

A CONTROL OF SURFACE QUALITY IN SELECTIVE LASER SINTERING ADDITIVE MANUFACTURING WITH RECLAIMED POLYAMIDE MATERIALS

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Abstract

In selective laser sintering (SLS) additive manufacturing (AM), a substantial amount of polyamide 12 materials remains un-sintered, recyclable, and reusable. However, using reclaimed polyamide 12 powder in SLS results in undesirable part surface finish. Very limited research has been done on the improvement of part surface quality and results barely exist on improving or modifying the surface quality of parts using extremely aged powders (powders held close to the heat-affected zones). Aiming to improve the surface quality, we propose a novel approach for SLS with (extremely) aged polyamide 12 powders. By combining material preparation, powder and part characterizations, and SLS with a customized method of post-heating, we obtain parts with improved surface quality (e.g., reduced roughness and porosities, and eliminated un-sintered particles). Particularly, parts 3D-printed using the 30%-30%-40% new-aged-extremely aged mixed powders exhibit the smoothest and flattest surface with no unmolten particles and nearly zero porosity.

Introduction

Selective laser sintering (SLS) is one widely adopted technique in additive manufacturing (AM) to manufacture high-quality polymeric and metallic components [1-4]. Compared with other SLS materials such as metals and ceramics, polymeric powder materials offer benefits in low processing temperatures, controllable flowability and high corrosion resistance [5-7] in trade of strength. Particularly, as one of the most important semi-crystalline thermoplastic polymer materials, polyamide 12 and its reinforced/filled forms generate SLS parts with superior mechanical properties over general amorphous materials [2].

The microstructures of polyamide 12 materials consist of a series of carbon atoms and the amide groups (-NHCO-), and show both amorphous regions and crystalline regions [1, 8]. Due to the open chain ends, the molecular structures of polyamide 12 materials are prone to molecular changes at high temperatures and during laser-material interactions. In particular, post-condensation, chain scission and chain crosslinking reactions form the essential degradation mechanisms of polyamide 12 powders [9, 10], and induce different material properties.

The formation of large particles aggregated from small pieces is a predominant property change for reclaimed polyamide 12 powders [1]. These large particles cause deteriorated surface finish with unmolten high-melting-point pieces in the specimens [11]. With multiple times of reuse and repeated expansion/shrinkage in the fabrication cycles, the surface of the reclaimed polyamide 12 powders exhibits increasing cracks and fragments, lowering the part surface quality [12]. Also, compared to 3D printed parts using new powders with fibrillar spherulites

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dominating the morphologies, parts using reclaimed powders contain coarse spherulites with rough and uneven surface finish due to post-crystallization and spherulite growth [1, 13].

More at the level of part quality, S. Dadbakhsh et al. [1] clarified the effects and the corresponding mechanisms of in-process aging on the microstructures of polyamide 12 specimens in SLS. M. Pavan et al. [14] investigated how thermo-temporal effects on the SLS polyamide 12 impact part quality at both micro- and macro-levels by testing the samples using a mixing ratio 50/50 new/recycled powder. D.T. Pham et al. [15] and W. Yusoff et al. [13] developed different amendment strategies through optimizing the important SLS process parameters to reduce or eliminate the “orange peel” surface texture when using reclaimed polyamide 12. J. Guo et al. [16] presented an experimental and analytical study to clarify the interrelations between surface quality and process parameters.

Despite the aforementioned literature, it remains not clear how to maximize surface quality when using reclaimed powders. Moreover, there is a lack of understanding in surface characteristics of parts sintered using extremely aged polyamide 12 powders. Held close to or wrapped by the heat-affected zones (HAZs)[†], these powders go through severe degradations during the sintering process. We show, however, that such expensive materials can be reused to produce parts with fine surface textures, reduced porosities, and free from unmolten particles. By material preparation, powder and part characterizations and SLS with controlled post-heating, we obtain a series of parts using differently degraded powders and different combinations. Then after surface cleaning, we examine the surface morphologies of these parts and evaluate the characteristics of the surface morphologies. The result is that the undesirable surface finish of parts printed using reclaimed polyamide 12 powders can be drastically improved after using the proposed strategy.

Design of the proposed SLS for reclaimed polyamide 12

The proposed method involves material preparation (Step 1) and SLS with post-heating (Step 2). More specifically, Step 1 includes powder collection and powder preprocess, and Step 2 covers powder mixing, parameter control, SLS with post-heating and evaluation. Details of these procedures are explained in the following parts.

Material preparation

Powder collection

New polyamide 12 powders were purchased from EOS Corp, and the reclaimed powders were collected from standard SLS processes on an EOS P 390 machine. Polyamide 12 powders with 3 different degradation levels were used in this work: (1) new polyamide 12 powders with no additional heat treatment; (2) aged polyamide 12 powders located far away from the HAZs in the SLS chamber and are reused in industries; (3) extremely aged polyamide 12 powders adjacent to or wrapped by the HAZs that are not reused in industries currently.

