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ORIGINAL ARTICLE

Direct metal laser sintering of Ti-6Al-4V parts with reused powder

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Abstract
Ti-6Al-4V alloy is characterised by having excellent mechanical properties and corrosion resistance combined with low specific weight and biocompatibility. This material is ideal for many high-performance engineering applications. It is increasingly used in additive manufacturing (AM) thanks to the possibility of producing very complex lightweight structures, often not achievable with conventional manufacturing techniques, as well as to easily customise products according to specific customer requirements. In powder bed fusion (PBF) processes, only a small percentage of the powder is actually melted and solidified to achieve the final part while most is left after the build. Since the surface morphology and chemistry, the shape and size distribution of the un-melted particles are inevitably modified during the process, and this may affect the resulting properties of the final products, many companies tend to use virgin powders for AM builds to keep compliance with manufacturing requirements and minimise risk. From both an economic and environmental point of view, it results crucial to develop recycling methods to reuse the metal powder as many times as possible while maintaining compliance with manufacturing standards. In this work, the effect of Ti-6Al-4V powder reuse on the evolution of powder characteristics and mechanical properties of final products additively manufactured is investigated through a systematic approach based on design of experiments.

Keywords Additive manufacturing (AM) · Powder bed fusion (PBF) · Powder reuse · Ti-6Al-4V · Design of experiments (DOE)

1 Introduction

Although early use of additive manufacturing in the form of rapid prototyping was almost limited to the production of visualisation models, it is nowadays being used to fabricate end-use products in many different fields, spreading from aerospace and automotive, to the production of biomedical implants and biological tissues, and even fashion goods. AM allows not only to reduce the delivery time and total cost of complex components, but also to enhance the performance, weight and functionality of the components themselves [1, 2].

Direct metal laser sintering (DMLS) is based on the laser powder bed fusion technique and uses a Yb (Ytterbium) fiber

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laser to locally melt a powdered metal to build up highly complex solid structures additively layer by layer [3–5].

Along with the well-known repeatability, reproducibility [6] and anisotropy [7, 8] issues, a drawback of all PBF processes is that only a small percentage of the powder is actually melted and solidified to achieve the final part while most is left after the build. However, the surface morphology and chemistry, the shape and size distribution of the un-melted particles are inevitably modified during the process, so that the resulting properties of the final products may be negatively affected [9–11]. Therefore, many companies tend to use virgin powders for AM builds to keep compliance with manufacturing requirements and minimise risk, thus resulting in a huge amount of *out-of-spec* powder, either stored or wasted. This policy contributes to higher costs and a larger environmental footprint of AM processes, so it results crucial to develop qualified methods to reuse the metal powder as many times as possible while maintaining compliance with manufacturing standards [12–14].

Both academic and industrial research works have faced this issue, focusing on the most widely used materials, from steels [15–26] and aluminium alloys [15, 27–32] to Inconel [29, 33–40] and titanium alloys [29, 33–35, 39, 41–55]. Generally speaking, some of the results obtained for the effects of metal powder reusing seem to be inconsistent across the studies. This is ascribable to the large number of side factors involved, such as the specific AM equipment or brand of powder used, the process parameters, the procedure for reuse, etc. [56].

This work addresses the effect of *EOS Ti64* (Ti-6Al-4V) powder reuse on the evolution of powder characteristics and mechanical properties of final products additively manufactured. A systematic approach based on design of experiments (DOE) and analysis of variance (ANOVA) was used to ensure effectiveness and reliability of the experimental results [57–61].

Table 1 One-way ANOVA results for the effect of number of reuses

Variable	Source	DOF	Adj SS	*f*-value	*p*-value
YS0.2%	Number of Reuses	9	17988	26.67	0.000
	Error	50	3747		
	Total	59			
UTS	Number of Reuses	9	12874.6	85.17	0.000
	Error	50	839.8		
	Total	59			
A	Number of Reuses	9	5.066	1.02	0.440
	Error	50	27.681		
	Total	59			

2 Materials and methods

2.1 Virgin powder and powder reusing method

All the specimens for mechanical characterisation were produced on an EOSINT M280 using the optimised processing parameters provided by the producer, the EOS Part Property Profile *Ti64 Performance 30* μm, and heat treated at 650 °C for 3 hours. Both DMLS process and heat treatment were performed in Argon inert atmosphere.

