

The Effects of Powder Recycling on the Mechanical Properties of Additively Manufactured 17-4 PH Stainless Steel

Pooriya Dastranjy Nezhadfar^{1,2}, Arash Soltani-Tehrani^{1,2}, Amanda Sterling^{1,2}, Nicholas
Tsolas^{1,2}, Nima Shamsaei^{1,2,*}

¹National Center for Additive Manufacturing Excellence (NCAME), Auburn University,
Auburn, AL 36849, USA

²Department of Mechanical Engineering, Auburn University, Auburn, AL 36849, USA

*Corresponding Author: shamsaei@auburn.edu

Abstract

The booming interest in Additive Manufacturing (AM), is seeing a rising number of industries and research entities adopting this technology into their manufacturing practices. Of particular interest is Laser Powder Bed Fusion (L-PBF) process, a common AM method for fabricating metallic components. However, one obstacle is the high cost of powder feedstock. A popular tactic to offset this cost is to reuse the powder between prints, but there is no in-depth understanding of how the powder feedstock may change or affect the mechanical properties of the produced parts. By incorporating unique powder/part characterization methods, this study quantifies the rheological properties of continually recycled 17-4 precipitation hardening (PH) stainless steel (SS) powder through successive printing of mechanical test specimens. The AM specimens are subjected to tensile tests, to correlate mechanical behavior to changing powder quality, including particle size/shape distribution, flowability, and density.

Keywords: 17-4 PH stainless steel; Laser powder bed fusion; Powder characterization; Powder recycling; Mechanical properties; Tensile behavior

Introduction

Additive manufacturing (AM) has become a well-known layer-based manufacturing technique which helped in overcoming several limitations inherent to subtractive manufacturing methods – allowing end users the ability to fabricate near-net shaped parts directly from computer aided design (CAD) models [1, 2]. AM facilitate the production of parts with complex geometries which are unobtainable through the traditional manufacturing methods. Laser powder bed fusion (L-PBF) is one of the most common AM processes for fabrication of metallic components [3]. In this process, parts are fabricated by repeatedly melting and fusing a thin layer of powder by a high-powered laser source to successively build components layer-by-layer. Titanium and aluminum alloys, various grades of stainless steels and Nickel-based super alloys are the most implemented materials by this method [4]. L-PBF systems are preferred due to their ability to produce finer resolution and higher quality parts compared to other AM methods [5].

Although AM has its advantages, there are some issues, which must be thoroughly investigated before these parts are being used as critical load bearing parts. Various defects

inherited from the process such as porosity, lack of fusion voids and surface roughness are the main drawbacks, which may affect the mechanical performance of the parts, especially under cyclic loading. Utilizing appropriate process parameters, build orientation and powder conditions have been reported as important parameters to control or reduce the aforementioned issues [6]. Considering the powder is one of the most crucial elements in the L-PBF method, many studies in the past have examined how powder characteristics effect the mechanical behavior of fabricated parts. Recently, morphological characteristics comprising size, shape and surface roughness have been studied. Irrinki et al. [7] studied gas- and water-atomized powders with different mean particle sizes, and observed lower porosity and higher part densities when fabricated with finer particle sizes. Parteli et al. [8] developed a particle-based numerical simulation and reported larger surface roughness values when parts are fabricated with powders containing a larger size distribution of particles. This was attributed to the finer particles tendency to form larger agglomerates, which increases porosity as a result of larger particles inability to fill voids during packing. Sutton et al. [9] has mentioned that the flowability and layer homogeneity are highly influenced by particle size and size distribution. Moreover, the large particles and agglomerates may produce voids because of the reduction in packing density.

Other studies have also investigated how recycling and reusing can change powder characteristics and its relation to the mechanical properties of fabricated parts. Powder reusing is of high importance to industries because of its economic and environmental perspective. Currently, using either the un-melted metal powder from the previous builds or mixing with virgin powder are potential strategies. However, it has been indicated that recycling powders comprised of powder particles that have been exposed to the building chamber conditions, with condensate powder particles splattered from the melt pool can have an effect on the fabrication fusion process, which can lead to variation in the overall material properties of the part [10].