Powder preprocess

Due to the large particles or part debris caused by the high temperature and laser induced degradation/aging, when recoating with the extremely aged powders, the powder bed is uneven and rough on surface. A sieving process was applied to preprocess the extremely aged powders for an even recoating surface. This process was conducted in a fume hood using a sieve with a mesh size of 200 μm . The time for sieving a batch of 500 ml powders is around 2 hours including

[†] Areas close to the laser-material interaction during sintering.

preparation and post cleaning. After sieving, 84%~90% of the reclaimed materials can be collected and well recoated.

Proposed SLS and post-heating

Powder mixing

Four different kinds of powder combinations were used in this work: (i) pure powders, (ii) new and aged powder mixture, (iii) new and extremely aged powder mixture, and (iv) new, aged, and extremely aged powder mixture. We conducted various volume mixing percentages for these powder combinations.

Parameter control

Six key parameters were tested in the experiments: preheating temperature, laser power, laser speed, scan spacing, layer thickness and layer numbers of samples. A variety of parameter settings are suitable for new powders. However, existing parameter settings seldom apply to the case using extremely aged powders in presence of the degraded material properties. The general principles to select the proposed parameter settings are that (i) these parameters are equal or close to industrial norms and (ii) the pure new, aged and extremely aged polyamide 12 powders can all be successfully printed into parts. The nominal parameter settings selected in this work were: preheating temperature, 160 °C; scan speed, 3000 mm/s; laser power, 18 W; scan spacing: 0.3 mm; layer thickness, 150 μm. In this work, samples are all printed with 3 layers and using the optimized parameter settings selected to explore the part surface quality improvement.

SLS with post-heating

Process: The SLS machine used in the paper is an in-house built open-configuration research testbed, with features comparable to commercial machines. The proposed SLS and post-heating control apply tailored heating after the core laser-material interaction with optimized processing parameters. The post-heating here keeps parts at the preheating temperature (160 °C) for a controlled time after the sintering process. In this work, we tested 0 second (no post-heating), 60 seconds, 120 seconds, and 300 seconds of post-heating to different specimens to explore the influences of post-heating on part surface morphologies. Table 1 shows details of the proposed five-stage SLS. In Stage 1, we 3D printed the benchmark part using 100% new powders with no additional post-heating, and the part was used as a reference to evaluate the surface qualities of other parts. In Stage 2, parts were printed using 100% new powders with different post-heating time (60 seconds, 120 seconds, and 300 seconds) to identify the influences of post-heating on part surface qualities when using 100% new powders. In Stage 3, we printed parts with 100% extremely aged powders at different post-heating time. After comparing the surface qualities of parts using reclaimed powders with and without post heating, 300-second post heating appears most effective for reclaimed powders. In Stage 4, parts were 3D printed using different powder mixtures; parts were also 3D printed with and without 300-second post-heating to study the effects of post-heating on part surface quality when using mixed powders. In Stage 5, we used the mixtures of three differently degraded powders (new, aged, and extremely aged powder mixtures) with and without 300-second post-heating. The results form the basis to identify the effects of post-heating on part surface morphology.

Table 1 Post-heating based SLS process (The mixed powders are in volume percentages)

Stages	Objectives	Combinations	Post heating
Stage 1	To print the benchmark sample	100% new powder	No
Stage 2	To explore the influences of post-heating on part surface morphologies	100% new powder	60 s
		100% new powder	120 s

	when using new powders	100% new powder	300 s
Stage 3	To explore the influences of post-heating on part surface morphologies when using reclaimed powders	100% extremely aged powder	No
		100% extremely aged powder	60 s
		100% extremely aged powder	120 s
		100% extremely aged powder	300 s
Stage 4	To explore the influences of post-heating on part surface morphologies when using mixed powders	50% new + 50% aged	No
		50% new + 50% aged	300 s
		70% new + 30% extremely aged	No
		70% new + 30% extremely aged	300 s
		50% new + 50% extremely aged	No
		50% new + 50% extremely aged	300 s
		30% new + 70% extremely aged	No
		30% new + 70% extremely aged	300 s
		10% new + 90% extremely aged	No
10% new + 90% extremely aged	300 s		
Stage 5	To explore part using new-aged-extremely aged mixed powders	30% new + 30% aged + 40% extremely	No
		30% new + 30% aged + 40% extremely	300 s

Evaluation: After surface cleaning to remove debris on the part surface, we used Scanning Electron Microscope (SEM) to compare surface morphologies between the benchmark part and the evaluated parts in Table 1. Compared features include particle coalescence performances, part porosity, the number of unmolten particles, surface microstructures and roughness. The details are explained in the next section.

Experimental results and discussions

Surface quality improvements of the 3D-printed parts

Stage 1: printing the benchmark sample

Figure 1 presents the SEM images of the 3-layer benchmark sample printed using 100% new polyamide 12 powders without additional heat treatment. In Figure 1 (a) - (c), the part exhibits a smooth and flat surface with no unmolten particles. Meanwhile, high porosity is observed from these images, suggesting an insufficient densification. At a high magnification ratio of 10000 (Figure 1d), some fine lamellae or spherulitic regions in an amorphous matrix are observed. The spherulites radiate from the center and grow in a ringed extinction pattern. These surface characteristics of the benchmark part are used as references to evaluate the surface quality of the other parts.

