THE EFFECT OF RECYCLING POWDER STEEL ON POROSITY AND SURFACE ROUGHNESS OF SLM PARTS

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The paper deals with the influence of the recycling of 1.2709 powder steel on porosity of the components produced by selective laser melting technology (SLM). One of the main requirements for the parts produced by SLM is to achieve good mechanical properties. The presence of pores, typical defects in the SLM produced material, is one of the causes of the mechanical properties reduction. In particular, to extend the high cycle fatigue life, it is necessary to achieve relative densities close to the full material and to minimize the pore size near the surface of the parts. One of the possible cause of pore formation and the degradation of the properties of the powder material is the standard recycling by sieving to remove the contaminants produced during the build job. Using a series of recycling tests, possible qualitative and quantitative changes of powder material and their consequences on the porosity of the manufactured parts are monitored. The paper proposes recommendations for the recycling process setup, compares the influence of the input parameters on the porosity.

KEYWORDS

SLM, selective laser melting, powder recycling, porosity, 1.2709, 18Ni 300, martensitic steel

1 INTRODUCTION

Selective laser melting (SLM) is one of the additive technologies (AM) in which the powder material is joined by the laser beam layer-by-layer. A high-power laser melts the metallic powder only at the places given by actual cross section of the product geometry, when scanning each layer. Once solidified, a new layer is applied, and the process is repeated until a complete component is created. The development of SLM in recent years shows the high potential of this technology in the production of geometrically complex parts, especially in small-scale production. High strength tool steel 1.2709 (maraging steel) is suitable, for the production of molding tools and molds for injection molding. Fabrication using SLM enables the production of molds with conformal cooling. Maraging steel contains substitution elements in the low carbon, iron-nickel martensitic matrix, which allows hardening by precipitation aging.

Several papers [Casalino 2015], [Badrossamay 2009], [Kempen 2011] dealt with optimization of the processing parameters of maraging steel 1.2709. Authors examined the impact of critical parameters such as laser power, scanning speed, layer thickness and scanning strategy on relative density, porosity, surface quality, and mechanical properties. The relative density values above 99.5% were reached and mechanical properties similar or better compared to material prepared by conventional technologies indicate that 1.2709 is well-processable by SLM technology.

It has been shown that elimination of porosity is an essential prerequisite for achieving good mechanical properties and low surface roughness. The character and degree of porosity are different in static loading than in the fatigue loaging [Santos 2016], [Gu 2014], [Benedetti 2016]. Although, for many materials, by increasing relative density was achieved comparable Rm and Rp0.2 values as those of conventional materials [Hermann 2016], [Spierings 2013], [Riemer 2014], high cycling fatigue is more susceptible to the presence of pores, especially those of subsurface, which are the most common crack initiator [Brandl 2012], [Vandersse 2011]. It was shown that to increase the resistance of the material against crack initiation it is important to reduce the pore diameter below 50 µm [Hermann 2016], [Mayer 2003], [Kim 2011], [Akita 2016]. It is then necessary to eliminate the pores caused by the imperfectly molten powder, which in its non-spherical shape acts as the most important stress concentrators [Casalino 2015]. To exploit the potential of SLM with the possibility of almost wasteless production and increasing the production economics, it is necessary to investigate the recycling process of metallic powder unused during build job. The studies [Gu 2014, Liu 2011], [Spierings 2009], [Spierings 2011] show that the properties of powder material such as particle size distribution and its shape, together with the layer thickness parameter, have a major influence on achieving high relative density. It has been shown that the presence of small particles can have a positive effect on the density of the deposited layer and its higher thermal conductivity. Large particles, due to the thickness of the layer, then complicates the application of even layer and are harder to melt with the laser. Its high concentration in particle size distribution can increase the porosity of the component and cause the formation of pores of irregular shape, which have more negative effects on the mechanical properties than the spherical pores.

