Determination of Roll Compaction Parameters Required for High Green Density, Defect free Ti-6Al-4V strips

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Abstract: Direct powder rolling (DPR) is an unconventional powder metallurgy technique for fabricating flat mill products. It offers significant cost reduction by using low processing temperatures and reduced fabrication steps compared to ingot metallurgy. Roll compaction is the first stage in the DPR process, hence proper selection of roll compaction parameters is vital for achieving high density defect free (no visible cracks, alligatoring or centre split) green compacts. Unsuitable roll compaction parameters can result in fragile low green density strips which could be difficult to handle during subsequent processing or high green density strips with defects. The focus of this study was therefore, to determine the range of roll compaction parameters required in producing high density, defect free strips. The Ti-6Al-4V strips studied were produced by mixing 150 μm titanium hydride de-hydride powder with a 45 μm 60Al40V master alloy. The powder mix was roll compacted at varying roll gaps and rolling speeds. The compacted strips were evaluated by measuring the green strip density and thickness. It was concluded that for the Ti-6Al-4V powder studied in this work, the preferred roll compaction parameters for achieving high density, defect-free green strips are 0.3 mm to 0.5 mm roll gap with roll speeds from 3 rpm to 10 rpm.

1. INTRODUCTION

Direct powder rolling (DPR) of metal powders is a process that has been around for many years [1] but is however still not yet well understood [2] as there are multiple factors (machine design parameters, process parameters as well as properties of the feed material) affecting the roll compaction stage of the process [3,4]. The DPR process typically consists of roll compaction, sintering and/or hot rolling, mechanical working and/or heat treatment [5–7]. Roll compaction is a critical stage of the DPR process in achieving dimensional stability and full density of the compact after subsequent processing [8]. The DPR process entails using powder (pure powders, blended elemental powders or pre-alloyed powders) as feed material for a roll compacting mill. The roll compacting mill consolidates the feed into a green compact which is then sintered to produce the final product [9–11].

The key process variables in roll compaction include powder properties [9,10], compaction pressure, roll gap, rolling speed [10,12–14], roll surface finish [12,13,15] and roll diameter [16]. Roll gap is the most critical parameter of compaction as it has a significant effect on the resulting green density [12]. Decreasing the roll gap has been found to yield an increase in green density of roll compacted powder materials [10,17]. Roll speed determines the dwell time for particle compaction which in turn also affects density. Rolling at high speeds can result in inadequate compaction of material passing through the rolls [12] which can result in formation of a non-continuous strip [16] or no compaction taking place at all; contrary, rolling at low speeds could result in over-compaction [18].

Roll compaction parameter selection plays an important role in the consolidation process as it has a marked effect on the compaction behaviour of powders. Unsuitable roll compaction parameters will result in a green strip with side cracking, alligator cracking, centre splitting, non-uniform thickness distribution etc. [19,20]. Defects obtained in the roll compaction stage may be difficult to rectify or eliminate with subsequent processing. This in turn could result in the green strip being discarded thereby leading to loss in material. It is, consequently, of utmost importance to define roll compaction processing parameters for a given powder particle size and morphology [9]. In the attempts to evaluate the potential application of DPR on commercially pure titanium powder, Park *et al.* [9] were able to show side cracking and the alligatoring phenomenon by rolling -100 mesh Ti powder. Joo *et al.* [21] used direct reduced iron (DRI) powder to study the maximum allowable strip thickness before alligatoring can occur. Park *et al.* [9] and Joo *et al.* [21] successfully demonstrated roll compaction defects associated with unsuitable roll compaction process parameters.

In their work, Hong *et al.* [22] used -200 mesh and -400 mesh Ti powder to study the effect of particle size and distribution, roll gap and roll speed on densification. Their results concluded that density increases with a decrease in roll gap and that roll speed has no significant effect on density and thickness. The results obtained from the work of Freeman *et al.* [17] showed a general trend that increasing the roll gap decreases density. Chikosha *et al.* [10] studied the effect of particle size and morphology on roll compaction of Ti based powders. In their work, they concluded that density and strip thickness are dependent on the roll gap. Their results showed a general trend where decreasing the roll gap resulted in an increase in density and a decrease in strip thickness.

In this paper, we study the effects of roll gap and roll speed to determine the range at which high density; defect-free green strips are produced. During the roll compaction of 150 µm Ti-6Al-4V powder mix, all other parameters such as powder properties, compaction pressure, diameter roll compaction mill were kept constant.