Stage 2: the influences of post-heating on part surface morphology when using new powders

Compared with Stage 1 (100% new samples with no post-heating), the parts obtained in Stage 2 (printed using 100% new polyamide 12 powders with 60 seconds, 120 seconds and 300 seconds post-heating) exhibit very similar surface morphologies of smooth and flat surfaces with high porosity and no visible unmolten particles. This is an indicator that post-heating barely has an effect on the surface qualities of samples 3D printed using new polyamide 12 powders.

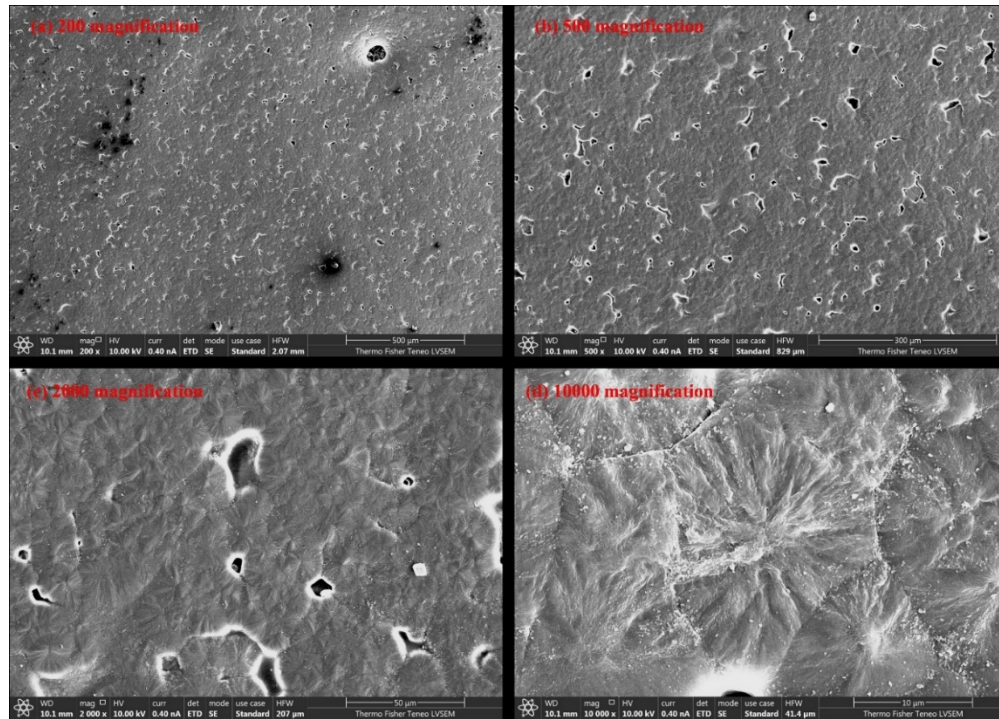
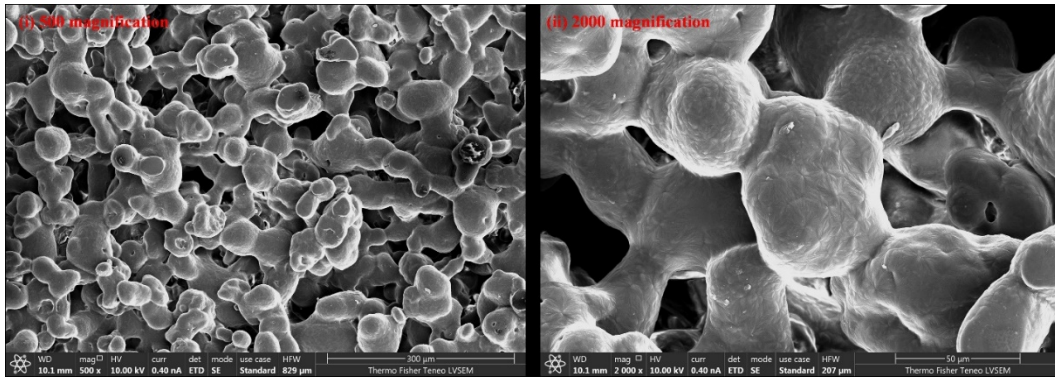


Figure 1 The SEM images of benchmark part using 100% new polyamide 12 powders at different magnification ratios (a) 200, (b) 500, (c) 2000, and (d) 10000