Different strategies can be used to recycle metal powder in PBF processes. In this work, the same procedure implemented by the authors in [16] was executed. It consists in producing a first DMLS build using virgin powder only, after which the un-melted powder left over in the build

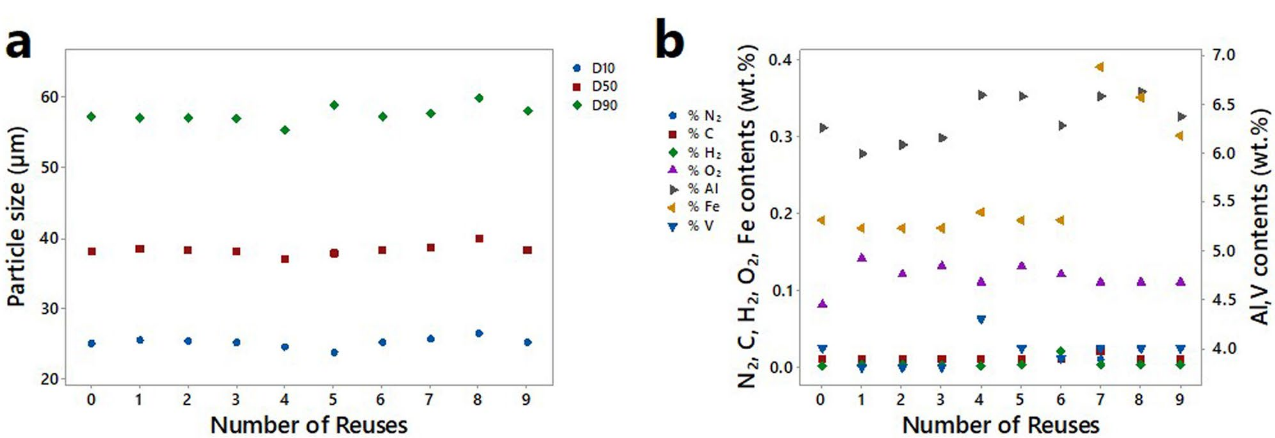

Fig. 1 Characteristics of Ti-6Al-4V powder over number of reuses: **a** *D*10, *D*50 and *D*90 of particle diameter, **b** Chemical composition