Debate remains on the methodology and effects of powder recycling on AM material properties. Some studies have reported no variation in mechanical properties fabricated via recycled powders [11-13]. Jelis et al. [11] studied the effects of powder recycling on the mechanical properties of AM 4340 steel fabricated through direct metal laser sintering (DMLS). Virgin and once-recycled powder were used and passed through an 80 μm sieving screen in order to eliminate the overexposed and splattered condensate particles. Insignificant changes in chemical composition of powders, and tensile properties were reported. Similarly, Jacob et al. [12] reported insignificant changes in shape, particle size and particle size distribution, but an increase in the apparent density and powder bed density were observed after recycling 17-4 PH stainless steel powders up to 11 times. Although, some changes in powder rheological properties were reported, there were no significant changes in the tensile behavior of tested specimens. In addition, it has been reported that IN718 powder properties and tensile deformation behavior had no change after 14 times powder recycling [13]. On the other hand, Tang et al. [14] reported an increase in the yield and tensile strength of Ti-6Al-4V parts after 21 times powder recycling. This was attributed to the increase in oxygen content absorbed during the fabrication process.

Although many studies have investigated the powder recycling effects on the tensile behavior of fabricated parts, the fatigue performance of the parts have not been studied. As mentioned earlier, changes in powder rheological properties induce porosity and surface roughness. Many studies have reported the detrimental effects of defects such as pores, lack of

fusion and surface roughness on the fatigue performance of AM parts [2, 3, 15, 16]. Therefore, understanding the powder recycling effects on the cyclic deformation behavior of AM parts is essential.

17-4 precipitation hardening (PH) stainless steel (SS) has drawn significant attention recently due to its outstanding characteristics, such as high tensile/impact strength, fracture toughness and corrosion resistance at service temperatures below 300 °C. This material is widely used for structural components in aerospace, petrochemical, power plants and marine environments [17, 18]. The goal of this study is to investigate the effects of powder degradation on the microstructural evolution, monotonic and cyclic deformation behavior of 17-4 PH SS after powder recycling. Moreover, identifying a measurable variable of powder properties indicative of the part quality is another goal of this ongoing study. The preliminary results of this broad powder study will be presented in the present paper. Following the introduction, the material and experimental procedures are presented. The powder characterization results and tensile deformation behavior are then discussed. Finally, important conclusions are drawn.

Experimental procedures

Argon-atomized 17-4 PH SS powder with the particle size distribution (PSD) of 15-45 μm provided by LPW Technology Inc. was used for this study. The chemical composition of the virgin powder is presented in [Table 1](#). 15 sets of prints were carried out using 80 kg of powder, with the aim of recycling the powder after each print. [Figure 1](#) shows the full print and half print layouts. The full print layout consists of tensile, fatigue and impact test specimens, while the half print layout only includes the tensile and impact test specimens. This strategy not only minimized fabrication time, but also afforded a greater number prints (number of powder recycling times). Batch numbers 1, 5, 10 and 15 followed the full print layout, while batches in between followed the half print layout. Specimens were fabricated vertically in a nitrogen atmosphere using an EOS M290 L-PBF system. Default build parameters were used, as well as the fabrication started with unused, virgin powder (batch 1). The un-melted powder after each fabrication was being collected and sieved through an 80 μm screen and mixed with the original powder in the feed bin to generate the feedstock for the next batch. After mixing the powder, a sample representative of all the powder in the feed bin was obtained according to ASTM B215-15 [19] (equal increments from different depths of the powder). This process was continued after every printed batch until the 15th print. It is worth mentioning that no virgin powder was added in between the fabrication of batches, in order to isolate the effects of powder recycling on the rheological properties and final mechanical response of the parts.