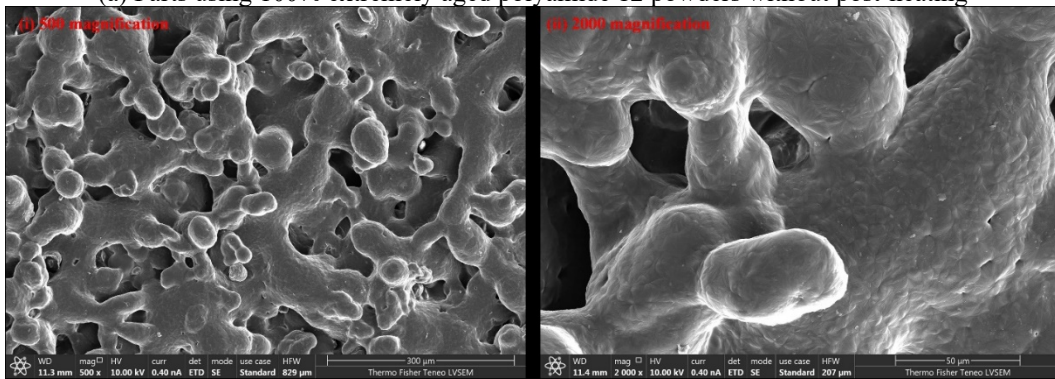
Stage 3: the influences of post-heating on part surface morphology when using reclaimed powders

Figure 2 exhibits the SEM images of parts using 100% extremely aged powders with 0 second (no post-heating), 60 seconds, 120 seconds, and 300 seconds post-heating at magnification ratios of (i) 500 and (ii) 2000. From Figure 2a to d, we observe a gradual melting and coalescing process with post-heating time increasing. In Figure 2a (no post-heating), multiple layers of insufficiently melt particles are observed, and every two or more particles form a neck-like bonding due to the reduction of the free surface energies of the particles triggered by high temperature and intense laser-material interaction. With 60 seconds of post-heating (Figure 2b), the particles bonded by the neck-like structures migrate together. The migration becomes stronger with longer post-heating (Figure 2c). Finally, a large unit, a well-consolidated surface forms with little porosity and few unmolten pieces (Figure 2d).

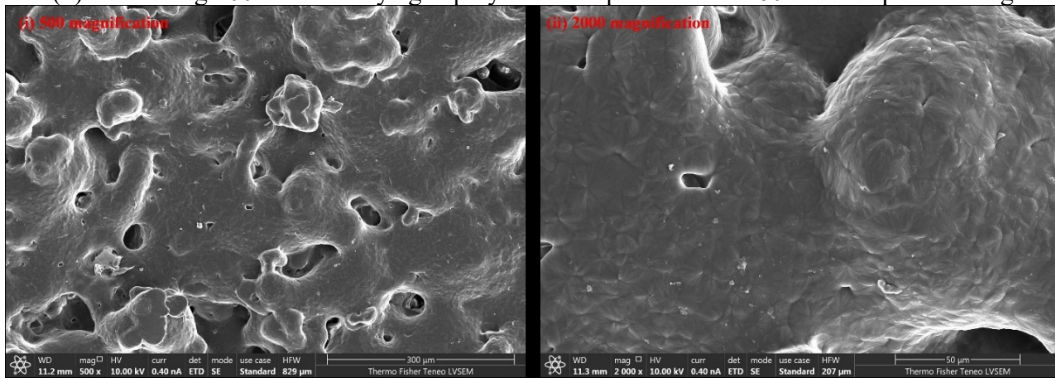
Comparing Figure 1 (100% new powders with no post-heating) with Figure 2a (100% extremely aged powders with no post-heating), we observe an obvious difference in surface morphology. Numerous visible unmolten particles arise and fuse together to form porous structures in the parts 3D printed using extremely aged powders (Figure 2a). The results indicate the existence of high-melting-point pieces in the extremely aged powders. Given the above, we conclude that the post-heating process helps to improve the surface quality of the printed parts using extremely aged powders by maximizing the coalescence and consolidation, and a well-consolidated surface obtains with 300 seconds post-heating (Figure 2d).



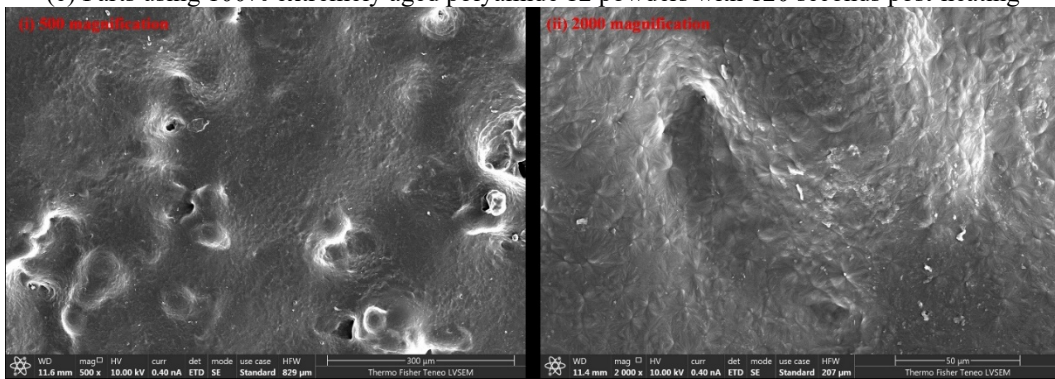
(a) Parts using 100% extremely aged polyamide 12 powders without post-heating



(b) Parts using 100% extremely aged polyamide 12 powders with 60 seconds post-heating