2. Experimental methods

2.1. Materials

The starting powders used in this study to produce a Ti-6Al-4V powder mix were titanium hydride-dehydride TiHDH supplied by Baoji Lihua Non-Ferrous Metals Co., Ltd and 60Al40V (60 wt.%Al and 40 wt.%V) master alloy supplied by Reading Alloys. The TiHDH powder was reported by the supplier to have a particle size distribution of 150 µm whereas the 60Al40V master alloy powder was reported by the supplier to have a particle size distribution of 45 µm. The chemical compositions of the powders according to the suppliers are listed on Table 1.

Table 1: Chemical composition (wt.%) of starting powders according to supplier.

Material ID	Fe	C	N	H	O	Cl	Al	V	Ti	Other
150 µm TiHDH	0.018	0.002	0.017	0.002	0.10	0.020			Bal.	0.40
45µm Master alloy	0.500	0.100	0.050	0.015	0.10		54-60	40-45		1.4

2.2. Powder characterisation and mixing

The powders were characterised for morphology, particle size distribution and oxygen content. Powder morphology analysis was done using the Jeol JSM-6510 Scanning Electron Microscope (SEM). Particle size distribution was analysed using the Microtrac Bluewave X3500 particle size analyser and the oxygen analysis was achieved using ELTRA ONH 2000 analyser. Powder feed preparation was achieved by mixing the Ti and master alloy powders for 30 minutes on a Diex Tubular mixer operating at a speed of 96 rpm. The powder feed formulation used in the study consisted of 90 wt. % titanium and 10 wt. % master alloy mix.

2.3. Roll compaction

Roll compaction of the powder was carried out on a reversible two roll mill supplied by Chico precision machine Co (China). The diameter of the rolls was 400 mm with the roll face width being 30 mm. The process parameters used in the study were roll speeds of 3, 5, 7, and 10 rpm; roll gaps of 0.2, 0.3 and 0.5 mm

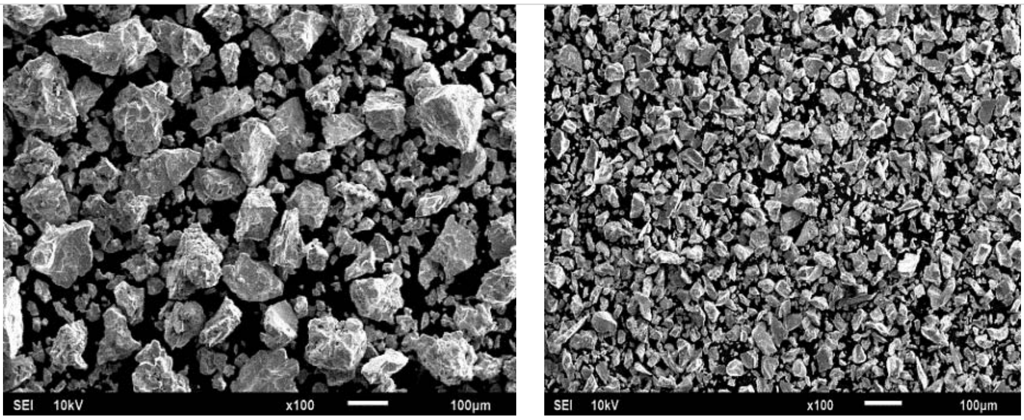
2.4. Density and thickness analysis

Bulk densities of the roll compacted strips were measured using volumetric density which uses the mass, length, width and thickness of the strips to calculate the density. The relative densities of the strips were then calculated as the ratio of the bulk density to the theoretical density (4.42 g/cm^3). The mass of the strips were measured using the OHAUS Voyager Pro balance and the length and width were measured using a digital calliper and the thickness using a TA micrometer screw gauge.