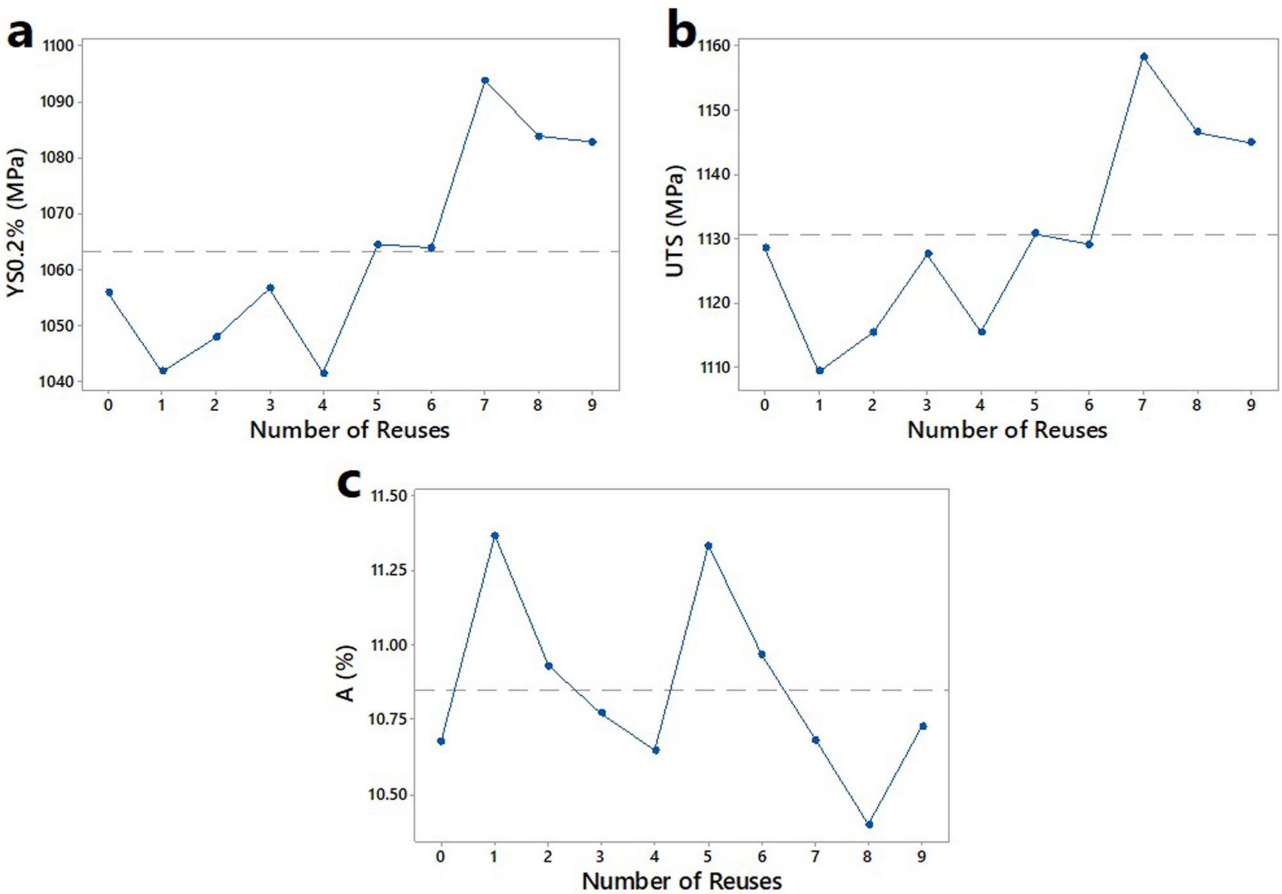

Fig. 2 Main Effects Plots for the effect of number of reuses on Ti-6Al-4V tensile properties: **a** yield strength, **b** ultimate tensile strength and **c** elongation at break

volume and the overflow compartment is collected, sieved and then loaded above the residual powder in the feeding compartment to start the subsequent run, and so on for each DMLS run up to the last one.

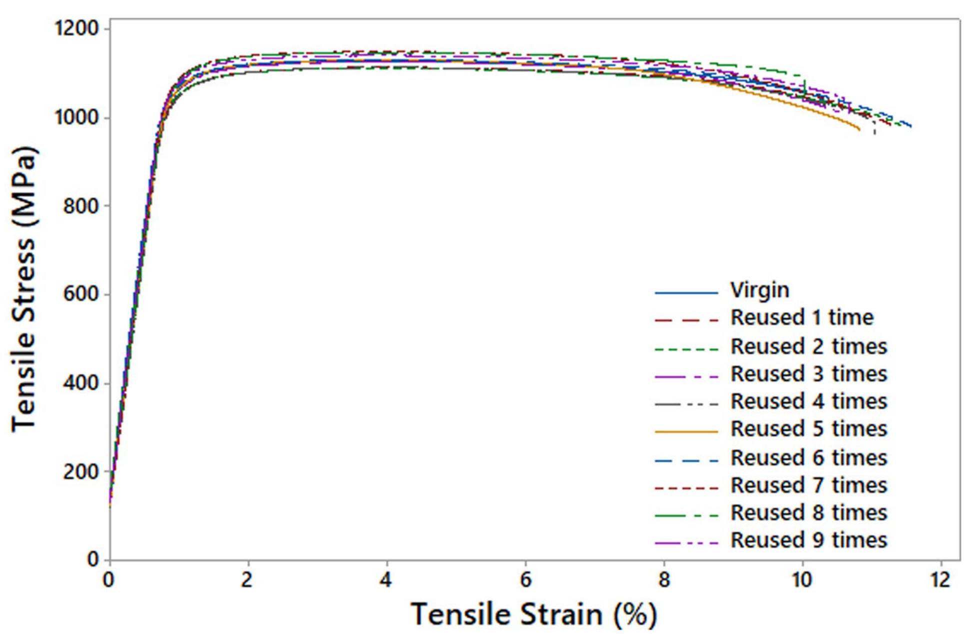

Fig. 3 Stress-strain curves of Ti-6Al-4V samples produced with virgin and reused powder