To characterize the rheological properties of the powder used in fabricating batches 1, 5, 10 and 15, a Freeman Technology rheometer (FT4) machine was employed. This instrument is capable of measuring various rheological powder properties including bulk/apparent and tapped (maximum) density, compressibility, aeration ratio, cohesion, permeability, and shear stress. This instrument quantifies the powder's rheological properties measuring the amount of work done in moving the blade through the powder from the top to the bottom of the vessel. In the current stage of this study, only powder bulk density, aeration ratio, and cohesion were measured.

Table 1. Chemical composition of 17-4 PH stainless steel powder

	C	Cr	Ni	Cu	Mn	Si	Nb	Mo	N	O	Fe
(Wt. %)	0.01	15.6	4.03	3.89	0.24	0.29	0.33	<0.01	0.01	0.05	Bal.

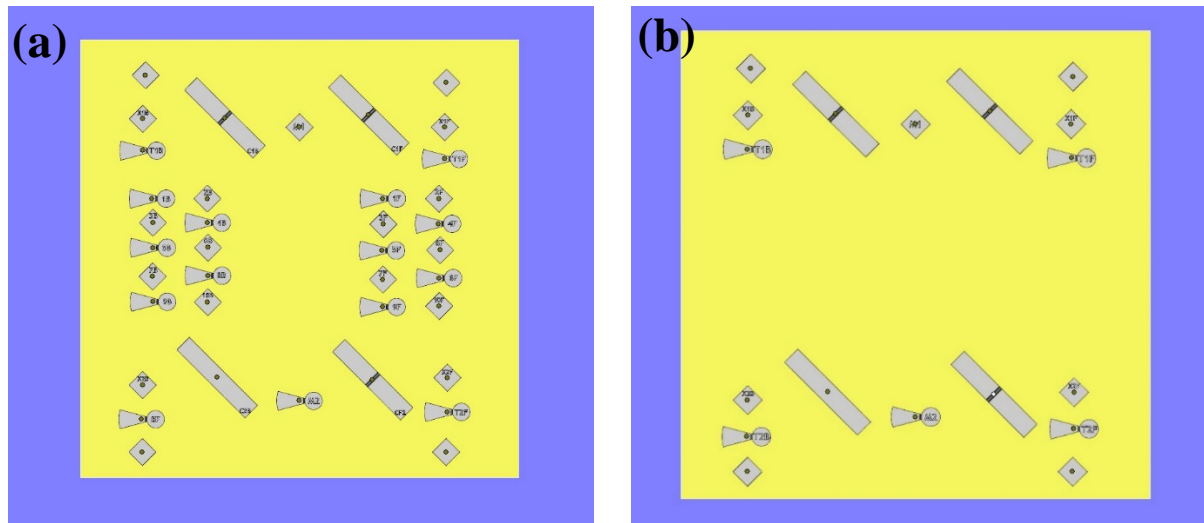


Figure 1. The layout of prints: (a) full print, (b) half print

Specimens were heat treated, which included a 30-min solution treatment at 1050°C, followed by air cooling to the room temperature, and subsequent annealing for 4-hrs at 552°C, followed again by air cooling to room temperature.

To evaluate the mechanical properties, specimens were subjected to uniaxial tensile tests at room temperature at 0.001 s⁻¹ strain rate using MTS Landmark servo hydraulic test system with 100 kN load cells. Tests were carried out in two steps, strain-controlled and displacement controlled. Since the elongation to failure of the material is higher than the resolution of the extensometer, the strain-controlled step was carried up to 0.045 strain and continued in displacement control to the final fracture by removing the extensometer. For certainty, three tensile tests were conducted for each batch to see the consistency of the results and eventually, the average is presented.