(c) Parts using 100% extremely aged polyamide 12 powders with 120 seconds post-heating



(d) Parts using 100% extremely aged polyamide 12 powders with 300 seconds post-heating

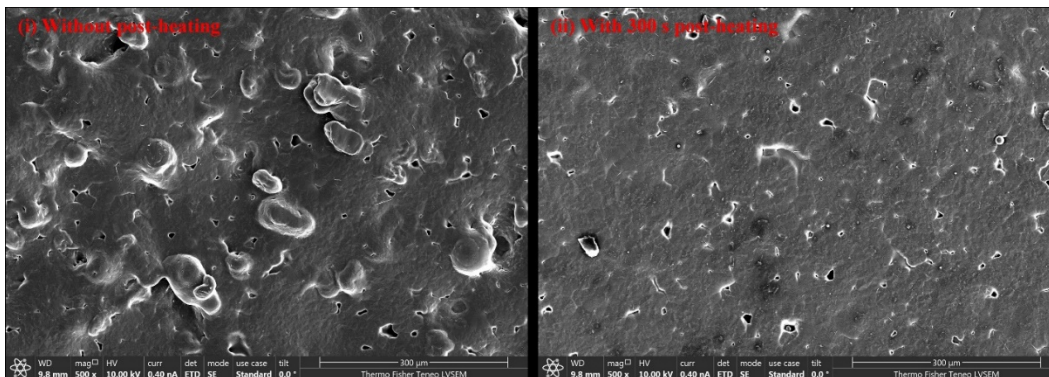
Figure 2 Parts printed using 100% extremely aged polyamide 12 powders with and without post-heating

Stage 4: the influences of post-heating on part surface morphology when using mixed powders

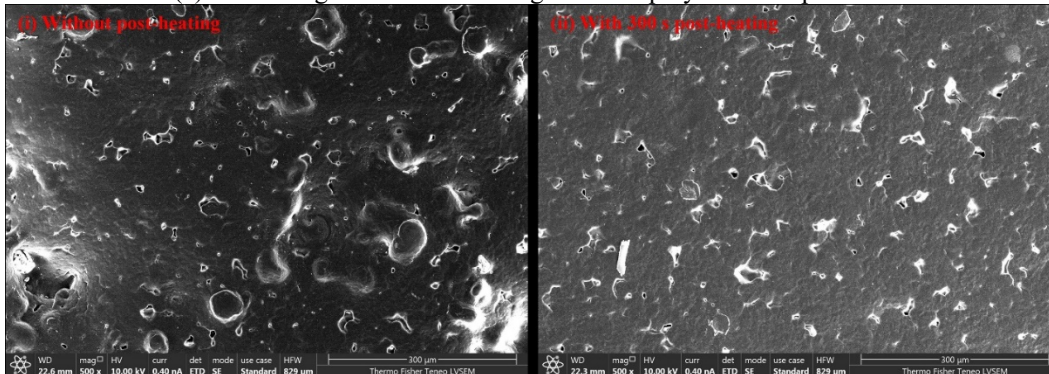
Figure 3 presents the SEM images of the parts using new-aged and new-extremely-aged polyamide 12 powder mixtures with (i) no post heating and (ii) 300 seconds post-heating. Figure 3 (a) - (c) shows the SEM images of parts 3D printed using 50%-50% new-aged mixed powders, 70%-30%, and 50%-50% new-extremely-aged mixed powders, respectively. Smooth and flat surfaces with several unmolten particles are observed with no post heating (Figure 3 a (i), b (i), and c (i)), suggesting that good particle coalescence behaviors are achieved when using 50% or more new powders. Meanwhile, the unmolten particles dramatically decrease, and smooth and flat surfaces with high porosity are obtained with 300 seconds post-heating (Figure 3 a (ii), b (ii), and c (ii)). These surfaces are similar to those of parts using 100% new powders, suggesting a significant improvement of surface qualities by our post-heating.

Figure 3 (d) show the SEM images of parts 3D printed using 30%-70% new-extremely-aged mixed powders. With no post-heating, samples exhibit severe deteriorated and distorted surface morphologies with irregular holes or porous and plenty of unmolten particles (Figure 3 d (i)). With 300-second post-heating, the improved surfaces have relatively flat morphologies with several unmolten particles (Figure 3 d (ii)).

Figure 3 (e) exhibit the SEM images of parts 3D printed using 10%-90% new-extremely-aged powder mixtures. With no post-heating, the SEM images exhibit little completed surfaces with numerous unmolten particles and multi-layer porous structures (Figure 3 e (i)). These surfaces are similar to the images when using 100% extremely aged powders. With 300-second post-heating, the images exhibit very smooth and flat surfaces with almost no porous and several unmolten particles (Figure 3 e (ii)). The quality of the obtained surfaces is even better than the part printed using 100% new powders.