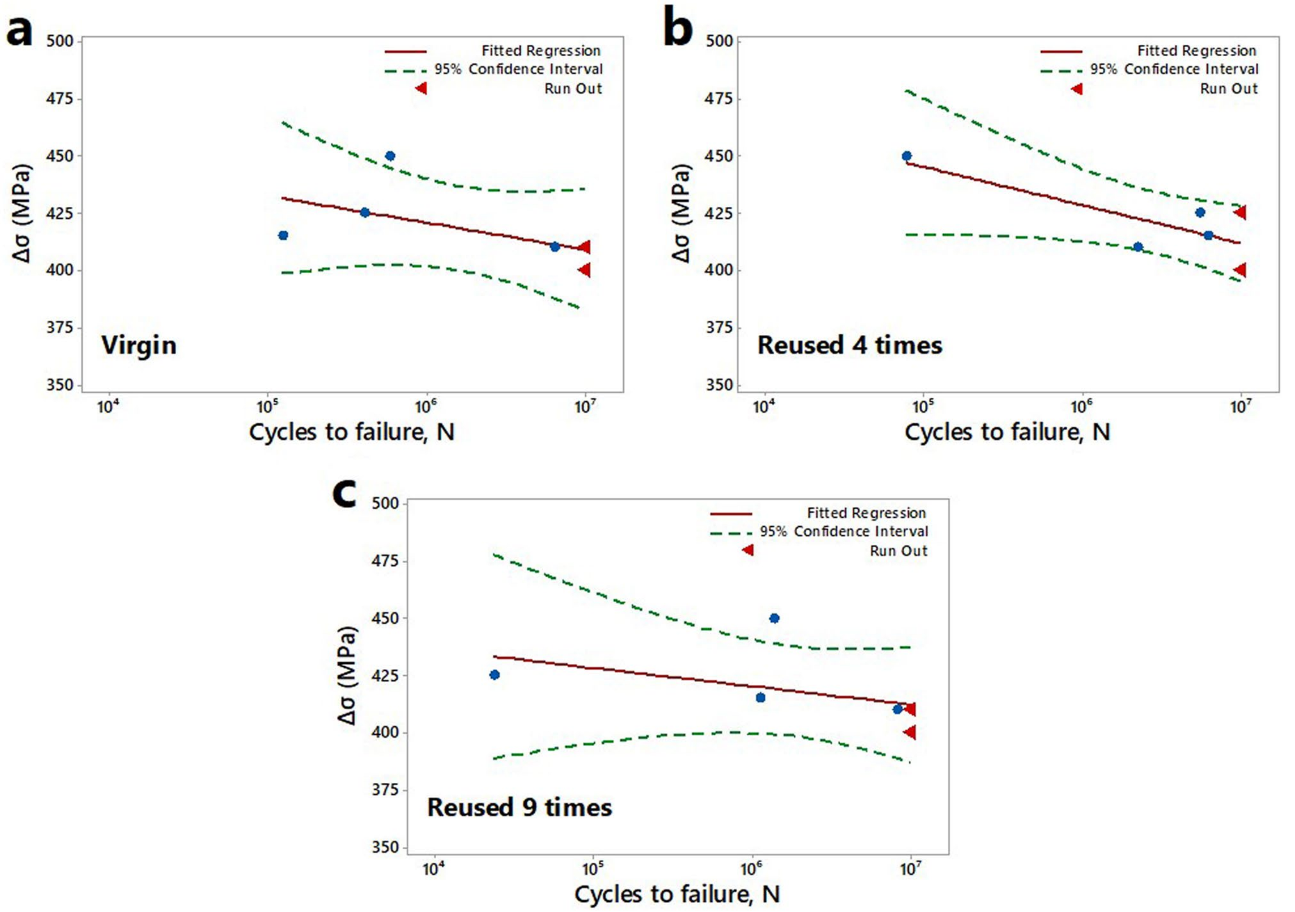

Fig. 4 Wohler curves of the Ti-6Al-4V HCF samples produced with virgin and reused powder: **a** virgin, **b** reused 4 times and **c** reused 9 times

2.2 Samples and testing standards for the evaluation of powder characteristics and mechanical properties

Before starting each DMLS run, powder samples were collected, in compliance with ASTM B215-15 [62], from the feeding compartment to undergo physical/chemical characterisation, as suggested in [63].

Particle size distribution (PSD) was determined according to ASTM B822-17 [64] using a Malvern MS2000 laser diffraction analyser, while powder chemical composition was evaluated through inductively coupled plasma, infrared absorption and inert gas fusion techniques (depending on the chemical elements to be detected) according to ASTM E2371-13 [65] and ASTM E1019-11 [66].

