

Analysis of Ageing and Degradation Behavior of Polyamide-12 for Sustainable Additive Manufacturing

G. Venkata Nagamani¹, Sri Harsha Arigela^{2,*}

Abstract

Additive Manufacturing (AM), particularly High-Speed Sintering (HSS), has emerged as a transformative technology capable of producing complex thermoplastic parts with high efficiency and minimal material waste. The HSS process involves selective deposition of radiation-absorbing ink followed by infrared heating to consolidate polymer powders-most commonly polyamide-12 (PA12). While PA12 is widely used due to its excellent thermal stability and mechanical properties, the sustainability of HSS strongly depends on the reuse of unsintered powder. Recycled powder is subjected to repeated thermal exposure during successive build cycles, which induces ageing and degradation. However, systematic studies on the influence of powder ageing in HSS remain limited. This work investigates the degradation behavior of PA12 across three recycling iterations without refreshing. Powder and part qualities were evaluated using Scanning Electron Microscopy (SEM) for morphological changes, Melt Volume Rate (MVR) for rheological behavior, and Differential Scanning Calorimetry (DSC) for thermal transitions. Additional mechanical testing provided insight into strength retention and ductility. Results indicate progressive increases in crystallinity and melt enthalpy, accompanied by reduced bulk density, particle agglomeration, and a noticeable decline in ultimate tensile strength and elongation at break with each iteration. Thermogravimetric Analysis (TGA) further confirmed changes in thermal stability of aged powders. The findings demonstrate that powder ageing exerts a direct and significant influence on part performance in HSS. Specifically, R3 powder retained properties closer to virgin PA12, while R2 powder showed marked degradation. These results provide valuable guidance for optimizing refresh ratios and designing sustainable job boxes, thereby reducing waste and ensuring reliable part quality. Overall, the study contributes to sustainable additive manufacturing by establishing performance thresholds for PA12 powder reuse in HSS.

Keywords: High-speed sintering, additive manufacturing, PA12, differential scanning calorimetry (DSC), scanning electron microscopy (SEM), melt volume rate (MVR)

INTRODUCTION

The manufacturing industry is rapidly evolving, becoming increasingly complex, integrated, and globally connected. To stay competitive and meet diverse consumer demands, manufacturing organizations are adopting advanced knowledge management practices. The ongoing fourth industrial revolution has introduced numerous technologies, with additive manufacturing (AM) emerging as one of the most prominent. Historically, goods were handcrafted or manually assembled until the 18th century, when industrial manufacturing methods began to develop. Since then, innovations have accelerated significantly. Today, AM allows the creation of parts with highly complex geometries without incurring tooling costs, making it ideal for direct manufacturing. High-Speed Sintering (HSS) is particularly valuable in this context, as it enables the efficient production of functional components.

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Mechanical properties are critical in rapid prototyping applications, particularly when parts are intended for function testing. HSS facilitates the production of geometrically intricate parts, but the powder reuse strategy directly affects part quality and cost.

Research Objectives and Scope

In the HSS process, the final part is created by sintering PA12 powder layer-by-layer using infrared (IR) radiation. However, only about 25% of the powder bed is sintered into parts. The remaining unsintered powder, though reusable, is thermally exposed and may degrade over time. To maintain part quality and reduce production costs, manufacturers often refresh aged powder by mixing it with virgin material. However, arbitrary addition of virgin powder can either increase cost (if excessive) or reduce quality (if insufficient).

Thus, quantifying powder degradation is essential to optimize the refresh ratio. This study aims to characterize the ageing and degradation behavior of PA12 powder during the HSS process. Mechanical performance is assessed through tensile testing, while morphological and thermal degradation is evaluated using SEM, DSC, and Thermogravimetric Analysis (TGA). The study also investigates how repeated recycling affects powder usability and identifies thresholds beyond which mechanical performance deteriorates.

Literature Review

Additive Manufacturing (AM) technologies have progressed rapidly over the past decade, with Polyamide-12 (PA12) gaining significant attention due to its excellent mechanical, thermal, and recycling characteristics. Literature consistently highlights both the strengths and challenges associated with reusing PA12 powders, particularly regarding material degradation during thermal cycles. Wiles and Scott [1] explored the degradability of polyolefins, establishing early principles for understanding environmental degradation in thermoplastics. Similarly, Pawlak and Galeski [2] examined cavitation during tensile deformation, providing foundational insights applicable to the stress response of AM polymers like PA12. Further research by Camargo et al. [3] demonstrated that graphene-reinforced PLA composites printed via FDM exhibit superior mechanical behavior, suggesting potential strategies for enhancing the strength of PA12-based parts.

The sustainability of polymer composites was emphasized by La Mantia and Morreale [4], who discussed the role of eco-friendly materials in advanced manufacturing. Jakus et al. [5] further showcased the feasibility of 3D-printing high-content graphene scaffolds, illustrating possibilities for advanced PA12 composites in biomedical and electronic applications. Studies focusing on failure analysis, such as that of Torrado and Roberson [6], highlighted anisotropy in 3D-printed parts, reinforcing the need for parameter optimization. Battagazzore et al. [7] investigated the physical aging of PA12, correlating structural changes with mechanical performance loss. Similarly, Bujanovic et al. [8] provided a comprehensive overview of PA12's processing and reuse in additive manufacturing, offering a valuable framework for sustainable application.

Kumar et al. [9] and Lindner et al. [10] delved into the degradation and fatigue behavior of PA12 under environmental and mechanical stress, while Song et al. [11] evaluated performance retention in parts manufactured using recycled powder. These studies collectively confirm that PA12 can withstand multiple cycles of reuse with minimal loss of quality, provided appropriate control measures are implemented. Sauerwein et al. [12] and Buj-Corral and Bagheri [13] emphasized that long-term powder reuse significantly affects microstructure and mechanical reliability, underlining the importance of sustainability-focused research in AM. Aging effects on crystallinity and melting behavior were further substantiated by Lu et al. [14], and Rahim et al. [15] examined crystallization kinetics, aiding predictions of part quality in reused PA12.

Mechanical performance assessments by Dizon et al. [16] and sinter ability studies by Lehner and Schmid [17] contributed critical data on how powder origin and reuse history impact layer adhesion and porosity. Long-term thermo mechanical stability studies, such as those by Smith and Gupta [18], and environmental aging assessments by Sharma and Ramesh [19], reinforced the necessity of quality monitoring in AM environments.

Insights from Arigela and Kumar [20] on nonlinear tool path generation in FDM, although not specific to PA12, support the broader understanding of process optimization in AM. Meanwhile, comprehensive reviews by Dizon et al. [21], Schmid et al. [22], and Pantazopoulou et al. [23] have detailed material property shifts and degradation indicators relevant to reused powders. The influence of moisture and storage conditions on PA12 degradation was examined by Ranjan and Chopra [24], while Gibson et al. [25] and Bertoldi et al. [26] highlighted the significance of integrating PA12 into Industry 4.0 frameworks and its transition from powder to printed part. Early foundational work by Pukanszky [27] and more recent studies by Yao et al. [28] and Dotchev and Yusoff [29] addressed interfacial adhesion and anisotropy issues in sintered PA12 components.

The incorporation of fillers to improve composite performance was reviewed by Yadav et al. [30], while morphological and chemical assessments of PA12 and silica powders were explored by Uchino et al. [31], Alloul et al. [32], Ellerbrock et al. [33], and Al-Harbi et al. [34]. These analyses are critical for determining reusability and part quality in high-precision AM applications. Ning et al. [35] and Alo et al. [36] analyzed resin and powder modification techniques for performance optimization, while Costache et al. [37] conducted detailed studies on recycled PA2200 powder. Sanders et al. [38] provided a direct evaluation of the physical aging effects in PA12 through multiple reuse cycles in powder bed fusion, which is particularly relevant to the scope of the current study. Finally, the broader sustainability implications of AM technologies were addressed by DM [39], Gebler et al. [40], and McCarthy et al. [41], emphasizing the global significance of material reuse, process control, and environmental impact in the AM industry.

Problem Statement

High-Speed Sintering (HSS) is a recent powder bed fusion technique based on inkjet printing. During the manufacturing process using PA12 powder, significant quantities of unused powder are generated in two primary forms:

- R2: Excess powder surrounding the built parts, collected during post-processing
- R3: Powder that falls into the lower chamber after being spread over the bed

To enhance sustainability and reduce waste, it is essential to understand the degradation behavior of these recycled powders. This study evaluates the ageing effects of R2 and R3 powders across successive build iterations, analyzing their properties to determine usability in future builds.

Experimental results suggest that R3 powder retains properties comparable to those of virgin powder, whereas R2 powder shows partial degradation. The research aims to optimize the reuse of both powders by determining the ideal mixing ratio with virgin powder. Quantitative analysis through DSC, TGA, SEM, and mechanical testing will be used to assess ageing and define parameters for a sustainable job box design.