Results and discussion

Powder Quality

Figure 2 represents the variation in density of powder samples obtained from batches 1, 5, 10 and 15. As observed, the powder density increased with the number of recycling times. This is attributed to the change in the powder particles size distribution. Finer particles melted and solidified, while particles greater than 50 μm were pushed away from the build plate via the recoater arm since the pre-adjusted gap between the substrate and the recoater arm was smaller

than 50 μm . It has been previously reported that due to the shape, frictional forces, particle interlocking and changes in the moisture level of the powder, particles are expected to agglomerate [14]. As such, any agglomerated particles passed through the sieve screen were broken down into finer particles. This effect will narrow down the PSD with each recycling time. Any changes in PSD will affect the porosity distribution in the powder bed. In fact, the recycled powder with the narrower PSD has a lower porosity in the powder bed leading to a higher density [9].

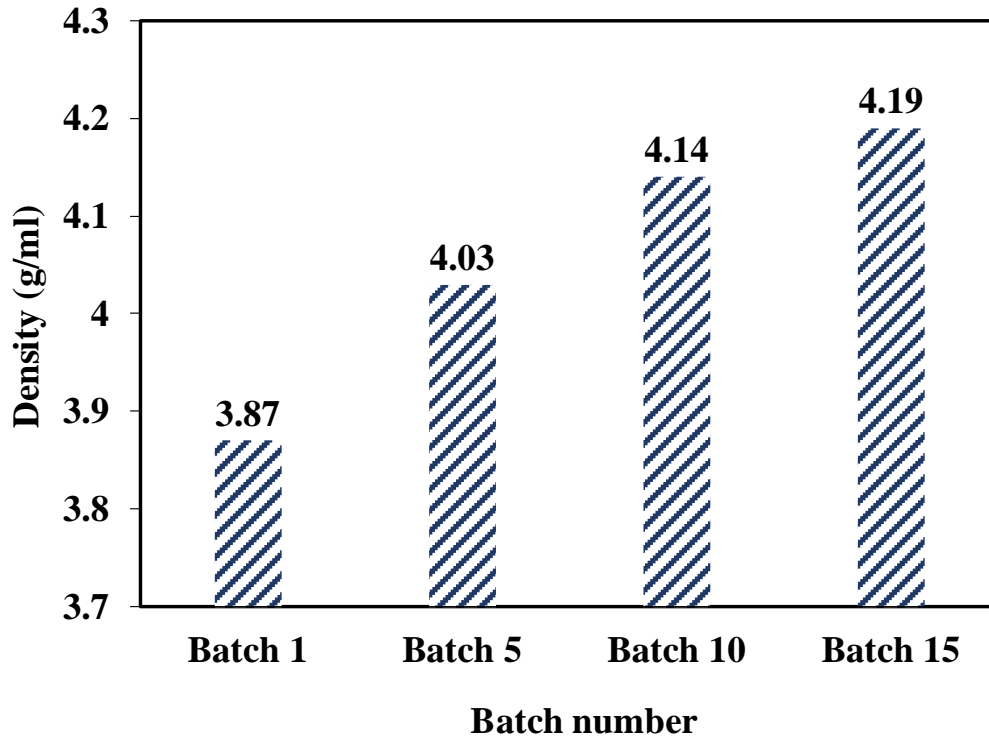


Figure 2. Powder density as a function of recycling

[Figure 3](#) compares the aeration ratio of the reused powder for batches 1, 5, 10 and 15. Aeration ratio is an indication of powder sensitivity to air pockets. The aeration ratio represents how easily the gas can flow within the powders and transfer them to the building platform. As shown, the aeration ratio decreased from batch 1 to 5, and gradually increased up to batch 15. Accordingly, batch 5 possesses a higher sensitivity to air pockets, which represents less resistance to inter-particle forces, consequently increasing the flowability of the powder. The aeration ratio has a direct relation with cohesion, which means that powders with higher aeration ratios have higher cohesion due to frictional forces and particle interlocking. Accordingly, cohesivity of batch 5 is lower than batches 1, 10, and 15, which makes the powder fluidized and thus improves the flowability of powder over the build plate.

