koç E 2018 The effect of sintering time, temperature, and graphene addition on the hardness and microstructure of aluminum composites *Journal of Composite Materials* **52** 553-563
[11] Sturgeon G M, Jackson G, Barker V and Sykes G M H 1968 The production of stainless-steel strip from powder *Powder Metallurgy* **11** 314-329