METHODOLOGIES

General Procedure for Printing Specimens

Specimens were designed using CAD software and imported into Rapix3D for slicing on the voxeljet HSS machine. The system was preheated for one hour before initiating the printing process. The following machine parameters were used throughout:

- *Part bed temperature:* Max 173°C
- *Print density:* 7 (on a 0–7 scale)

- *Startup layers*: 60
- *Finish layers*: 30
- *Recoated powder delay*: 10 cycles

Preliminary tests on orientations of 0° , 30° , 45° , 60° , and 90° revealed that the 0° orientation yielded the best mechanical performance. This orientation was therefore used consistently in all experiments.

The Build Orientation Schematic, as shown in Figure 1, illustrates the orientation axes: +X (front of the machine), +Z (upward build direction), and +Y, which is inclined at 45° from the X-axis.

Sustainable Job Box Design

This research emphasizes designing a sustainable job box to minimize recycled powder generation. The R2 and R3 powders are collected separately and quantified. The sustainable build strategy involved optimizing space utilization within the job box with a minimum spacing of 10 mm between parts.

Table 1 outlines the five major part categories used in the sustainable job box design for High-Speed Sintering (HSS) with PA12 powder. Each category represents a type of printed component, along with its refilling percentage, which indicates how much of the job box volume is allocated to that part type.

By designing an average job box with these categories, the amount of waste powder was significantly reduced. Six consecutive jobs were printed entirely using virgin powder. The collected recycled powders were categorized as R2 and R3 for further evaluation.

Procedure for Homogeneous Mixing

Sieving was the initial step to eliminate oversized particles generated during post-processing. A single pass sieving process was preferred to prevent the powder from becoming overly dry and coarse due to excessive handling.

After sieving, powders were thoroughly mixed to ensure homogeneity. The mixed powders (R2 and R3) were then prepared on specific ratios for subsequent testing and evaluation.

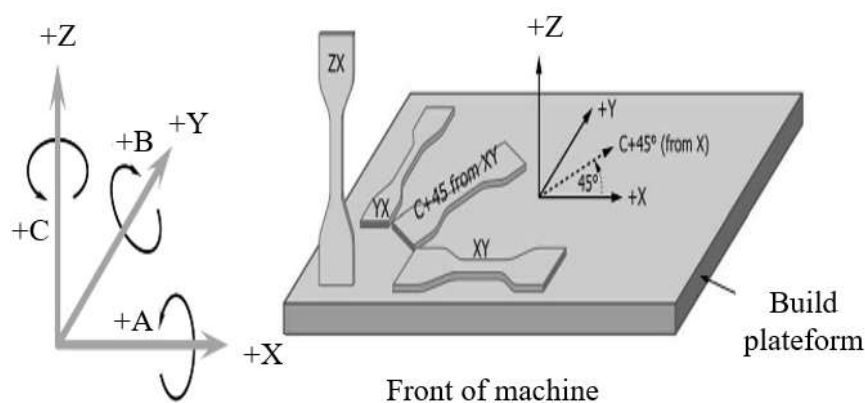


Figure 1. Possible angle of orientation possible for printing specimens.

Table 1. Part Categories

Type	Description	Refilling percentage
Type 1	Parts with lattice structure	35%
Type 2	Flexible parts	35%
Type 3	Gear shift knobs and prototypes	10%
Type 4	Micro nanotubes/Tensile specimens-	10%
Type 5	Emission filters and Housing prototypes	10%

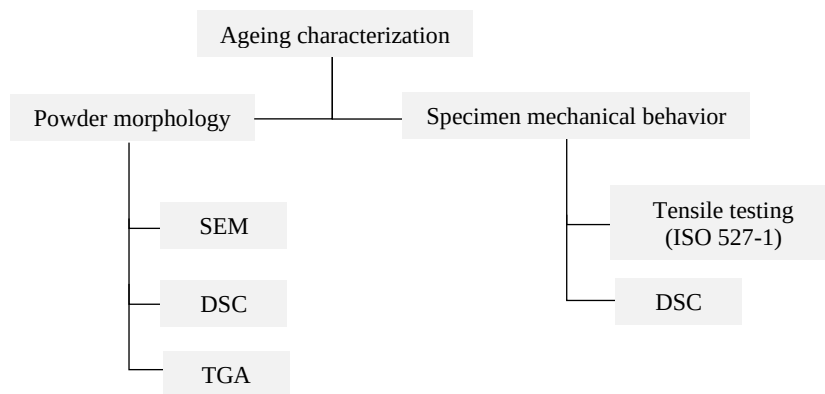


Figure 2. Work plan for the characterization of PA12 on HSS.

Quantification Techniques

To effectively evaluate the aging process and determine the quality degradation of PA12 powders, a variety of tests were performed, as illustrated in Figure 2.

- Scanning Electron Microscopy (SEM) for morphological analysis
- Differential Scanning Calorimetry (DSC) for thermal transitions (melting and crystallization)
- Thermogravimetric Analysis (TGA) for thermal stability and degradation
- Tensile testing for mechanical properties (tensile strength, Young's modulus, elongation at break)

Table 2 provides the material specifications for the polymer used in the High-Speed Sintering (HSS) experiments—EOS PA2200, which is a commercial grade of Polyamide-12 (PA12)

The Scanning Electron Microscopy (SEM) set up parameters represented in Table 3, used to analyze the morphology of PA12 powder across different recycling iterations. These settings are crucial for ensuring consistent imaging quality and accurate interpretation of particle shape, size, and agglomeration behavior.

The tensile test setup parameters used to evaluate the mechanical properties of PA12 specimens across different recycling iterations referred in the Table 4.

RESULTS & DISCUSSION

Effects of Orientation on Mechanical Properties

A series of experiments were conducted using specimens printed at varying build angles (0°, 30°, 45°, 60°, and 90°) to determine the optimal orientation for mechanical performance.

Table 2. Material used.

Property	EOS PA2200 (PA12)
Commercial name	EOS PA2200 (PA12)
Powder type	Nylon 12
Mean particle size	56 μm
Bulk density	0.45 g/cm^3
Melting point	172–180°C

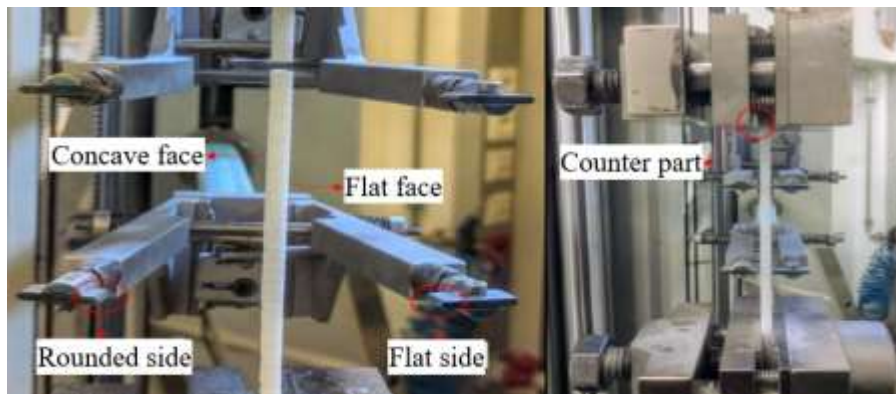
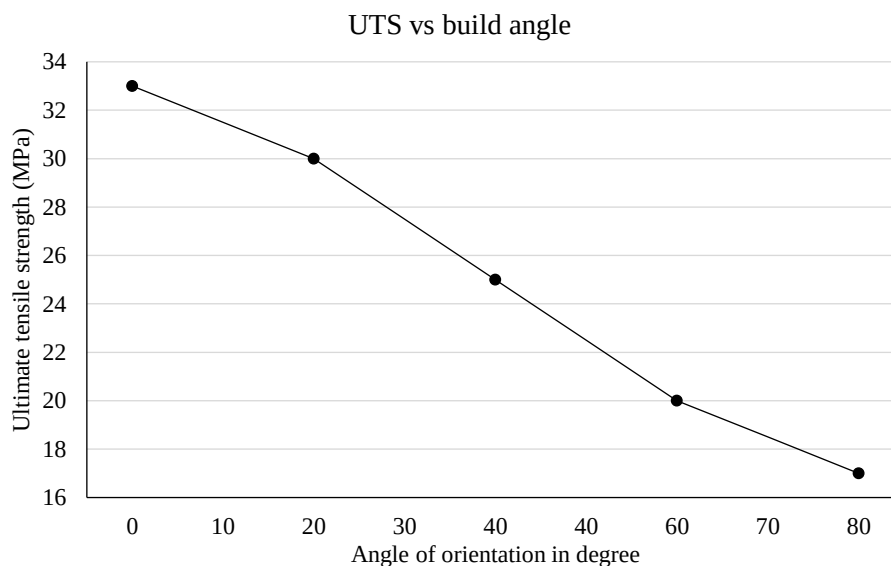
Table 3. SEM parameters.