Particle size distribution and particle shape are the most affected by the sieving process. The studies examining other materials [Seyda 2012], [Ardila 2014] suggests that the recycling process can increase the average particle size, extend the particle size range, and reduce the smallest particle size in the used powder. This may have a positive effect on the flowability of the powder and, on the other hand, the negative effect on the increase of the size of the pores. Due to the increased particle size, the surface roughness also increases with repeated recycling. The authors also point to the risk of powder oxidation by increasing the humidity due to contact with the atmosphere during sieving, handling and storage. The conclusion of the articles is that the materials under investigation are recyclable and can reach relative densities above 99.8% even with repeated recycling.

2 MATERIAL AND METHODS

2.1 SLM machine and sieving station

The main objective was to analyze the influence of recycling of the 1.2709 steel powder on the relative density and roughness of the surface and to identify the porosity character during the selective laser melting production.

Tests were carried out on the machine M1 Cusing (Concept Laser GmbH) and the QM Powder Module (Concept Laser GmbH), where the effect of repeated powder recycling on its parameters and the relative density and porosity of the build parts were investigated. The QM Powder Module equipped with a sieve with a mesh size of 63 µm was used to sieve the powder. As the powder material was supplied by the manufacturer of the device, the recommended process parameters listed in Tab. 1 were used.

Concept Laser M1 Cusing	
Laser power - hatch	180 W
Scanning speed - hatch	600 mm/s
Laser power - conture	180 W
Laser speed - conture	600 mm/s
Layer thickness	30 µm
Hatch distance	105 µm
Scanning strategy	Chessboard 5x5 mm
Built plate heating	Without heating
Inert gas	N_2

Table 1. Overview of processing parameters for maraging steel 1.2709 recommended by SLM machine producer

2.2 Powder material

The 20 kg batch of tool steel 1.2709 (EN), X3NiCoMoTi18-9-5 (DIN), 18Ni300 (AISI) also known as Maraging steel, was used in the study. Material in powder form was produced by atomisation in inert gas and supplied by Concept Laser GmbH under the designation CL50WS. The particle size distribution and its characteristic values measured by laser diffraction are shown in Fig. 1 and Table 2.

	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)	D_{95} (µm)
Particle diameter	18.1	25.9	37.2	41.2

Table 2. Characteristic values of particle size distribution of used powder

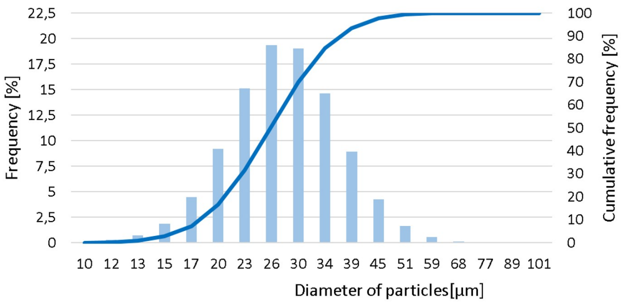

Figure 1. Particle size distribution chart

Table 3 shows nominal chemical composition of powder 1.2709.

Elements Wt (%)		CL50WS
Fe		Bal.
Ni		17-19
Mo		4.5-5.2
Ti		0.8-1.2
Co		8.5-10
Cr	Al	≤ 0.25
Mn		≤ 0.15
C		≤ 0.03
P,S		≤ 0.01

Table 3. Chemical composition according to AISI/SAE

2.3 Powder recycling test

The test was used to determine the extent to which the sieving process affects the powder material and the consequences of porosity and its character. The aim was to monitor its qualitative and quantitative changes through repeated powder recycling and analyze how the relative density of the built component and the morphology of the pores change with the increasing number of recycling cycles.

The recycling test consisted of 11 build jobs (33 pieces) of 10 x 10 x 10 mm cubes on a 250 x 250 mm platform with a reduction of 90 x 90 mm. After each build job the samples were removed and the powder was collected from the machine (Figure 2). The entire powder volume was sieved on the QM Powder Module (Concept Laser) and subsequently used for the next build job. Finally, the 20 kg of the originally new powder underwent 10 recycling operations.