(a) Parts using 50% new + 50% aged mixed polyamide 12 powders



(b) Parts using 70% new + 30% extremely aged mixed polyamide 12 powders

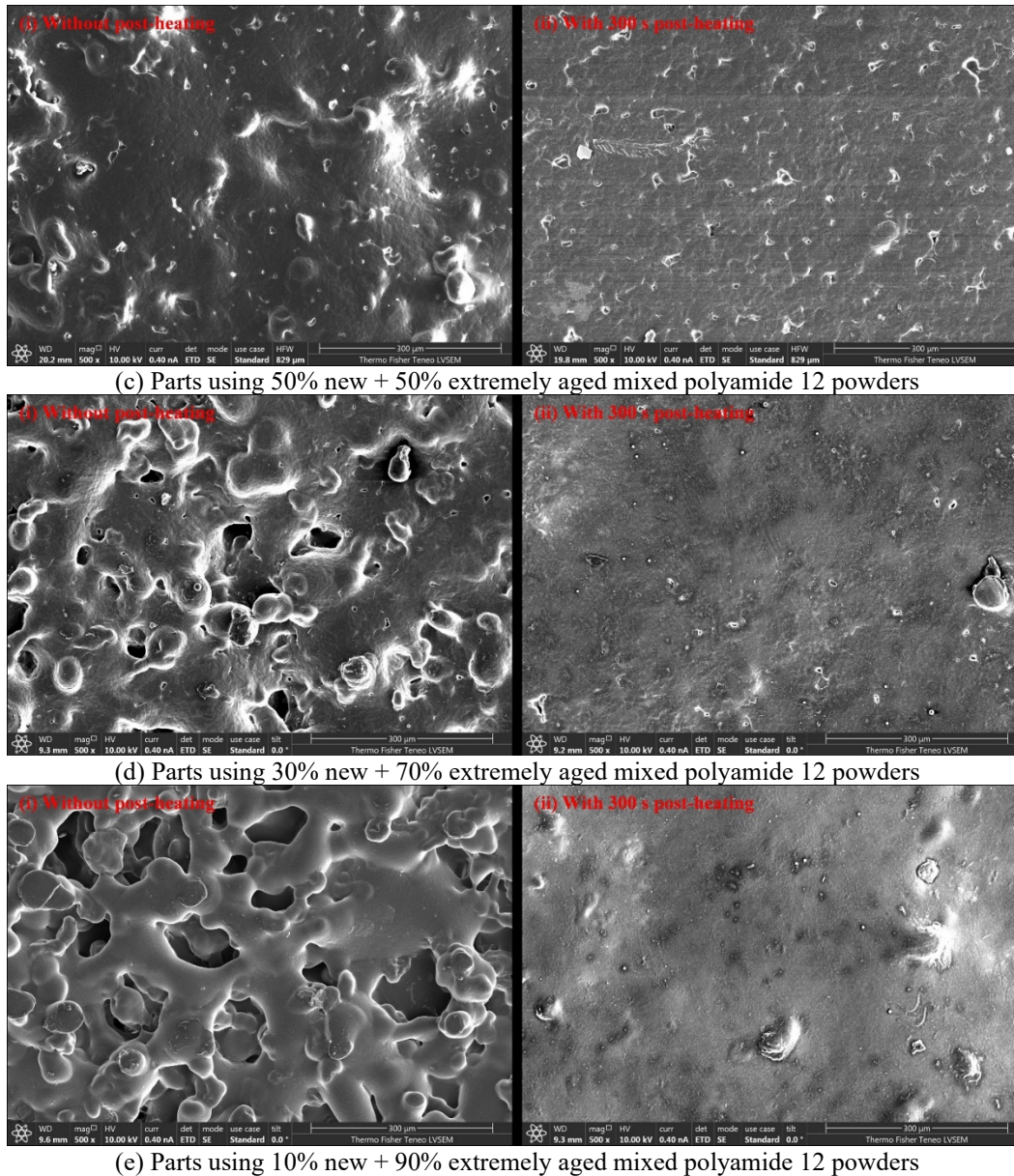


Figure 3 Parts using new-aged mixed or new-extremely-aged mixed polyamide 12 powders with no post-heating and with 300 seconds post-heating

Stage 5: the influences of post-heating on part surface morphology when using new, aged, and extremely aged powder mixtures

Figure 4 demonstrates the images of parts using 30%-30%-40% new-aged-extremely-aged mixed polyamide 12 powders with (i) no post-heating and (ii) 300 seconds post-heating. In the combination, 30% new powders and 70% reclaimed powders were used. When using 30% new powders with no post-heating, the parts in Figure 4 (i) have better-coalesced surfaces compared to the part in Figure 3d (i). Because much more extremely aged powders were used in Stage 4 (Figure 3d, 70%) than in Stage 5 (Figure 4, 40%), making it more difficult to fuse the materials. With 300-second post-heating, the part 3D printed using 30%-30%-40% new-aged-extremely-aged mixed powders has the smoothest and flattest surface with no unmolten particles and almost no porosities (Figure 4 (ii)).