Parameter	Value
Scintillator	Si (Li)
Cathode	LaB ₆ (Lanthanum Hexaboride)
Detector	EDS Si (30 mm ² active area)

Analysis Software	INCA 4.11
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Table 4. Tensile test setup.

Parameter	Value	Units
Preload	—	MPa
Load cell	—	KN
Initial grip distance	115	mm
Clamp type	Manual	—
Initial test speed	1	mm/min
Final test speed	50	mm/min
Test temperature	23	°C

**Figure 3.** Illustration of the tensile setup**Figure 4.** Ultimate tensile strength vs angle of orientation curves.

Ultimate Tensile Strength (UTS) vs. Build Angle

The setup for the tensile test, as depicted in Figure 3, demonstrates the arrangement and components used to measure the mechanical strength and elasticity of the material. Tensile strength and elongation at break (EAB) were the primary metrics evaluated.

Figure 4 shows the UTS values decreased nearly linearly as the angle of orientation increased. Specimens built at 0° exhibited the highest UTS, indicating optimal layer adhesion and minimal

anisotropy. This confirms that the 0° orientation is ideal for mechanical performance under the given process settings.

Elongation at Break vs. Build Angle

Figure 5 illustrates Elongation at Break (EAB) also decreased with increasing build angle. Specimens printed at 0° retained higher ductility, while those printed at steeper angles exhibited brittle behavior. This trend reinforces the selection of 0° orientation for all subsequent mechanical testing.

These results emphasize the critical role of part orientation in HSS, with optimized orientation enhancing performance consistency and process reliability.

Experiments on Different Mixing Ratios of PA12 – Iterations

After implementing a sustainable job box design, approximately equal quantities of R2 and R3 powders were generated. Based on this observation, a 50:50 mixing ratio was selected for evaluation. Several build iterations using this mixed powder were conducted to analyze powder ageing and degradation. Table 5 presents the quantities of recycled PA12 powder—categorized as R2 and R3—collected after each High-Speed Sintering (HSS) print job. This data is crucial for understanding powder yield and planning sustainable reuse strategies.

Powder Analysis

Sem Measurements

SEM images of powders from various iterations are taken, the size, shape and the agglomeration growth are analyzed

Figure 6 displays virgin PA 12 Powder, where the particles are consistently shaped and spherical. The structure suggests excellent flowability, low agglomeration and optimal morphology for effective sintering processes

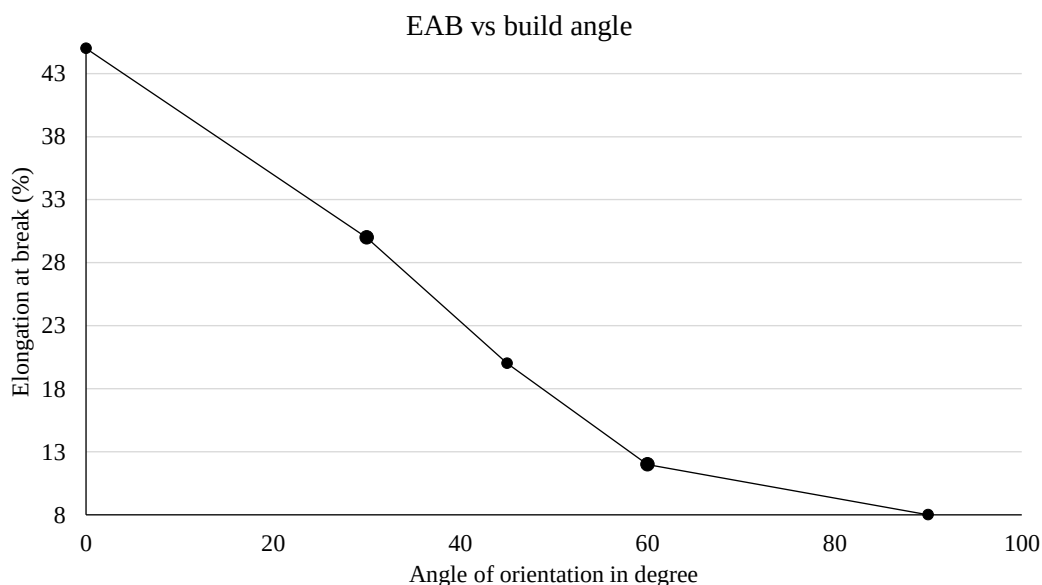


Figure 5. Ultimate tensile strength vs angle of orientation curves.

Table 5. Amount of recycled powder obtained in each job.

R2 Powder (Kg)	R3 Powder (Kg)	Print Number
2.52	2.49	1
1.85	1.85	2
3.41	3.34	3

2.44	2.40	4
1.50	1.42	5

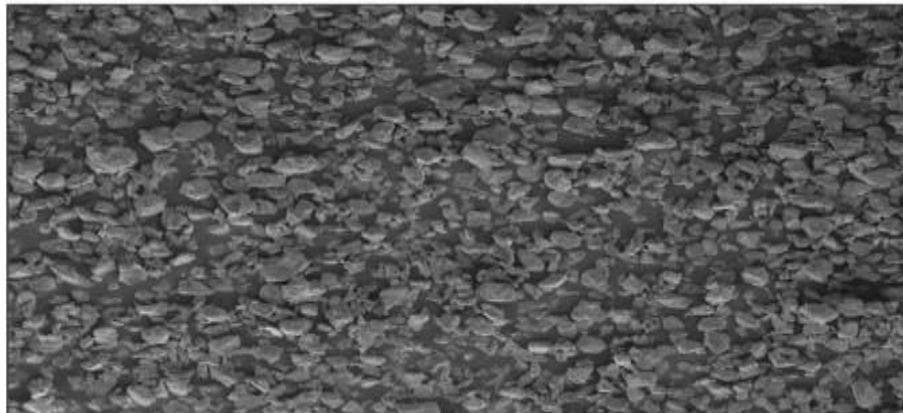


Figure 6. NN powder SEM image.

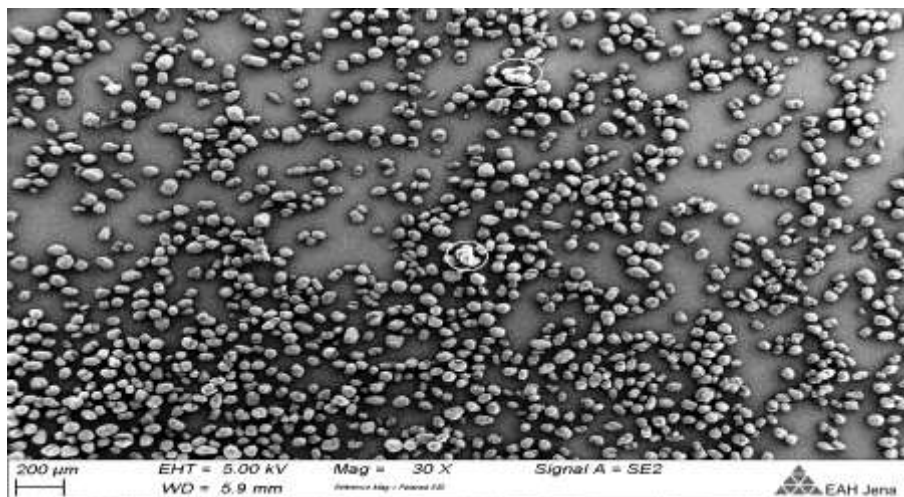


Figure 7. Iteration 1 SEM image.

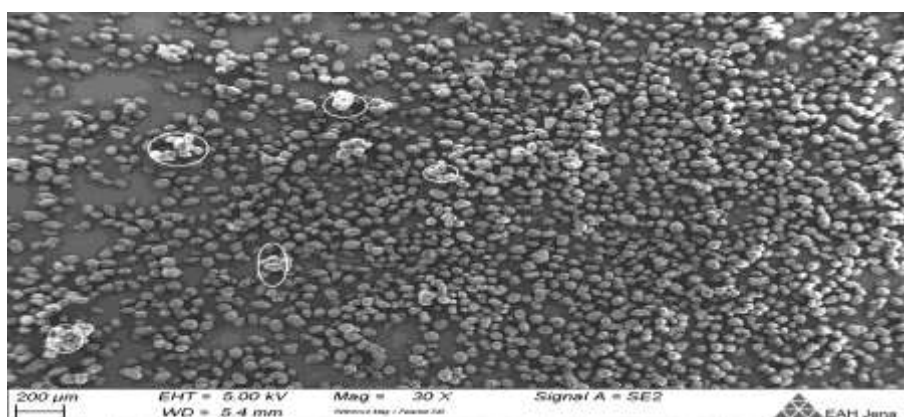


Figure 8. Iteration 2 SEM image.