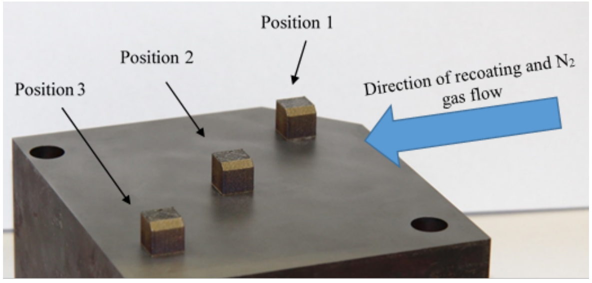

Figure 2. Position of testing cubes

2.4 Particle size distribution analysis

The Horiba LA 950 was used to analyze the particle size distribution of the used metal powder to identify changes caused by recycling. The device works on the principle of laser diffraction. Diffraction data is processed into a distribution and cumulative curves. The accuracy of the Horiba LA 950 is ± 0.6% for particles with sizes from 10 nm to 3 mm.

2.5 Relative porosity analysis

To obtain the relative porosity values of the whole volume of the cube samples, GE Phoenix v|tome|x L240 was used. After sample scanning and reconstruction into 3D data, porosity evaluation was performed in VGStudio Max 3.0. The voxel resolution was 5.5 µm, while a minimum detectable pore size in the analysis was 8 voxels. This corresponds to pores with diameter of about 11 µm. It is worth mentioning the threshold value parameter in prosity/inclusion analysis, which has a significant effect on the gray-shade value at the pore boundary evaluated as a full material. A default value of 0.5 was used, which complied with the visual inspection and showed a low capture of the false pores on the sample surface.

2.6 Surface roughness

To evaluate the surface roughness of the cubes was used BrukerContour GT X8 device operating on the principle of interferometry.

The evaluation was done in Vision64. A lens with 5x magnification and 0.55x lens was used for measurement. The resulting magnification is therefore 2.75x. The “Data restore” function for missing data calculation, a plane fit to remove the effect of geometry and a “Gaussian regression filter” function to remove waves with a wavelength greater than 2.5 mm were used for evaluation. Measurement area was 2.17 x 1.63 mm with a lateral resolution of 3.39 µm. The observed surface roughness parameters were Sa and Sz.

3 RESULTS AND DISCUSSION

3.1 Influence of powder recycling

The first phase of the work was the Concept Laser M1 Cusing recycling test. For the sieving of the powder, a sieving station without a protective atmosphere was used. Using laser diffraction, it was verified that the used CL50WS powder did not contain larger particles than indicated by the manufacturer. Also optical microscopy confirmed the spherical shape of the particles. The recycling test revealed that even after 10 sieving, the powder did not degrade, and the particle size distribution remained preserved (Figure 3). The D_{50} value ranged within 1.5 µm (Table 4).

Sample	Number of sievings	D_{10} [µm]	D_{50} [µm]	D_{90} [µm]	Deviation (µm)
P0	new	18.61	26.72	38.18	7.83
P1	0x	18.09	25.94	37.22	7.62
P2	1x	18.46	27.45	44.65	19.35
P3	2x	19.47	26.68	36.65	6.73
P4	3x	18.92	26.26	36.38	6.83
P5	4x	18.94	26.38	36.62	6.91
P6	5x	18.35	26.92	39.15	8.56
P7	6x	18.39	26.08	36.89	7.26
P8	7x	18.54	26.42	37.51	7.53
P9	8x	18.79	26.30	36.75	7.03
P10	9x	19.09	26.32	36.33	6.74
P11	10x	19.4	27.12	37.93	7.38

Table 4. Overview of the particle size distribution characteristics for rising number of powder recyclation

This undermines the hypothesis that the smallest and lightest particles can be reduced by repetitive recycling due to dusting into the surrounding atmosphere during handling and sieving, due to the fact that there is no closed handling of the powder [Seyda 2012]. The powder proved to be resistant to such changes because it does not contain particles smaller than 10 µm and the particle size distribution D_{10} = 18.5 µm. This corresponds to the results of the article [Ardila 2014], where the powder with similar distribution was used without the presence of particles below 10 µm.