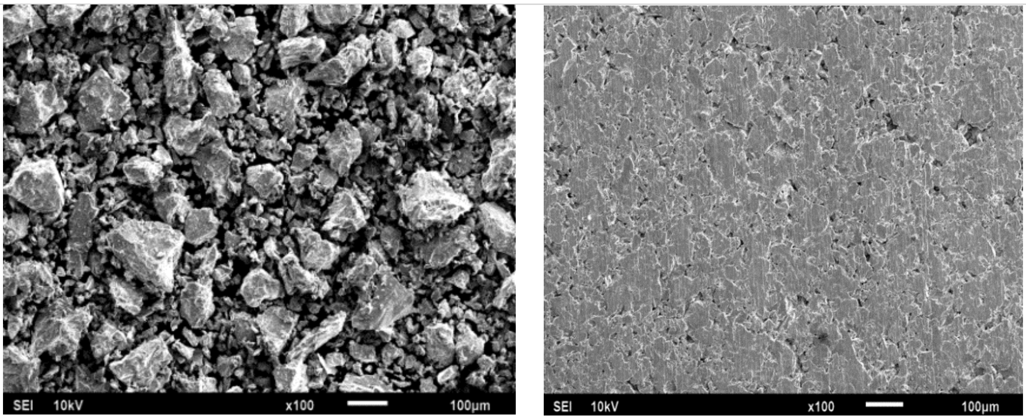
3. Results and Discussions

3.1. Powder characterisation

The 150 µm TiHDH, 45 µm 60Al40V (60 wt.%Al and 40 wt.%V) master alloy powder and the mixed Ti-60Al-40V alloy powder was analysed for powder morphology, particle size distribution and oxygen content. Figure 1(a,b,c) shows particle size distributions and morphologies of the powders whereas Figure 1(d) shows how the powder deforms and interlocks to form a strip after roll compaction. All powders possessed an angular morphology. The particle size distribution of the powders showed a narrow distribution; this is also confirmed by the D_{10}, D_{50} and D_{90} values on Table 2. The 150 µm TiHDH and the 150 µm Ti-60Al-40V both show a somewhat bimodal distribution with peaks at around 45 µm and 150 µm. The measured oxygen content of the powders were higher than those indicated by the supplier; consequently, the resulting 150 µm Ti-60Al-40V mixture has higher oxygen content as compared to the ASTM B265-15 [23] standard for Ti-6Al-4V alloy.

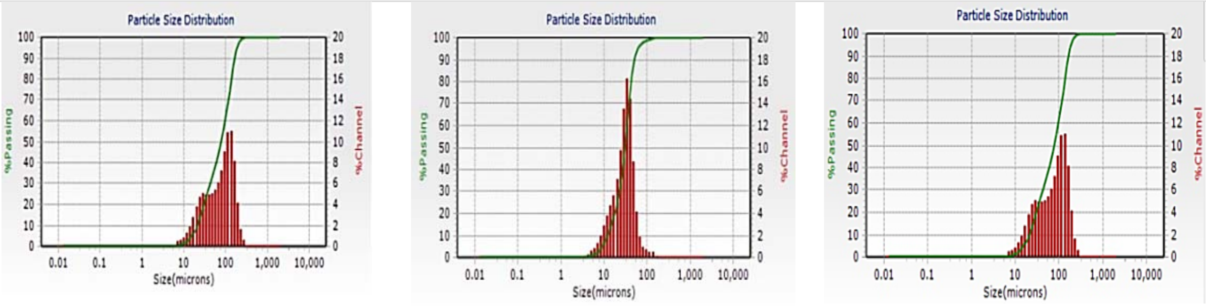


(a) **(b)**



(c) (d)

Figure 1. Particle morphology of the powders: (a) 150 µm TiHDH, (b) 45 µm master alloy, (c) 150 µm Ti-60Al-40V, (d) roll compacted powder forming a strip



(a) (b) (c)

Figure 2. Particle size distribution of the powders: (a) 150 µm TiHDH, (b) 45 µm master alloy, (c) 150 µm Ti-60Al-40V

Table 2. Particle size distribution and oxygen analysis of the starting powders.

Material ID	Particle size distribution			Oxygen content
	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)	(%)
150 µm TiHDH	35.4	125.1	160.1	0.27
45 µm Master Alloy	22.2	45.7	150.7	0.29
150 µm Ti-6Al-4V	28.1	92.1	142.7	0.27

3.2. Roll compaction

Table 3 shows the rolling parameters tested in this study. During roll compaction at a roll gap of 0.2 mm, the roll compacted strips resulted in side cracking and centre splitting. At 0.3 mm and 0.5 mm roll gap, the powder roll compacted to varying densities with no defects. Figure 3(a) shows a typical defective strip containing side cracks whilst Figure 3(a) shows a typical defect-free strip produced from the roll compaction process.

Table 3. Rolling parameters and resulting green density and thickness of strips used in the study.

Roll gap (mm)	Roll Speed (rpm)	Defects	Density (%)	Thickness (mm)
0.2	3	Yes	As a result of cracking, density and thickness of strips rolled at 0.2 mm roll gap were not measured.	
	5	Yes		
	7	Yes		
	10	Yes		
0.3	3	No	84	0.20
	5	No	87	0.21
	7	No	85	0.21
	10	No	86	0.21
0.5	3	No	83	0.21
	5	No	79	0.22
	7	No	52	0.22
	10	No	80	0.22

Figure 3. Roll compacted strips (a) defective strip showing side cracking and (b) defect free strip

3.3. *Density, and thickness characterisation*

The cracked strips rolled at a roll gap of 0.2 mm were not characterised for density and thickness as the cracking could make the results unreliable. Seeing as to how the strips cracked in different ways, the types and causes of cracking will be studied later to understand the relationship between density, roll gap and types of cracking.