Figure 7 indicates the powder after one HSS cycle (R2/R3 mixed). The particles are mostly spherical, but slight clustering begins to appear. It suggests early signs of thermal ageing due to partial melting.

Figure 9 shows the measurement of individual particles where the particle size slightly larger than the virgin powder larger grains can lead to uneven sintering, porosity and mechanical inconsistency.

The images in Figure 8 clearly show that agglomerates formed in the powder after two processing cycles, resulting from repeated thermal exposure.

Figure 10 illustrates the powder after three HSS Cycles, mixed R2 and R3. The number of agglomerates increased with each iteration, indicating progressive changes in powder characteristics, as seen in the SEM images. The bulk density of the powder reduces as the number of agglomerates increases. Figure 11 highlights morphological degradation due to repeated thermal exposure.

TGA is commonly used in AM to determine the upper processing and test temperature limit of polymer compounds. It is also employed in this study to assess any potential variations in temperature distribution induced by earlier exposure of the used powder to high temperatures and the detailing agent during HSS processing. TGA findings for all three PA12 powder and recycled powders

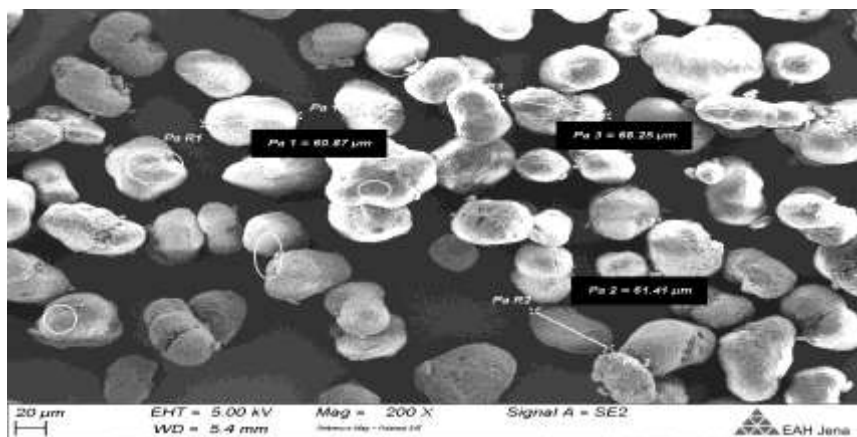


Figure 9. Grain size in iteration 2.

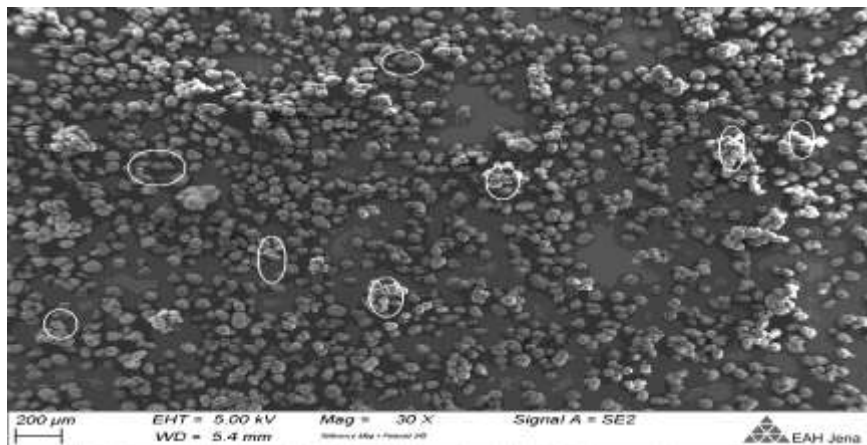


Figure 10. SEM image in 3rd iteration.

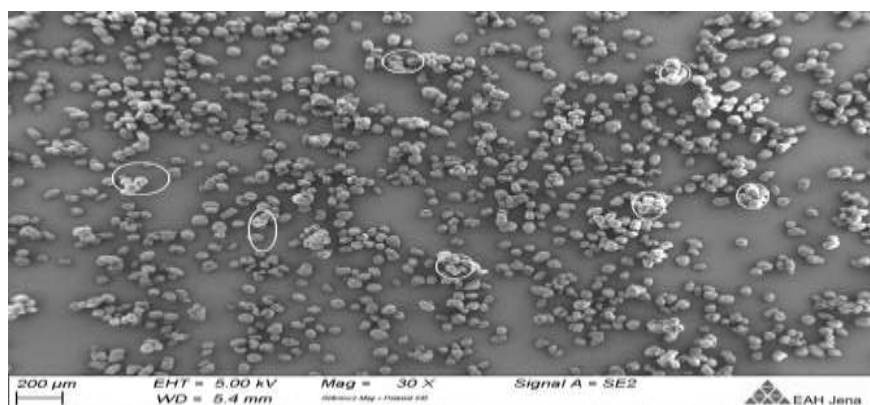


Figure 11. SEM image in iteration 4.

A 5% weight loss for the new PA12 powder began at 394 °C, whereas for the two recycled powders it began at 395 °C. There was no weight change seen at 100 C, showing that the powders had not absorbed large amounts of moisture even though no drying step was done prior to testing. At temperatures as low as 40 degrees Celsius, the detailing agent lost 5% of its weight and continued to lose weight rapidly until it reached 100 degrees Celsius. This behavior was expected, since water is one of the main components of the detailing agent. The detailing agent's weight decreased rapidly till it reached 191 degrees Celsius at 160 degrees Celsius. The detailing agent continued to lose weight between 191 C and 350 C.

According to Figure 12, all three PA12 powder ratios were thermally stable at the processing temperature. Phenomena such as thermo-oxidative degradation, post-polymerization, and crosslinking may have still taken place upon exposure to increased temperatures

DSC is used to track endothermic and exothermic activities during heating and cooling. A polymer must have a narrow melting range and a broad range between melting and crystallization. Figure 13 explains that the endothermic peak at 169.5°C is slightly lower than virgin PA12 (typically ~172–180°C) and the exothermic peak at 159.2°C shows where the polymer begins to reorganize during cooling.

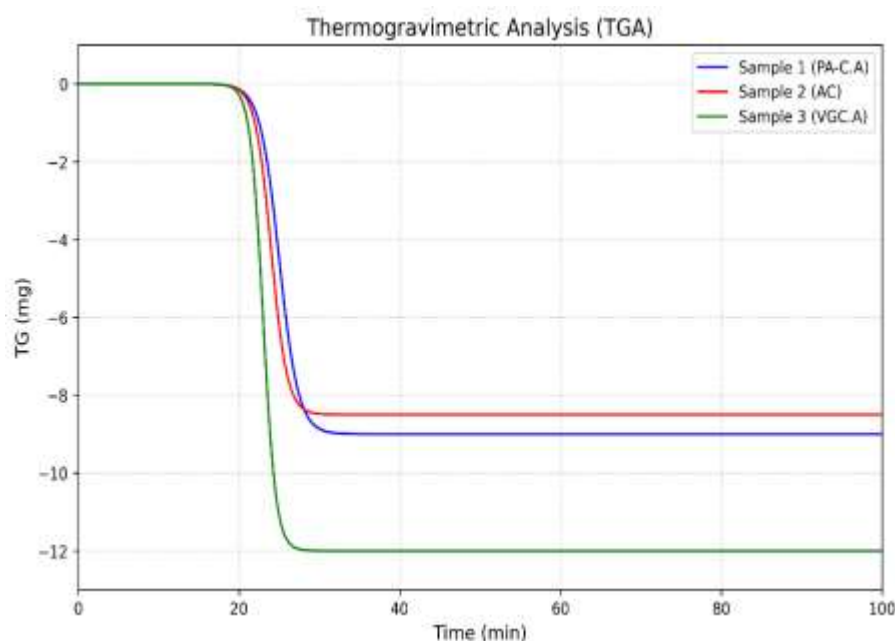


Figure 12. TGA analysis of virgin and recycled powders.