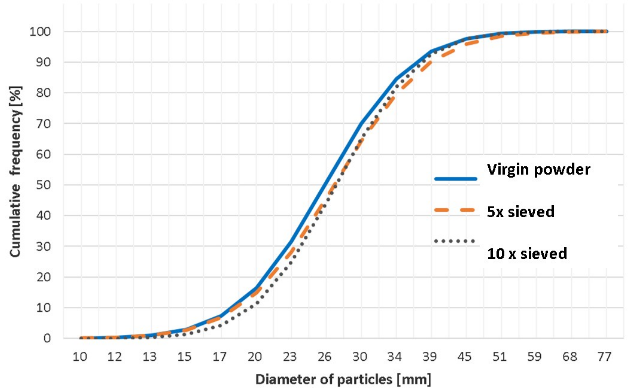

Figure 3. Change in particle size distribution during recycling test at M1 Cusing

The mesh size of 63 µm has proven to be appropriate because it does not remove any fraction of larger particles naturally occurring in a new powder whose size D_{90} = 37.2 µm. The next step could be recycling test with a build job with larger components and smaller amount of powder. The test would be used to verify whether the powder contaminated with more burnt particles can be perfectly cleaned from the excess particulate matter.

3.2 *µCT analysis*

Selected cubes have undergone porosity analysis by computed microtomography. The goal was to determine the porosity, its distribution in the material, the shape of the pores and how these properties change with the increasing number of recycling cycles. Three cube samples from the recycling test (all samples were built in the middle of the build platform) were selected to determine whether there was any recognizable trend of change, the first of the cubes was made of a new powder (V12 cubes), the second of the 5x sieved powder (V62) and the third of the 10x sieved powder (V112). Out of these samples the the 5 x 5 x 5 mm sections were separated by electro-erosive machining and analyzed by µCT (Table 5).

Sample	Number of sievings	Sample volume (mm^3)	Volume of pores (mm^3)	Porosity (%)
V12	0x	148.52	0.05	0.0337
V62	5x	151.4	0.049	0.0327
V112	10x	142.75	0.044	0.0309

Table 5. Change of relative porosity in volume of SLM samples

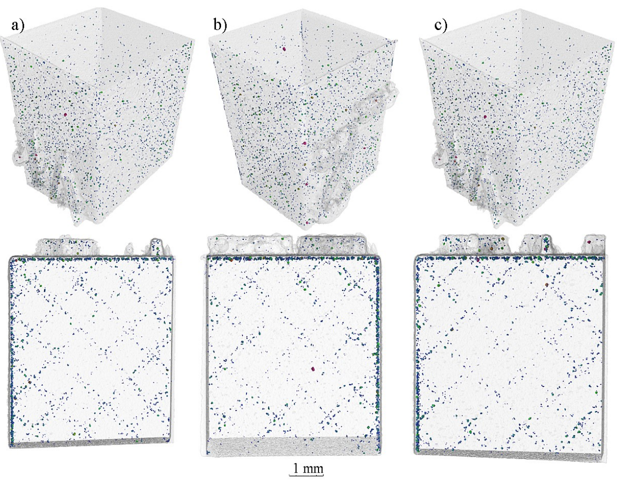

Figure 4. Visualisation of distribution of pores in the SLM samples in ISO view and top view – a) sample V12, b) sample V62, c) sample V112

The figure 4 shows the actual distribution of detected porosity inside the samples. Only the pair of vertical surfaces (from the spatial view in the figure) are the as-built surfaces of the samples, the others were created by electro-erosive machining. From the top view, it is obvious that the pores are dominantly located under the contour of the sample. Regularly spaced porosity in volume shows the formation of pores at the transition between individual squares of the chessboard scanning strategy. This suggests that a set overlap of 22.5 µm of individual squares may not be sufficient.