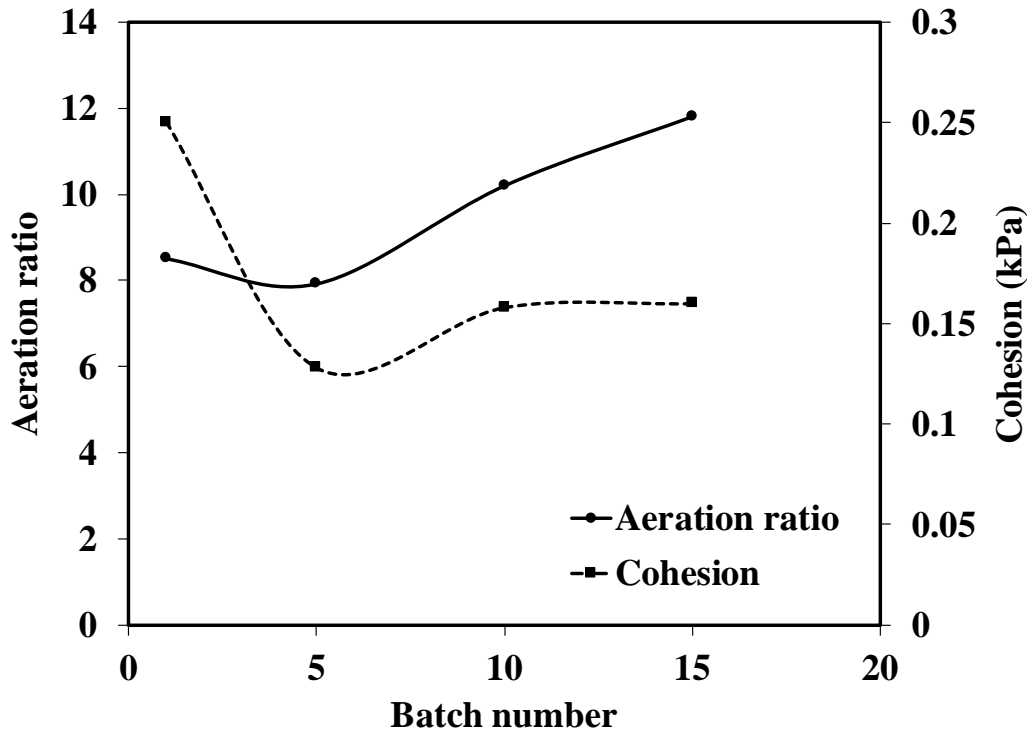


Figure 3. Aeration ratio and cohesion of the powder over recycling times

Tensile behavior

Figure 4 compares the tensile deformation behavior of specimens from batches 1, 5, 10 and 15. Figure 4a represents the strain-controlled step up to 0.045 strain before the extensometer was removed. Figure 4b shows the stress-displacement curve up to the failure. In addition, the tensile behavior of the different batches is compared in detail in Table 2. At first glance, it is observed that the tensile deformation behavior has not been affected significantly by incremental change in powder recycling times. However, details in Table 2 reveal a 20% increase in reduction of area (RA) and approximately 23% increase in true strain at fracture (ϵ_f) for 5th batch compared to 1st batch. On the other hand, it seems that incremental changes in powder recycling after the 5th batch has led to a slight decrease (~ 6-8%) in RA and ϵ_f . In other words, the ductility has increased after 4 iterations in powder recycling (5th batch) and decreases after a higher numbers of iteration times (10th and 15th batch). The increase in ductility of 5th batch is attributed to its lower cohesion compare to other batches (Figure 3). As mentioned earlier, lower cohesion means the powder possess higher flowability, which would affect the contiguity of the part. Accordingly, increase in the cohesion of powder would decrease the ductility of the part. Moreover, a slight decrease in strength is observed for the 15th batch.