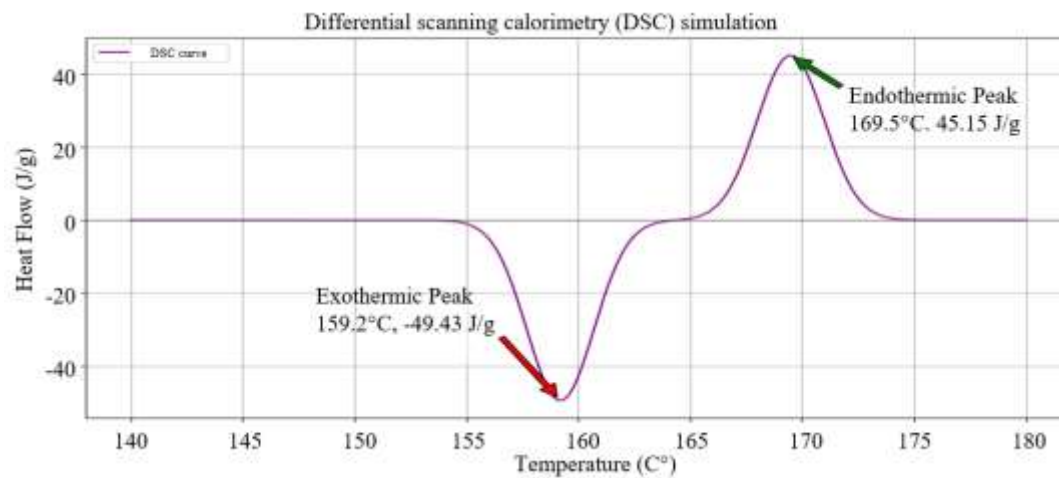


Figure 13. 1st iteration results.

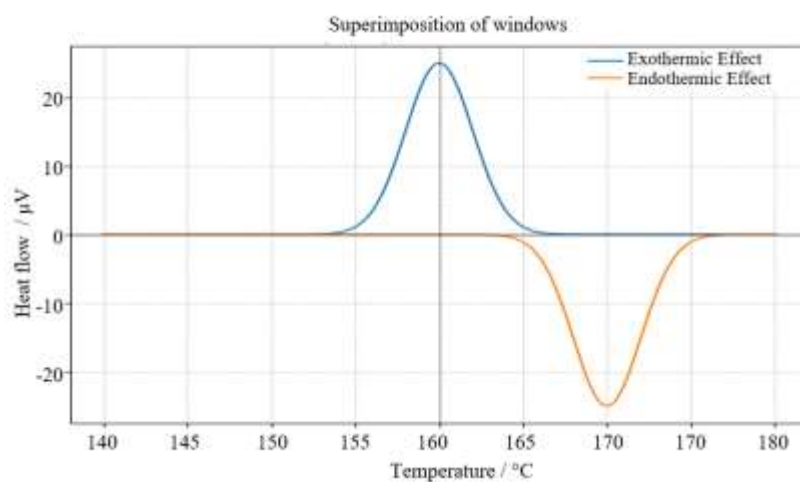


Figure 14. 2nd iteration results.

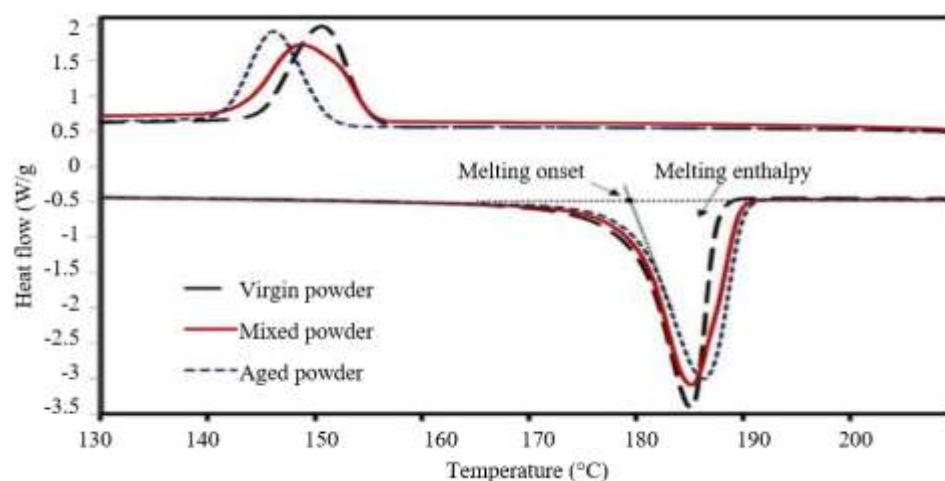


Figure 15. Integration of DSC results of all the powders

Figure 14 shows that the melting peak is slightly shifted and broadened compared to the first iteration as shown in Figure 13.

Figure 15 represents the DSC results of virgin powder, mixed powder and aged powder, where the R3 powder retains better thermal behaviour than R2, making it more suitable for reuse.

Taking semi-crystalline polymer crystallization and melting into consideration can help in the identification of new polymers that can be treated by AM. DSC is used to track endothermic and exothermic activities during heating and cooling as shown in Figure 16.

Mechanical Behavior of Parts Produced in Different Iterations

Table 6 summarizes the mechanical testing outcomes across different build iterations using recycled PA12 powder. It tracks how many tensile experiments were conducted, how many failed, and the corresponding job name. As the number of reuse cycles increases, agglomeration, reduced bulk density, and poor layer adhesion lead to higher failure rates. Iteration 3's success points to a potential threshold for safe reuse.

EAB and Tensile Strength Results With Different Mixing Ratios of PA12

Ten samples were utilized for the tensile testing. Before testing, all of the samples were thoroughly dried. To ensure the reliability of the mechanical property comparison throughout all iterations, all samples required to be in the same moisture content state. Because of the elastic wave repulsion caused by poor damping, several samples fractured into numerous pieces during testing. The thickness and breadth of the tensile samples were seen to shrink steadily as the number of repetitions increased.

Figures 17 to 19 represents stress-strain curves and tensile strength comparisons for PA12 parts produced using virgin and recycled powders across different iterations. In the Figure 18, the curves show brittle behaviour, with early fracture. Figure 19 shows the stress strain curves of the specimens printed using R3 powder where the curves represent the modern ductility and better strength retention.

A comparative analysis of Ultimate Tensile Strength (UTS) across 10 samples for three different PA12 powder conditions: virgin (NN), R2, and R3 represented by Figure 20. It's a key figure for quantifying how powder ageing affects mechanical performance in High-Speed Sintering (HSS). R3 powder can be reused with minimal impact on mechanical strength, while R2 requires careful refresh or blending.

Table 6. Amount of recycled powder obtained in each job.

Job Name	No. of Experiments	Failure of Jobs
Iterations 1	5	2
Iterations 2	4	2
Iterations 3	1	0

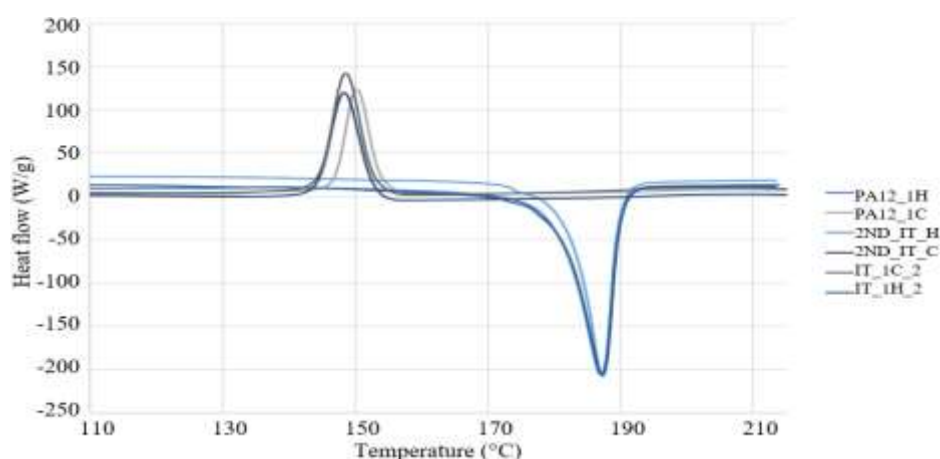


Figure 16. DSC curves of virgin, R2, R3 powders.

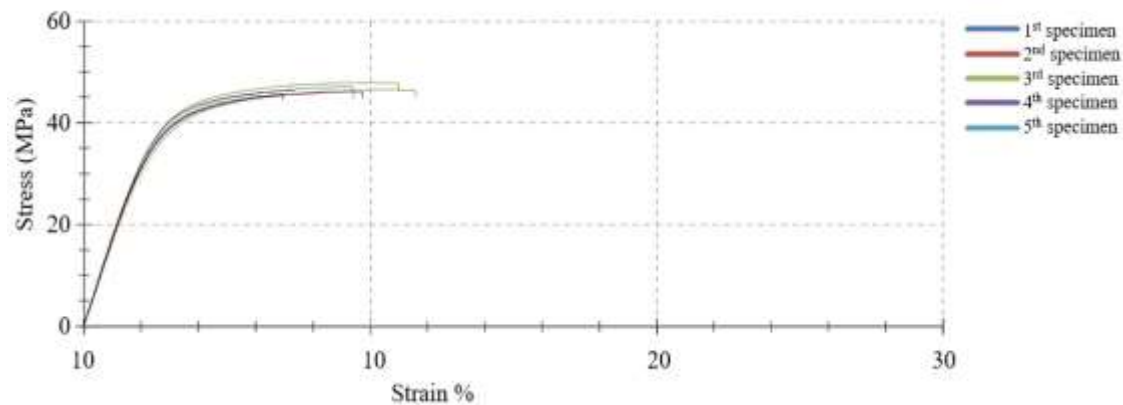


Figure 17. NN stress strain curves.