The depth of increased subsurface porosity is approximately 150 µm for all 3 samples. At a depth of 200 µm, the porosity is comparable to porosity in the hatched volume area. Analysis by computed microtomography showed that the built parts have a very high relative density of about 99.96%, which does not change significantly with the recycling of steel powder. This conclusion corresponds to the results of measurements of the particle size distribution by laser diffraction, which also did not show any significant changes. In terms of relative density, from the powder of almost the same properties are produced parts of homogeneous quality. It can also be argued, that setup of the process parameters for 1.2709 steel is optimal for minimization of volumetric porosity, but not for the transition area between contour and hatch.

From the point of view of porosity localization, it was found that the most significant number of pores is concentrated below the surface of the material at depths of 100 to 150 µm.

This porosity is caused by the inadequate connection of the hatching of the sample volume with the contour of the part.

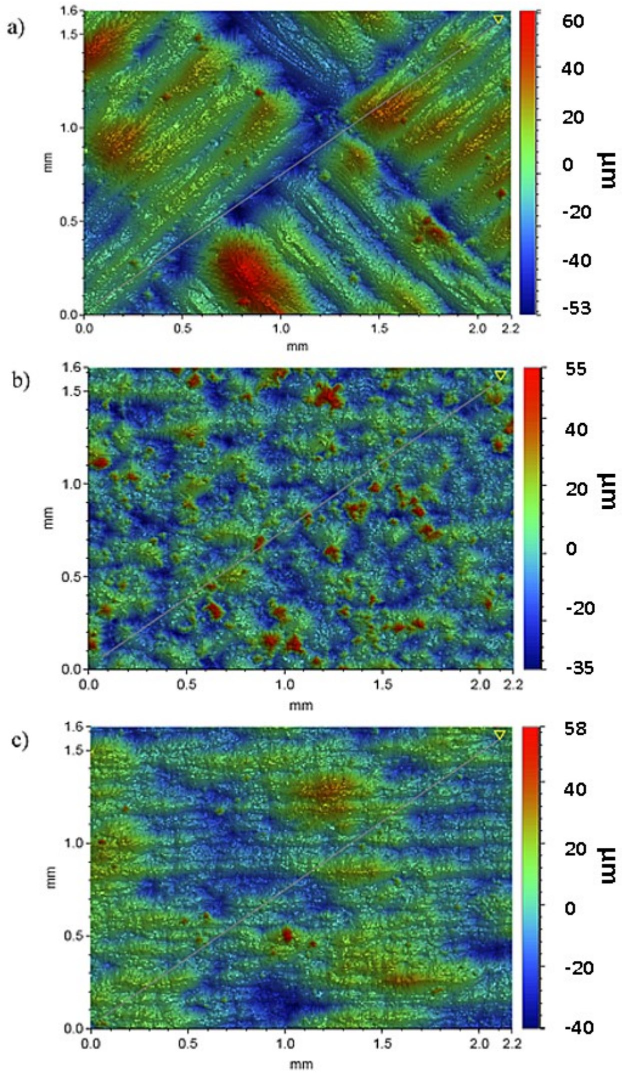

Figure 5. Surface roughness typical for a) horizontal surface; b) vertical surface; c) angled surface 45°

The depth of porosity also corresponds to the presumed weld width of approximately 150 µm. Since the scanning of each layer occurs first by hatching the surface and subsequent single scanning of the contour, the elimination of the subsurface porosity can consist of repeated contour scan before and after hatching the surface, or by doubling the contour by one hatch distance towards the material.

3.3 Surface roughness

Fig. 5 shows the surface structure typical for the surfaces with different orientation. The surface roughness parameters Sa and Sz were evaluated. A 95% confidence intervals were calculated from the measured values and the results were plotted to the chart depending on the surface orientation and the number of recycling cycles. Fig. 6 shows the course of parameter Sa for the sample at position 2 (the center of the platform) for all three evaluated surfaces. From the evaluated Sa and Sz parameters it is clear, that no noticeable trend has been developed with increasing number of recycling cycles. The result is therefore in accordance with previous knowledge.