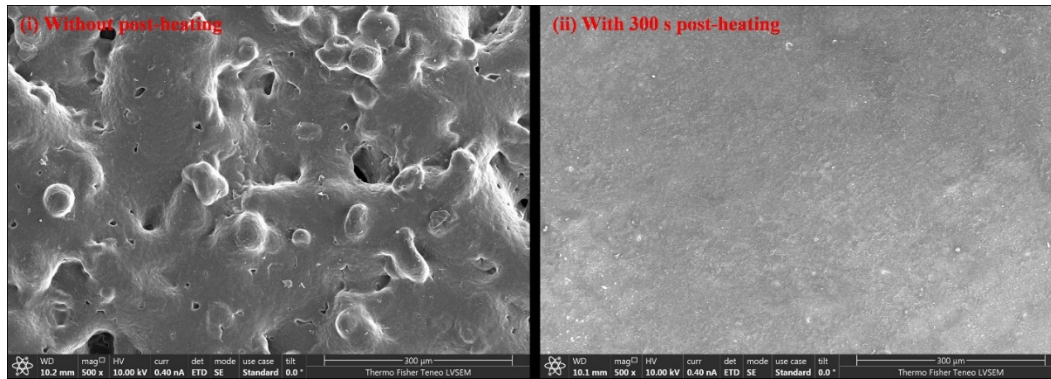


Figure 4 Parts using 30%-30%-40% new-aged-extremely-aged mixed polyamide 12 powders with no post-heating and with 300 seconds post-heating

Discussions

Unmolten particles, coalescence, roughness and porosity

Figure 5 shows the SEM images of 3D printed parts with no post-heating to compare the number of unmolten particles, coalescence performances and roughness. In general, parts using 100% new powders have the best coalescence performance, and almost no unmolten particles (Figure 5a). However, parts using more extremely aged powders exhibit worse coalesced surfaces with drastically increased unmolten particles (Figure 5 (b)-(h)). Parts using more new powders have smoother and flatter surfaces, while parts using mixed powders obtain worse surfaces as the percentages of reclaimed powders increase from 30% to 70% (Figure 5 (b)-(e) and Figure 5 (h)). In particular, when using 90%~100% extremely aged powders (Figure 5 (f)-(g)), numerous insufficiently melt particles are observed, and no consolidated surfaces form.

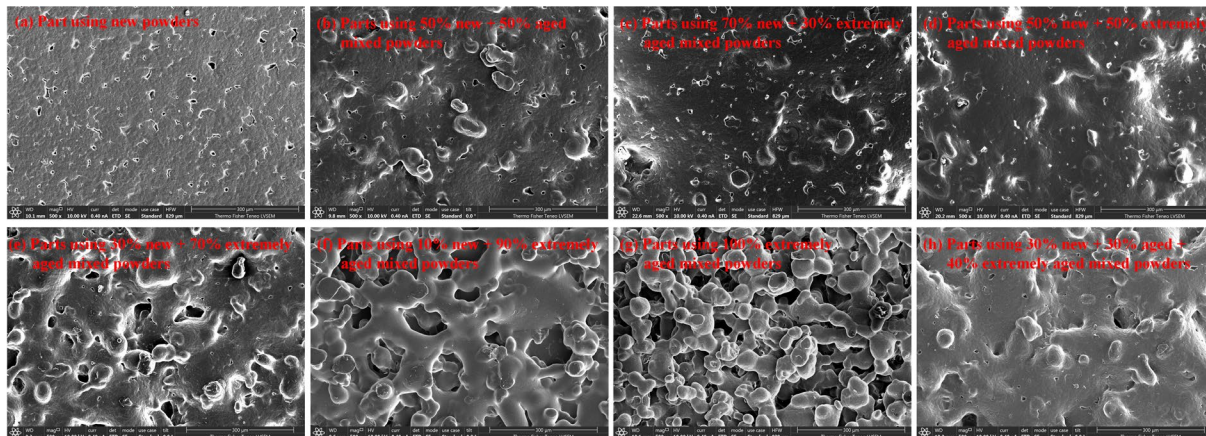


Figure 5 Comparisons of unmolten particles, coalescence and roughness of parts using polyamide 12 powders of different combinations without post-heating

Figure 6 presents the SEM images of 3D printed parts with 300 seconds post-heating to compare the number of unmolten particles, coalescence performances and roughness. Except for the part using 100% new powders, the images in Figure 6 exhibit better surfaces with enhanced coalescence, decreased unmolten particles and improved smoothness with the proposed post-heating. In Figure 6 (a)-(d) and (h), the images of parts printed using 100% new powders, 50%-50% new-aged mixed powders, 70%-30%, 50%-50% new-extremely-aged mixtures, and 30%-

30%-40% new-aged-extremely-aged mixed powders, respectively, the surfaces are smooth and flat without any unmolten particles. However, in Figure 6 (e)-(g), the images of parts printed using 30%-70%, 10%-90% new-extremely-aged mixtures, and 100% extremely aged powders, respectively, the parts exhibit several unmolten pieces. These results indicate that the parts using a high percentage of new powders obtain improved smooth and flat surfaces with no unmolten particles with post-heating. These surfaces are comparable to those of part using new powders. The parts using a high percentage of extremely aged powders get improved surfaces with post-heating, but there still exist several unmolten particles.