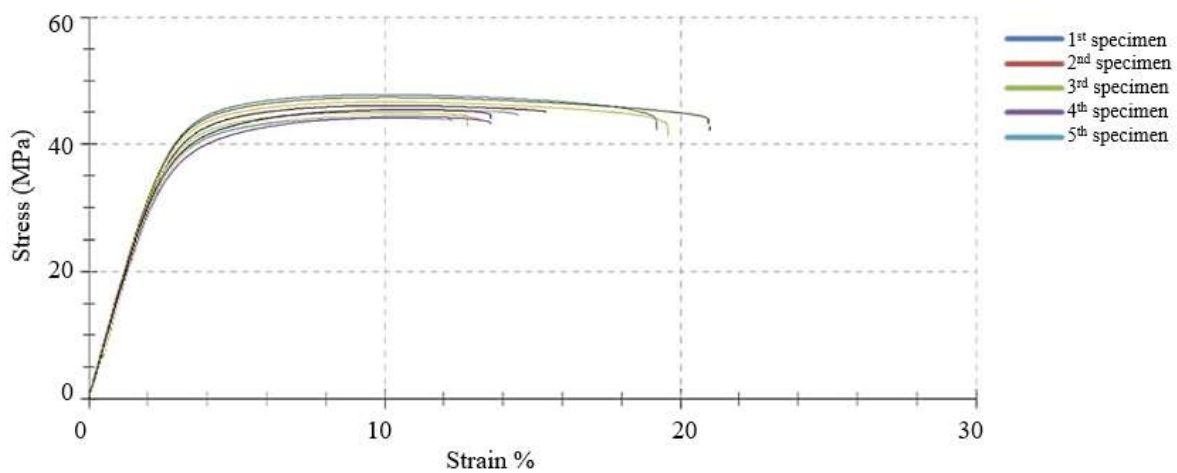


Figure 18. R2 stress strain curves.

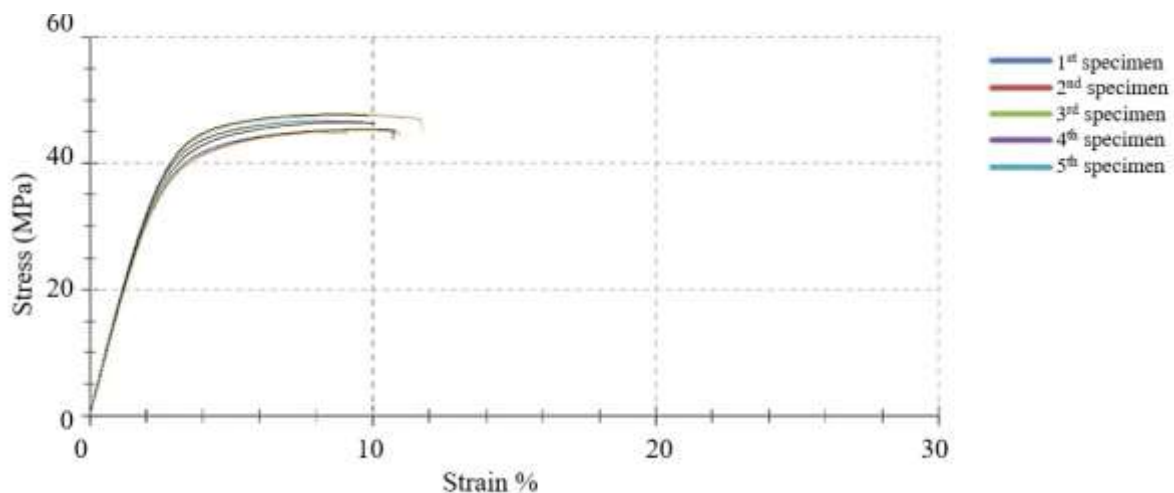


Figure 19. R3 stress strain curves.

The density of the powder clearly decreased with ageing due to the development of agglomerates. Because of the uneven and fairly large size of the agglomerated particles, the agglomeration impedes the sintering process and correct adhesion between the layers in the 3D construct. This powder change

causes a loss in surface quality, changes in surface structure and geometry, and an increase in the quantity of voids between the layers, resulting in deteriorating mechanical characteristics.

The curves in Figure 21 represents mixed recycled powder can still produce usable parts, but with reduced mechanical reliability.

Figure 22 represents the stress vs. elongation curves for specimens printed using a 50:50 mixed ratio of R2 and R3 powders during the second iteration. It demonstrates that even with balanced R2:R3 mix, mechanical reliability decreases in the Second reuse cycle. Figure 23 presents the stress vs. elongation curves for PA12 specimens printed using a 50:50 mix of R2 and R3 powders during the third iteration

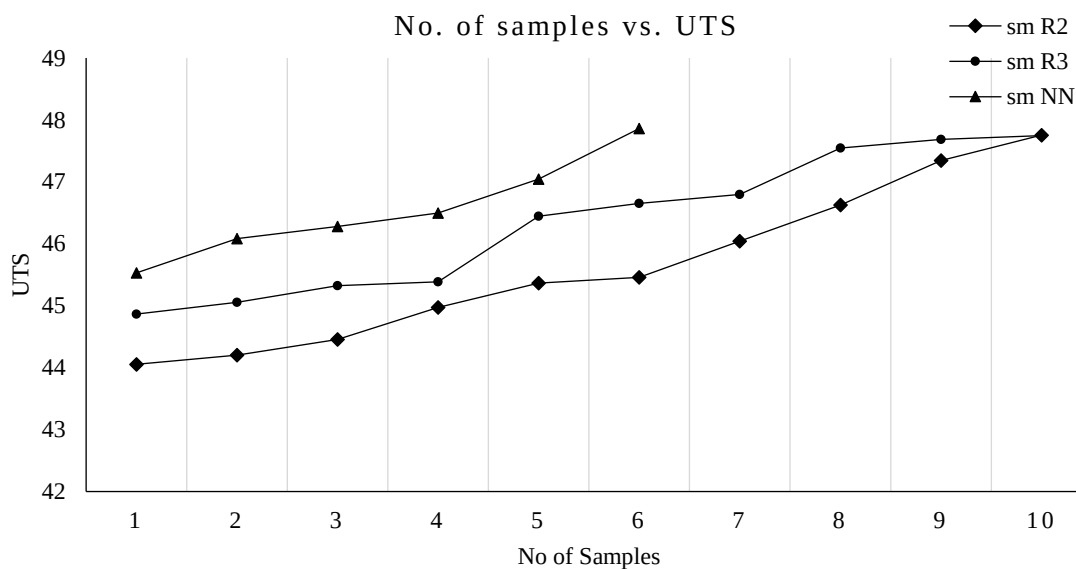


Figure 20. No. of samples Vs. UTS.

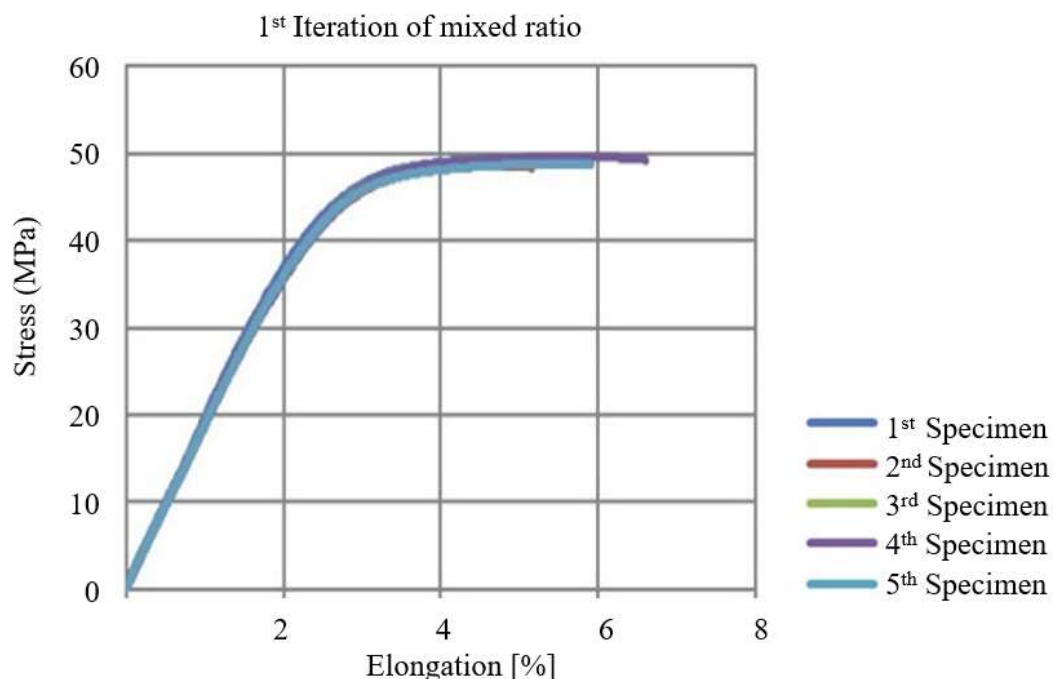


Figure 21. First iteration of mixed ratio.