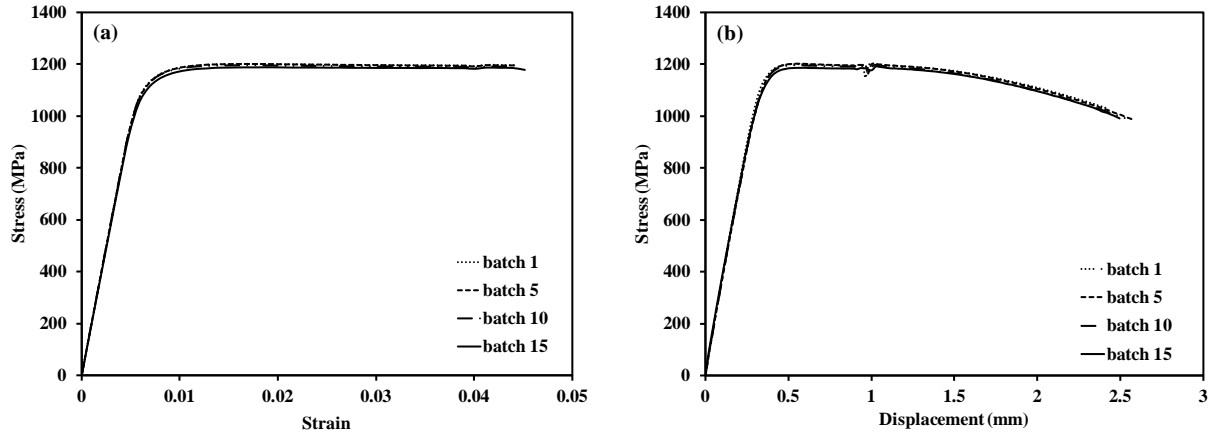


Figure 4. Tensile behavior of batches 1, 5, 10 and 15: (a) stress-strain curve up to the 0.045 strain, (b) stress-displacement curve up to the failure

Table 2. The details of tensile test results of batches 1, 5, 10 and 15

Print number	1 st	5 th	10 th	15 th
<i>RA</i> %	29.50	35.40	34.70	33.00
ϵ_f	0.35	0.43	0.42	0.40

Conclusions

This study was carried out to investigate the changes in rheological properties of recycled powder after 15 sets of prints and the subsequent effects on the mechanical properties of the parts. The goal of this study was to propose a specific change in powder rheological properties which causes variation in mechanical properties of the parts. To this end, 15 batches of specimens were fabricated continuously with a certain amount of powder without adding unused virgin powder. The remaining powder after fabrication of each batch was sieved and used for the next subsequent batches. Powder rheological properties were analyzed for print numbers 1, 5, 10, and 15, and compared to the unused powder. Moreover, the mechanical properties of the specimens belonging to the above mentioned set of prints were studied based on tensile mode of deformation. The following conclusions can be made based upon the results:

1. The apparent/bulk density of the powder increased with recycling times. This is attributed to the change in the PSD after recycling-fabrication-recycling loop. PSD becomes narrower after each recycling time.
2. Powder cohesion decreased from batch 1 to 5 and then increased from batch 5 to 10 and plateaued at 15.
3. Powder flowability of batch 5 was the highest amongst all batches examined.
4. Powder recycling up to 5th batch resulted in a 20% increase in *RA* and 23% increase in ϵ_f . This is attributed to the decrease in cohesion and an increase in the flowability of powder up to this level of recycling, which also affects the structural integrity of the part.

Acknowledgements

This work is sponsored by the Naval Air Systems Command (NAVAIR)