In Figure 6, the parts also display different porosities. In Figure 6 (a) to (c), the images of parts printed using 100% new powders, 50%-50% new-aged mixtures, and 70%-30% new-extremely-aged mixtures, respectively, there are more large size pores than the other parts. In Figure 6 (d), the images of parts printed using 50%-50% new-extremely-aged mixtures, the number and size of pores decrease significantly. In the remaining images of Figure 6 (e)-(h), almost no pores are observed. These results suggest that the parts using more new powders tend to have more pores. The parts using more extremely aged powders with post-heating display lower porosity due to better consolidation.

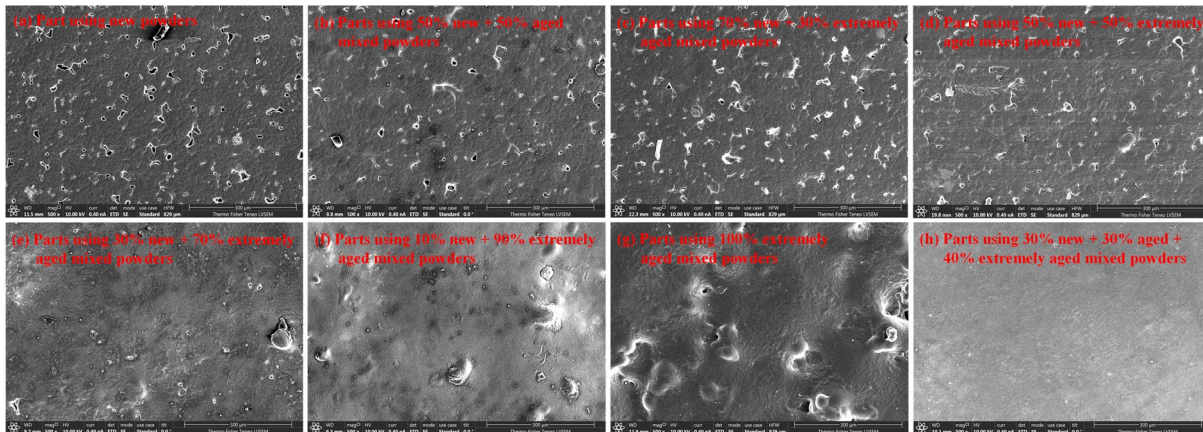


Figure 6 Comparisons of unmolten particles, coalescence and roughness of parts using polyamide 12 powders of different combinations with 300 seconds post-heating

Microstructures

Microstructure is another important variable in the printed parts when using reclaimed powders. Figure 7 a and b present the microstructures of the parts with no post-heating and with 300 seconds post-heating at a magnification ratio of 10000, respectively. Different powders and powder mixtures yield different microstructures. Parts using new powders exhibits fine fibrillar/lamellae spherulitic regions in amorphous matrixes (Figure 7a), due to the aggregations of chain-folded crystallites radiating from the center and growing to be spherical in shape. On the other hand, the parts using extremely aged powders present coarser spherulites spreading all over the matrix (Figure 7a). Due to the slightly aging, the spherulite roughness in the part using aged powders behaves in the middle between those observed in the parts using new and extremely aged powders.

The parts using different powders with 300 seconds post-heating show similar characteristics (Figure 7b). Parts using new powders show fine lamellae. Extremely aged powders lead to coarse spherulites. And parts using aged powders show intermediate

morphologies. It can be concluded that the microstructures of parts are largely impacted by the aging status of powders rather than post-heating.

(a) Polyamide 12 parts without post-heating

(b) Polyamide 12 parts with 300 seconds post-heating

Figure 7 Microstructure examinations of (a) polyamide 12 parts without post-heating, and (b) polyamide 12 parts with 300 seconds post-heating at a magnification of 10000

Conclusions

This work proposes an SLS with post-heating to improve surface quality of 3D printed parts using reclaimed polyamide 12 powders. The proposed method decreases roughness and porosity of the printed parts, and eliminates unmolten particles. The effects of post-heating on the surface quality using different powder mixtures were studied. In particular, SEM reveals surface features, including the number of unmolten particles, coalescence performances, roughness, porosity and microstructures.

The tests suggest that post-heating barely affected the surface quality of parts using 100% new powders. The parts using 100% extremely aged powders with no post-heating exhibit multiple layers of insufficiently melt particles. However, a 300-second post-heating yields a well-consolidated surface with little porosity and a drastically reduced un-molten particles. The unmolten particles disappear on the parts using 50% or more new powders with 300-second post-heating, showing smooth and flat surfaces with high porosity. When using 90% reclaimed powders, numerous visible unmolten particles and multi-layer porous structures occur in the case with no post-heating. With the proposed 300-second post-heating, we obtain smooth and flat surfaces with almost zero porosity and only few unmolten particles. The surface morphologies of parts 3D-printed using the 30%-30%-40% new-aged-extremely-aged mixed powders with 300-second post-heating are even better than parts 3D-printed using 100% new powders.

Acknowledgments

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