The most notable conclusions from these graphs are that all mechanical qualities eventually decrease as the number of iterations increases. The fundamental explanation for this is decreasing consolidation because of the ageing impact.

The UTS values of virgin and R3 recycled powders were approximately equal, around 47 MPa, there is deviation of UTS of R2 from the virgin and new powder, so this variation is considerable effect and further research, this variation is due to the range of thermal exposure to the respective powders represented in Figure 24. Ultimate tensile strength variation in the Iterations follows as expected there is the step wide decrement in the tensile properties of the parts built in the iterations represented by Figure 25. In the 1st and 2nd there is not a such deviation but as the recycling powder usage continues there is drastic change in the 3rd iteration, hopefully this decrement will continue further if the powder is processed in more iterations.

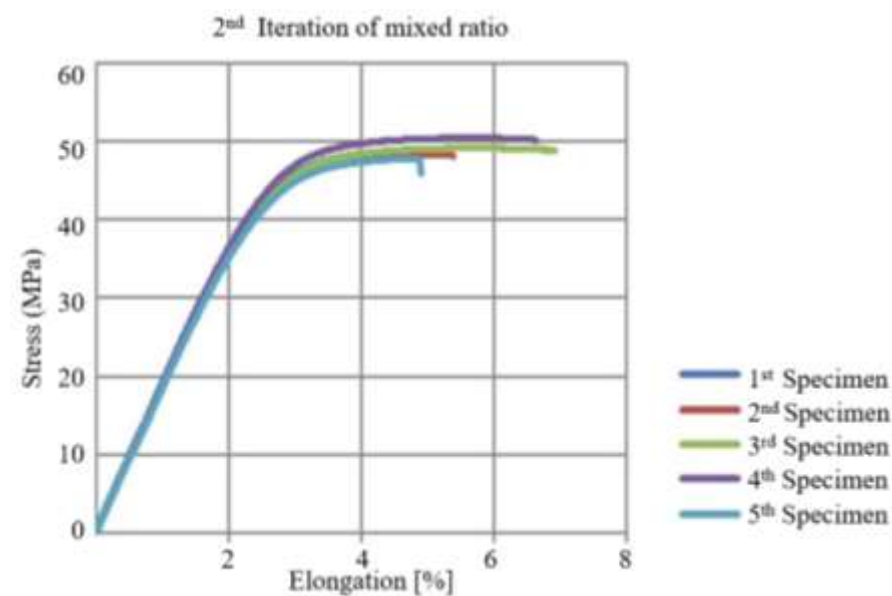


Figure 22. Second iteration of mixed ratio.

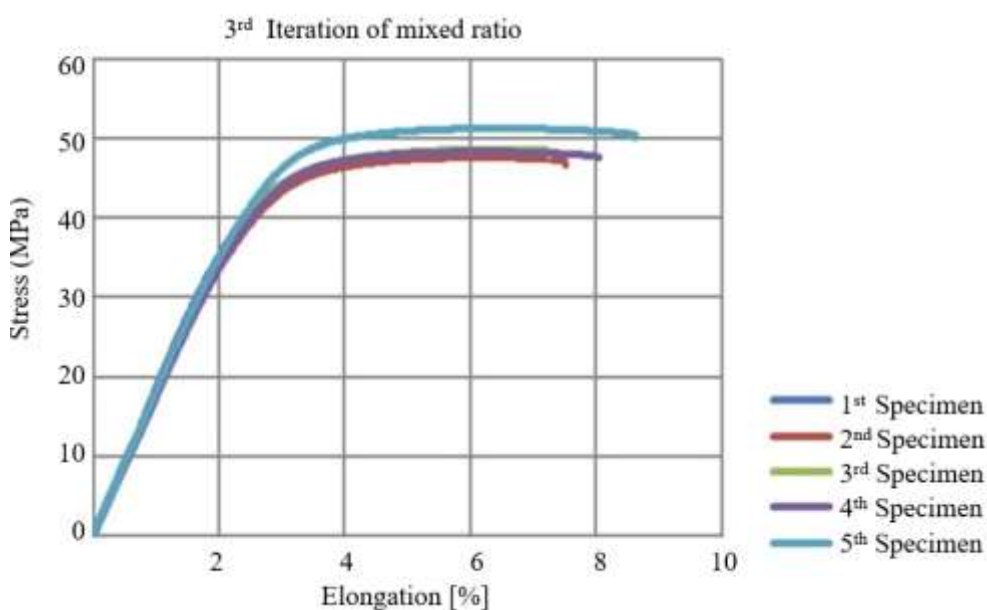


Figure 23. Third iteration of mixed ratio.

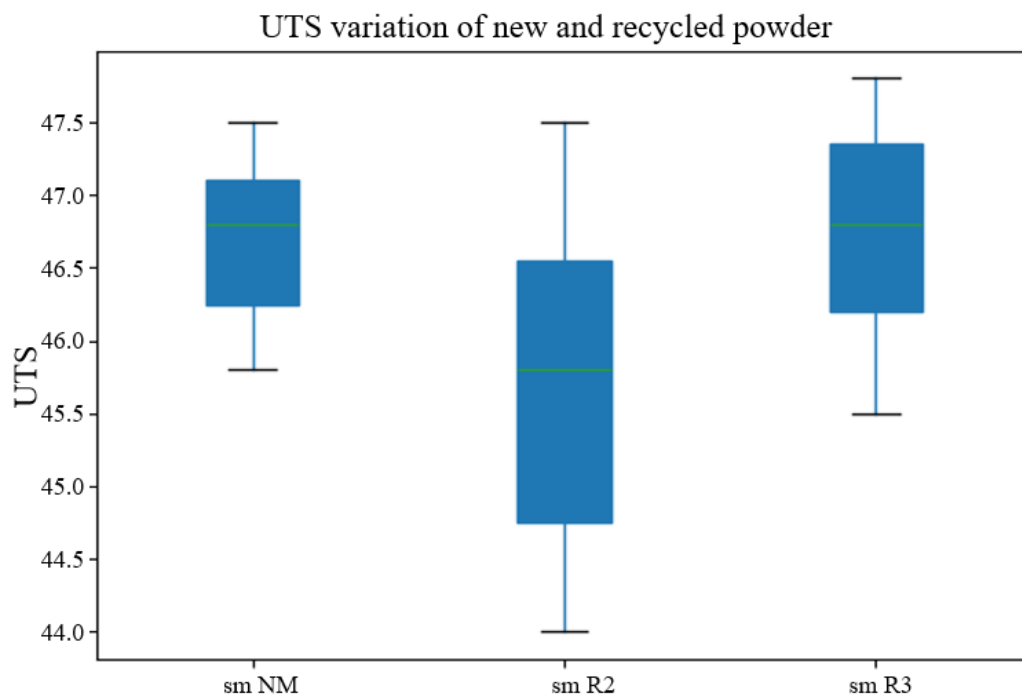


Figure 24. UTS variation of new and recycled powder.