References

- [1] T.D. Ngo, A. Kashani, G. Imbalzano, K.T.Q. Nguyen, D. Hui, Additive manufacturing (3D printing): A review of materials, methods, applications and challenges, *Composites Part B: Engineering* 143 (2018) 172-196.
- [2] A. Yadollahi, N. Shamsaei, Additive manufacturing of fatigue resistant materials: Challenges and opportunities, *International Journal of Fatigue* 98 (2017) 14-31.
- [3] N. Shamsaei, A. Yadollahi, L. Bian, S.M. Thompson, An overview of Direct Laser Deposition for additive manufacturing; Part II: Mechanical behavior, process parameter optimization and control, *Additive Manufacturing* 8 (2015) 12-35.
- [4] D. Herzog, V. Seyda, E. Wycisk, C. Emmelmann, Additive manufacturing of metals, *Acta Materialia* 117 (2016) 371-392.
- [5] W.J. Sames, F.A. List, S. Pannala, R.R. Dehoff, S.S. Babu, The metallurgy and processing science of metal additive manufacturing, *International Materials Reviews* 61(5) (2016) 315-360.
- [6] S.R. Daniewicz, N. Shamsaei, An introduction to the fatigue and fracture behavior of additive manufactured parts, *International Journal of Fatigue* 94 (2017) 167.
- [7] H. Irrinki, J.S.D. Jangam, S. Pasebani, S. Badwe, J. Stitzel, K. Kate, O. Gulsoy, S.V. Atre, Effects of particle characteristics on the microstructure and mechanical properties of 17-4 PH stainless steel fabricated by laser-powder bed fusion, *Powder Technology* 331 (2018) 192-203.
- [8] E.J.R. Parteli, T. Pöschel, Particle-based simulation of powder application in additive manufacturing, *Powder Technology* 288 (2016) 96-102.
- [9] A.T. Sutton, C.S. Kriewall, M.C. Leu, J.W. Newkirk, Powder characterisation techniques and effects of powder characteristics on part properties in powder-bed fusion processes, *Virtual and Physical Prototyping* 12(1) (2016) 3-29.
- [10] S. Dingal, T.R. Pradhan, J.K.S. Sundar, A.R. Choudhury, S.K. Roy, The application of Taguchi's method in the experimental investigation of the laser sintering process, *The International Journal of Advanced Manufacturing Technology* 38(9-10) (2007) 904-914.
- [11] E. Jelis, M. Clemente, S. Kerwien, N.M. Ravindra, M.R. Hespos, Metallurgical and mechanical evaluation of 4340 steel produced by direct metal laser sintering, *Jom* 67(3) (2015) 582-589.
- [12] G. Jacob, C. Brown, A. Donmez, S. Watson, J. Slotwinski, Effects of powder recycling on stainless steel powder and built material properties in metal powder bed fusion processes, *Advanced Manufacturing Series (NIST AMS)-100-6* (2017).
- [13] L.C. Ardila, F. Garcíandia, J.B. González-Díaz, P. Álvarez, A. Echeverria, M.M. Petite, R. Deffley, J. Ochoa, Effect of IN718 recycled powder reuse on properties of parts manufactured by means of selective laser melting, *Physics Procedia* 56 (2014) 99-107.
- [14] H.P. Tang, M. Qian, N. Liu, X.Z. Zhang, G.Y. Yang, J. Wang, Effect of powder reuse times on additive manufacturing of Ti-6Al-4V by selective electron beam melting, *Jom* 67(3) (2015) 555-563.
- [15] D. Greitemeier, C. Dalle Donne, F. Syassen, J. Eufinger, T. Melz, Effect of surface roughness on fatigue performance of additive manufactured Ti-6Al-4V, *Materials Science and Technology* 32(7) (2016) 629-634.

- [16] K. Essa, P. Jamshidi, J. Zou, M.M. Attallah, H. Hassanin, Porosity control in 316L stainless steel using cold and hot isostatic pressing, *Materials & Design* 138 (2018) 21-29.
- [17] S. Pasebani, M. Ghayoor, S. Badwe, H. Irrinki, S.V. Atre, Effects of atomizing media and post processing on mechanical properties of 17-4 PH stainless steel manufactured via selective laser melting, *Additive Manufacturing* 22 (2018) 127-137.
- [18] M. Mahmoudi, A. Elwany, A. Yadollahi, S.M. Thompson, L. Bian, N. Shamsaei, Mechanical properties and microstructural characterization of selective laser melted 17-4 PH stainless steel, *Rapid Prototyping Journal* 23(2) (2017) 280-294.
- [19] Standard Practices for Sampling Metal Powders, ASTM International (2015).