To evaluate the mechanical properties of parts additively manufactured with virgin and reused powder, ten subsequent DMLS builds were produced according the aforementioned procedure. Each build hosted six cylindrical bars to be used for the tensile tests and six near net shape samples for the high cycle fatigue (HCF) tests. Downstream of DMLS process, all specimens were machined to comply with ISO 6892-1:2016 [67] and DIN EN 6072:2011 [68] testing standards.

Tensile tests were executed at room temperature on an Instron 1185, with a cross head speed of 0.45 mm/min, according to ASTM E8/E8M-16a [69], while HCF tests were

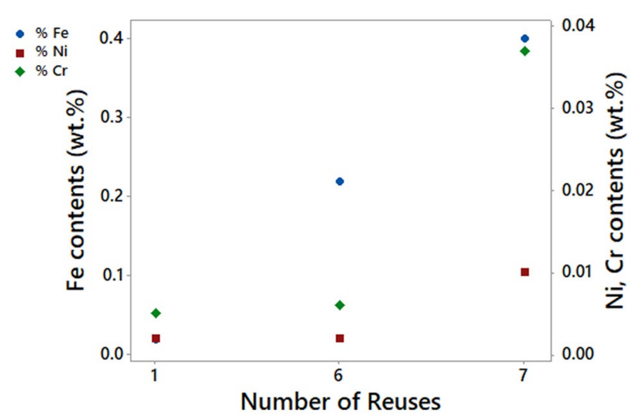

Fig. 5 Chemical composition of additively manufactured material belonging to DMLS builds produced with powder reused 1, 6 and 7 times

executed on a MTS Load Frame Model 312.21 with K_t=1 and R=0.1, according to ASTM E466-15 [70], and terminated at 10^7 cycles. The corresponding Wohler curves were obtained according to ASTM E739-10 [71].

3 Results and discussion

The 10th, 50th and 90th percentiles of the particle diameter (referred to as *D*10, *D*50 and *D*90, respectively) are