The density results on Figure 4 are represented with the standard error of the mean (SEM). The relative green density results (Figure 4 (a)) show a general trend that a smaller roll gap gives a higher relative green density regardless of the rolling speed employed. This behaviour is expected and has been observed in the work of Freeman *et al.* [17]. However, the strips rolled at a 0.3 mm roll gap and a rolling speed of 3 rpm yielded a significantly low green density compared to those rolled at higher roll speeds at the same roll gap. This behaviour is uncommon and unexpected as literature states that rolling at low speeds implies more dwell time for particle compaction [12] which could even result in over-compaction of the powder [18]. This behaviour could be attributed to particle packing of the powder feed prior to roll compaction whereby the fine powders could have agglomerated resulting in inhomogeneous densification after roll compaction. Apart from this unexpected result, the remaining results indicate that there is no significant change in density with rolling speed. This result is contrary to literature since rolling at high speeds is said to result in inadequate compaction of material passing through the rolls [12] hence low densities in compact rolled at higher speeds. However, Hong *et al.* [22] also found that there is no significant effect on density when rolling at high speeds.

Figure 4 (b) shows the effect of roll gap on strip thickness. The results show that increasing the roll gap results in an increase in the strip thickness. This behaviour is expected as increasing the roll gap means more material is passing through the roll gap during roll compaction and has been documented in the work of Chikosha *et al.* [10] and Hong *et al.* [22]. Varying the roll speed has no distinct effect on the strip thickness. This behaviour is also expected and has been documented in a study conducted by Hong *et al.* [22]. The strip thicknesses for all parameters were found to be thicker than the set roll gap. This behaviour has also been previously reported on by Zhang [16] who attributed to the roll spring back effect during roll compaction that occurs as a result of the elastic strain recovery after roll compaction. Overall, green density and strip thickness are strongly dependant on the roll gap where green density is inversely proportional to the roll gap with thickness being contrary. Both green density and strip thickness were found to be independent of the rolling speed. This behaviour was expected from the strip thickness and not from the density as rolling at high speeds results in inadequate compaction of material passing through the rolls.

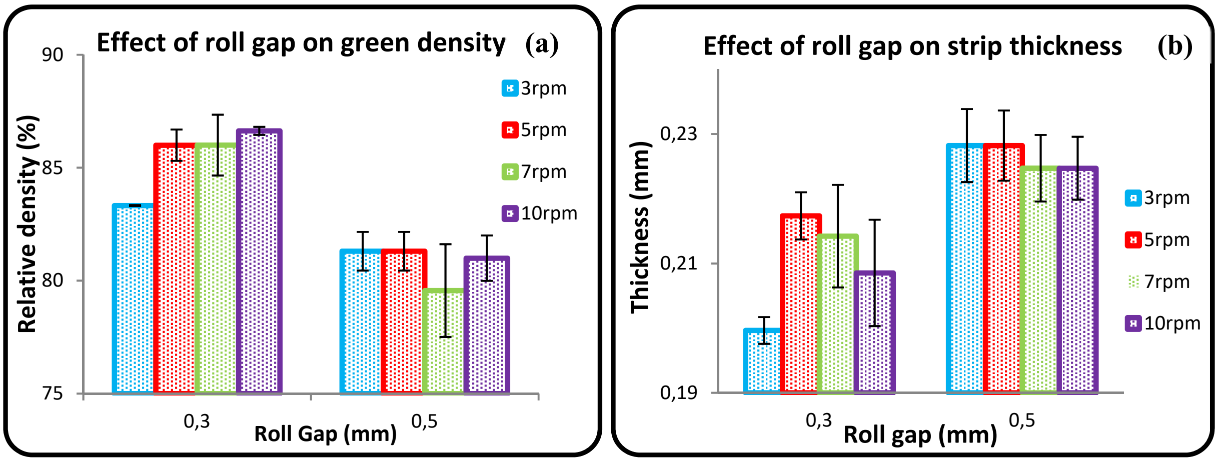


Figure 4. Effect of roll gap on (a) green density and (b) strip thickness.