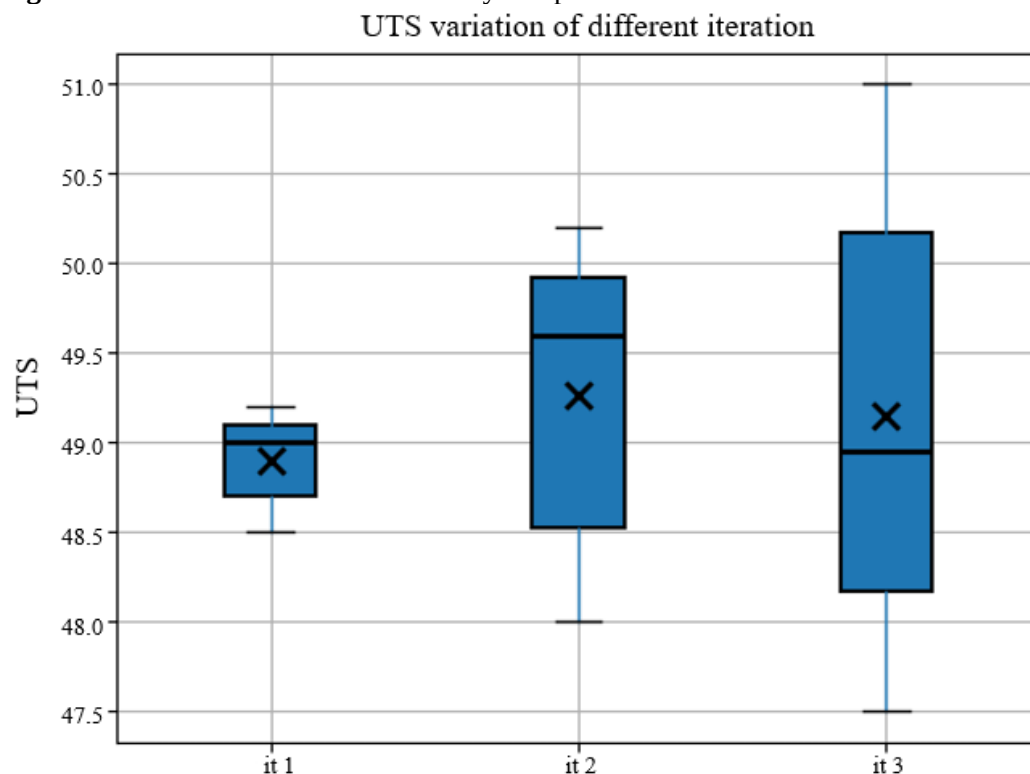


Figure 25. UTS variation in Iterations.

Figure 26 explains the elongation at the break also shows the same fashion of the ultimate tensile strength results, the results of virgin powder and R3 powder seems to be almost similar, but there is the deviation of results of R2 powder from these both that means the powder characteristics of virgin powder and R3 powder which is coming after the powder coating on the powder bed are almost similar. Figure 27 illustrates the mechanical performance analysis of PA12 parts across recycling iterations

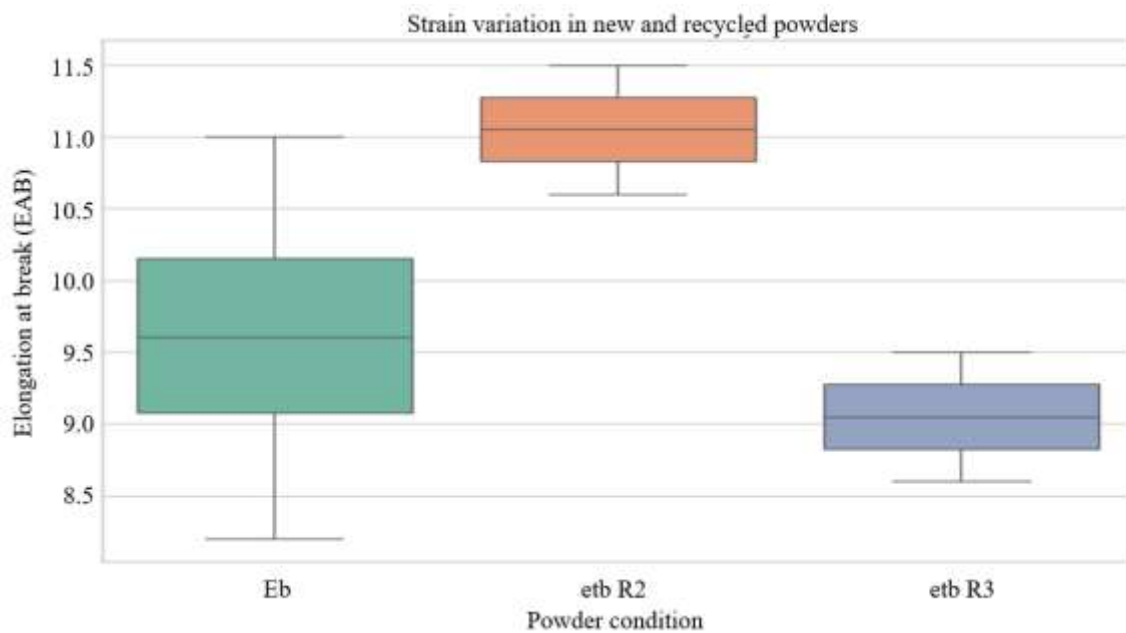


Figure 26. Strain variation in new and recycled powders.

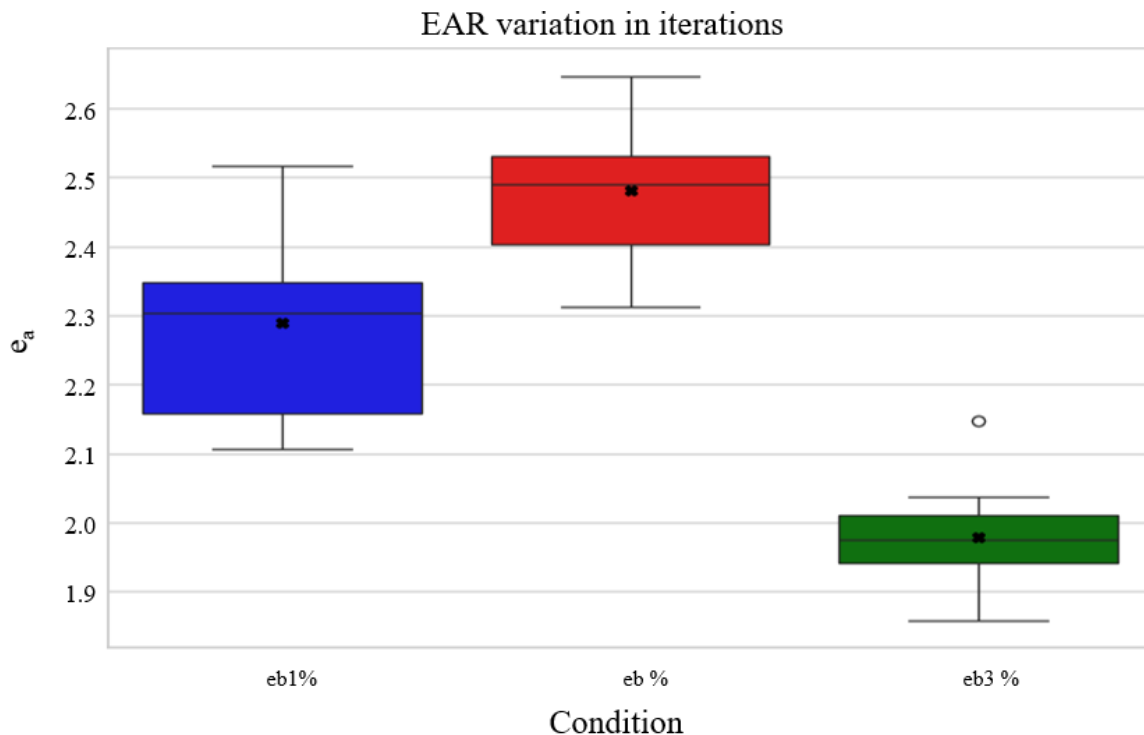


Figure 27. EAB variation in iterations.

CONCLUSION

When elongation at break is evaluated, the results reveal an intriguing trend: its variation is inversely related to that of the ultimate tensile strength (UTS). As previously discussed, there is minimal deviation in the first and second recycling iterations. However, by the third iteration, a significant shift is observed. The sharp decline in elongation at break, coupled with substantial variability, indicates that the material's mechanical integrity degrades beyond an acceptable threshold. This suggests that a critical limit must be defined for the number of recycling cycles, beyond which the mechanical performance of the powder becomes unreliable.

The High-Speed Sintering (HSS) process is strongly influenced by powder ageing, primarily due to particle agglomeration and thermal degradation, as discussed in the Differential Scanning Calorimetry (DSC) analysis. With each additional recycling iteration, the energy required for sintering increases, indicating a decline in powder reactivity. Consequently, maintaining component quality necessitates ongoing adjustments to processing parameters. However, in this study, the parameters were deliberately held constant to ensure that variations in the results could be attributed solely to the powder condition rather than process adjustments. While this approach supports experimental consistency, it also emphasizes the significant impact of powder ageing on processability. Effective handling of such variations requires skilled operators and may impose practical limitations on the extent of powder reuse.

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