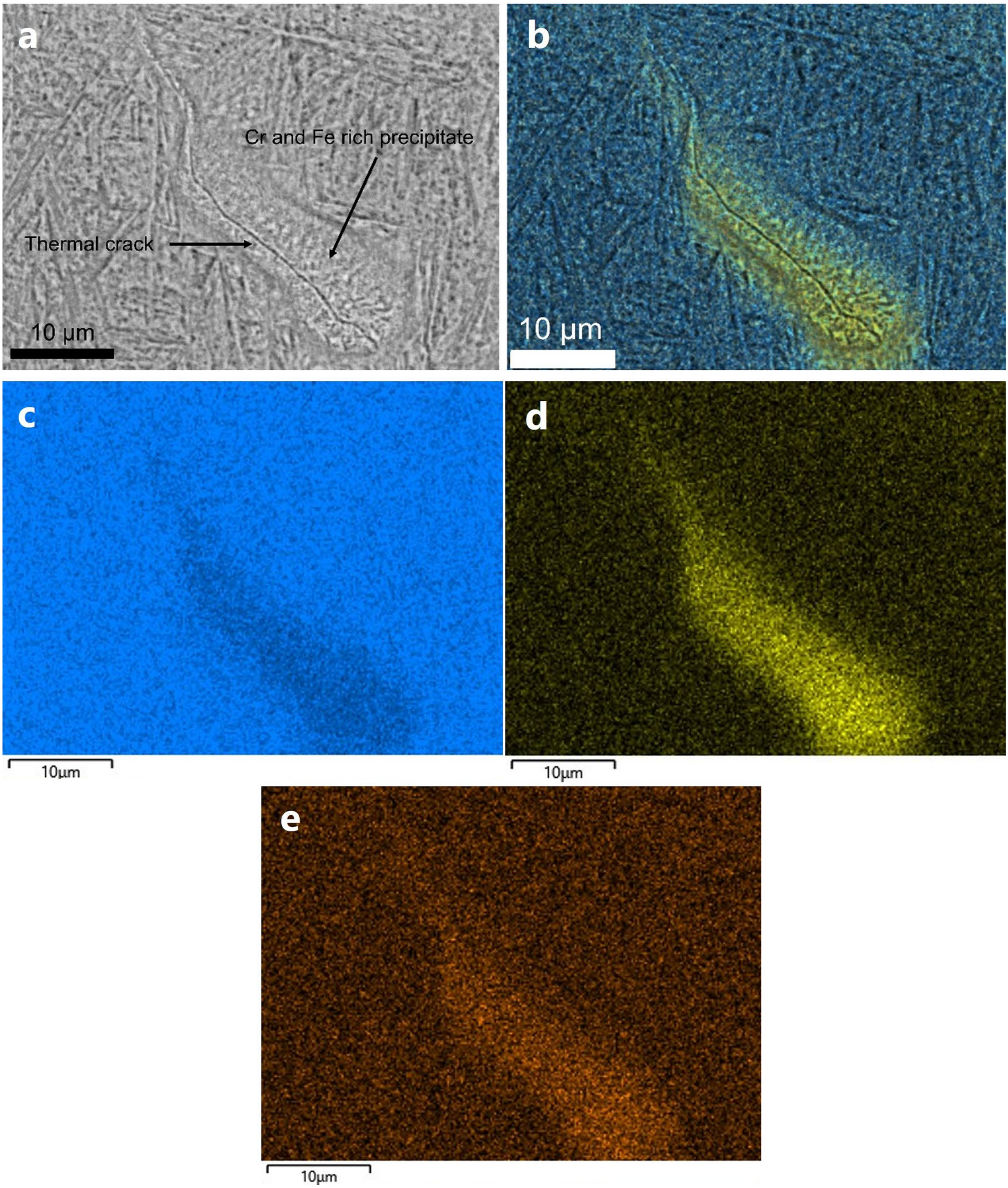

Fig. 6 SEM-EPMA analysis: **a** Fe- and Cr-rich precipitate in the Ti-6Al-4V matrix, **b** distribution of Cr (orange), Fe (yellow) and Ti (blue) in the precipitate, **c** distribution of Ti in the precipitate, **d** distribution of Fe in the precipitate, **e** distribution of Cr in the precipitate

reported in Fig. 1a, showing how the variability of particle size distribution over the number of reuses can be considered substantially physiological. Conversely, Fig. 1b points out an anomalous variation of chemical composition, in particular in terms of iron content, in the last reuse cycles.

The Minitab®18 software was used to perform one-way ANOVA, with a significance level $\alpha = 0.05$, on tensile properties, after diagnostic check of residuals. Once the ANOVA has been performed, the effect of a source of variability can be defined as statistically significant with respect to a particular response variable if the corresponding *p*-value results lower than α [57, 72].

The obtained results for yield strength (*YS0.2%*), ultimate tensile strength (*UTS*) and elongation at break (*A*) are reported in Table 1: the effect of powder resuse resulted to significantly affect yield strength and ultimate tensile strength (p-value $\ll \alpha$), but negligible with respect to elongation at break (p-value $\gg \alpha$).

From Fig. 2, that shows the trend of the mechanical properties over the number of reuses, it is possible to see how the effect of powder reuse is actually confined to the last three runs and how it involves a slight enhancement, rather than a decay, of material performance. However, it is worth noting that the differences highlighted by the ANOVA can be considered relatively small from a technological point of view. Indeed, the corresponding stress–strain curves resulted almost overlapable, as shown in Fig. 3. In addition, the measured values resulted all consistent with those guaranteed by the supplier and required by the company.

Figure 4 shows the Wohler curves of specimens produced with virgin and reused powder. The fatigue behaviour remained substantially stable, with minimum a high cycle fatigue strength ($\Delta\sigma$) of 400 MPa.

The abnormal mechanical properties of samples produced within the last DMLS runs are ascribable to the aforementioned change in powder chemical composition. The observed increase of iron content was found to be caused by a steel contamination [73] of powder during sieving operations: the sieve used in this work was made of AISI 316L steel, that is characterised by a much lower hardness than titanium, and the continuous rubbing of the powder against sieve walls had caused a kind of erosion effect, resulting in the inclusion of steel powder in the titanium one. This is confirmed by both the increase of Fe content in the powder samples analysed (see Fig. 1b) and by the chemical analysis of additively manufactured material. Indeed, Fig. 5 shows the evident increase of Fe, Cr, and Ni concentrations in the alloy for the last DMLS runs. It is worth considering that these three elements, in which the AISI 316L is particularly rich, belong to the class of β-eutectoids with a very low solubility in α-Ti [74–76]. When these elements exceed this level of solubility, they form $(Cr,Fe,Ni)_2Ti$ intermetallics [77], which can significantly modify the resulting mechanical properties of the alloy [78, 79]. Given that this is a contamination, it is not surprising that the content of these elements does not exhibit a real trend upstream and downstream of the seventh run (where the first contamination probably occurred) but rather appears as a random fluctuation. In fact, what is nevertheless evident is a substantial shift in Fe content downstream of the sixth run. The presence of these intermetallics was further highlighted by scanning electron microscope-mounted electron probe X-ray microanalysis (SEM-EPMA) and energy dispersive X-ray spectrometry (EDS). Figure 6a shows the presence of a thin precipitate inside the Ti-6Al-4V lamellar matrix. It is worth noting that a thin shrinkage crack is also evident within the precipitate: this crack does not extend to the surrounding metal matrix, which is further evidence of the embrittlement effect due to the presence of Cr and Fe. The colour maps presented in Fig. 6b-e further confirm the higher concentration of Cr and Fe in the precipitate compared to the Ti-6Al-4V matrix. Finally, in the EDS spectrum of the precipitate, presented in Fig. 7a, both Fe (peak at 6.404 keV) and Cr (peak at 5.415 keV) are clearly visible. Conversely, as shown in Fig. 7b,