As expected, the highest roughness and scattering occurs on a horizontal surface where the texture of the printing strategy is noticeable, ranging from Sa 10 to 12 µm. On the vertical surfaces and a 45 ° angled surfaces, the roughness values vary steadily slightly below 8 µm. On a sloping surface it is shown that a low layer thickness of 30 µm suppressed the staircase effect and does not cause higher roughness values.

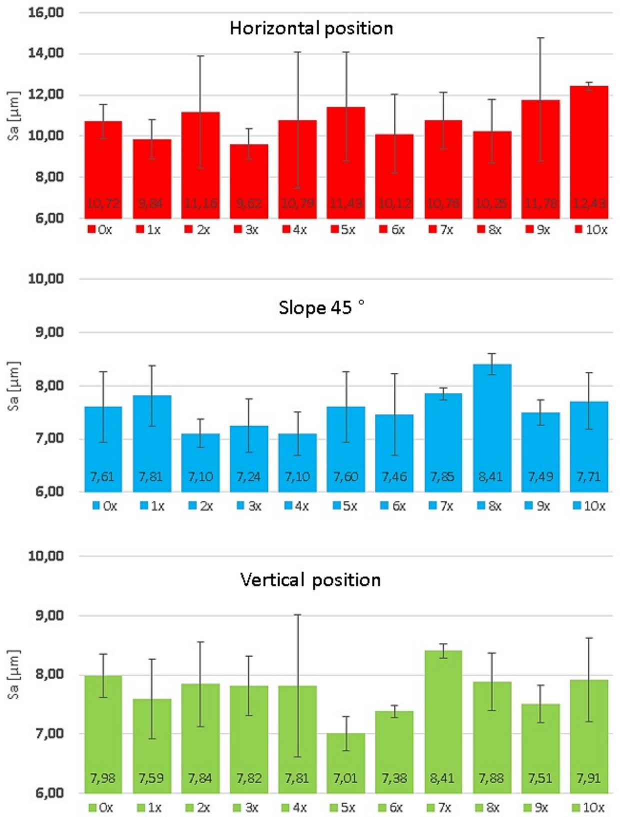

Figure 6. Sa evaluation parameter of roughness on the surfaces of cubes in position 2, depending on the number of recycling

However, abnormally high values of roughness on the vertical surface at position 1 were measured throughout the recycling test. Because the position is closest to the N_2 gas supply and the position of the cube is suitably arranged, this increase can not be explained by burnt particles contamination from the process at these platform locations.

4 CONCLUSIONS

A total of 33 cubes were built on Concept Laser M1 Cusing (Concept Laser GmbH) and 12 samples of powder were analysed. Measurement of particle size distribution showed that recycling did not have a significant effect on the powder characteristics. The mean value of D_{50} is in the range of 1.5 µm. Since the ratio between the weight of the powder used and the volume of the build job is very high, it was not significantly contaminated by burnt particles. This corresponds to the amount of weight loss before and after sieving, which ranged from 100 to 150 g. The mesh size of 63 µm, which is at the limit of the largest particles present in the powder, is sufficient to remove the sintered and burnt particles. There was also no loss of the smallest particles, which usually occurs during handling and sieving. This is most likely due to the parameters of the powder used, which does not contain the smallest particles with dimensions less than 10 or 5 µm.

Tomographic analysis showed a very high relative density of the hatched material - about 99.96%. These values did not change during the test, which corresponds to the results of laser powder diffraction. The nature of the pores was spherical with a diameter usually up to 50 µm, which corresponds to the metallurgical pores.

In terms of porosity localization, it was found that the most significant number of pores is concentrated below the surface of the material at depths of 100 to 150 µm. Sub-surface porosity is caused by the inadequate connection of the volumetric and the contour area of the part. The depth of porosity also corresponds to the presumed weld width of approximately 150 µm. Since the scanning of each layer occurs first by hatching the surface and subsequent single scanning of the contour, the elimination of the subsurface porosity can consist of repeated contour scan before and after hatching the surface, or by doubling the number of contour trajectory.

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