4. Conclusions

High density and defect-free strips could be achieved at roll gap ranges of 0.3 mm to 0.5 mm and roll speed of 3 rpm to 10 rpm specifically:

- Rolling 150 µm Ti-60Al-40V powder mix is unsuitable at a 0.2 mm roll gap as the powder over compacts resulting in defects.
- Rolling between 0.3 mm and 0.5 mm roll gaps would be suitable for achieving green densities of greater than 80%.
- Increasing the roll gap from 0.3 mm to 0.5 mm resulted in a decrease in the green density.
- Reducing roll gap from 0.5 mm to 0.3 mm increases the green density by 4.9% while reducing the strip thickness by 1.4%.
- Increasing the roll gap from 0.3 mm to 0.5 mm resulted in an increase in strip thickness.
- Green density and thickness are independent of the rolling speed.

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References

[1] Esawi A M K and El Borady M A 2008 Carbon nanotube-reinforced aluminium strips *Compos. Sci. Technol.* **68** 486–92

[2] Cantin, G M D&Gibson M 2015 Titanium sheet fabrication from powder *Titanium Powder Metallurgy: Science, Technology and Applications* ed M Froes, Francis. H. Sam & Qian (Butterworth-Heinemann) pp 383–403

[3] Cunningham J C 2005 *Experimental studies and modeling of the roller compaction of pharmaceutical powders*

[4] Yu S 2012 *ROLL COMPACTION OF PHARMACEUTICAL EXCIPIENTS* (University of Birmingham)

[5] Muchavi N S, Bam L, De Beer F C, Chikosha S and Machaka R 2016 X-ray computed microtomography studies of MIM and DPR parts *J. South. African Inst. Min. Metall.* **116** 973–80

[6] 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 Metall.* **54** 188–92

[7] Ro D H, Toaz M W and Moxson V S 1983 The Direct Powder-Rolling Process for Producing Thin Metal Strip *JOM J. Miner. Met. Mater. Soc.* **35** 34–9

[8] Mothosi K L, Chikosha S, Madyira D M and Chikwanda H K 2017 Determination of Residual Stresses in Roll Compacted Titanium Strips *Procedia Manuf.* **7** 309–15

[9] Park N K, Lee C H, Kim J H and Hong J K 2012 Characteristics of powder-rolled and sintered sheets made from HDH Ti powders *Key Eng. Mater.* **520** 281–8

[10] Chikosha S, Shabalala T C and Chikwanda H K 2014 Effect of particle morphology and size on roll compaction of Ti-based powders *Powder Technol.* **264** 310–9

[11] Cantin D, Kean P, Stone N A, Wilson R, Gibson M A, Ritchie D and Rajakumar R 2010 *Direct Powder Rolling (DPR) of Titanium. Light Metals Flagship*

[12] Srikant P, Akash J, Mahesh D and Astik S 2015 Roller compaction design and critical parameters in drug formulation and development: Review *Int. J. PharmTech Res.* **7** 90–8

[13] Hadzovic E 2008 *Roller Compaction of Theophylline*

[14] Reynolds G, Ingale R, Roberts R, Kothari S and Gururajan B 2010 Practical application of roller compaction process modeling *Comput. Chem. Eng.* **34** 1049–57

[15] Guigon P and Simon O 2003 Roll press design - Influence of force feed systems on compaction *Powder Technol.* **130** 41–8

[16] Zhang Y 2015 *A study of direct powder rolling route for CP-Titanium* (University of Cape Town)

[17] Freeman T, Vom Bey H, Hanish M, Brockbank K and Armstrong B 2016 The influence of roller compaction processing variables on the rheological properties of granules *Asian J. Pharm. Sci.* **11** 516–27

[18] Des E and Carmaux M D A- 2003 *Numerical Methods for Predicting Numerical Methods for Predicting*

[19] Sharma P . 1996 *A TEXTBOOK OF PRODUCTION TECHNOLOGY (Manufacturing Processes)* (New Dehli: S. CHAND & COMPANY PVT. LTD.)

[20] Youssef A H, El-Hofy A H and Ahmed H M 2012 *MANUFACTURING TECHNOLOGY Materials, Processes, and Equipment* (Florida: CRC Press Taylor & Francis Group)

[21] Joo S H, Chang H J, Bang W H, Han H N and Oh K H 2005 Analysis of Alligatoring Behavior during Roll Pressing of DRI Powder with Flat Roller and Indentation-Type Roller *Mater. Sci. Forum* **475–479** 3223–6

[22] HONG J-K, LEE C-H, KIM J-H, YEOM J-T and PARK N-K 2010 Ti STRIP PROPERTIES FABRICATED BY POWDER ROLLING METHOD *Surf. Rev. Lett.* **17** 229–34

[23] ASTM International ASTM B265-15, Standard Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate *ASTM Stand*