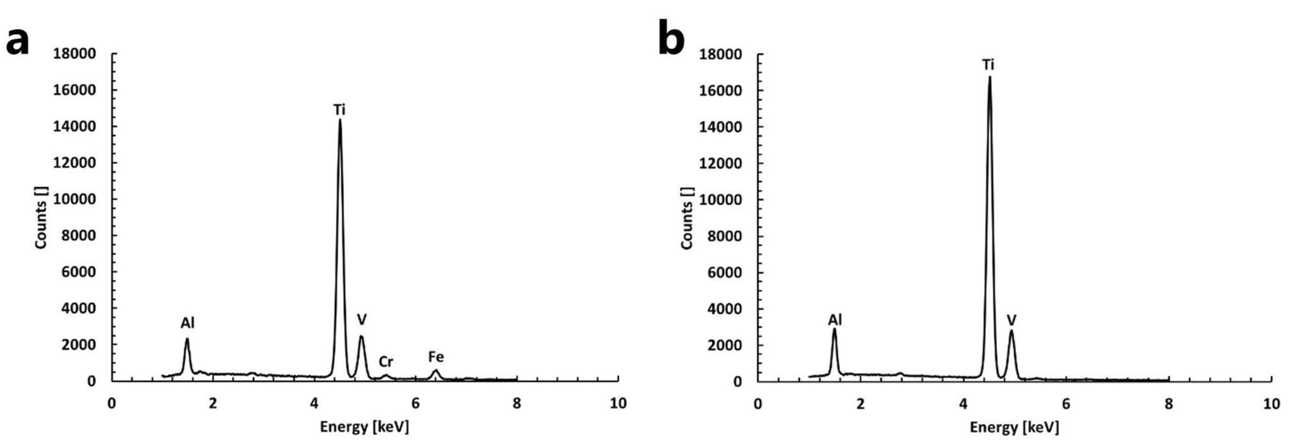

Fig. 7 EDS spectra: **a** Fe- and Cr-rich precipitate in the Ti-6Al-4V matrix, **b** Ti-6Al-4V matrix

neither of these two peaks is detectable in the EDS spectrum of the Ti-6Al-4V matrix.

4 Conclusions

Mechanical properties of titanium parts did not see a drastic variation due to powder reuse, which turned out to affect them only marginally, and in this specific case positively. The erosive effect of titanium powders on the walls of steel sieve, that caused the inclusion of steel powder in the titanium one, suggests the use of a sieve of the same material as the powder to be processed, or at least a harder one. In the specific case analysed in this work, the number of reuses was small enough not to show such detrimental changes in mechanical properties, but a higher number of reuses could have caused more significant, and probably negative, variation.

Obtained results confirm the reusability of AM powders, at least for a certain number of cycles, while maintaining compliance with manufacturing standards, contributing to the possibility for AM processes to affirm themselves as more affordable and environmentally friendly. On the other hand, these results also suggest the use of measures to minimise the undesirable effect of external contaminants, especially in the case of material that are very susceptible to contamination, such as titanium and titanium alloys.

The powder recycling strategy and the procedures for part quality assurance and data analysis proposed in this work can be adapted to other similar processes and other materials.

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Author contributions All authors contributed equally to this work.

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Data availability Not applicable.

Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

Consent for publication Provided by IMaST.

Conflict of interest The authors declare that they have no conflict of interest.

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