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Please cite this article as: M. Giovagnoli, G. Silvi, M. Merlin, M.T. Di Giovanni, Optimisation of process parameters for an additively manufactured AlSi10Mg alloy: limitations of the energy density-based approach on porosity and mechanical properties estimation, *Materials Science & Engineering A*, https://doi.org/10.1016/j.msea.2020.140613.

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1 Optimisation of process parameters for an additively manufactured

2 AlSi10Mg alloy: limitations of the energy density-based approach on porosity

3 and mechanical properties estimation

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8 Abstract

9 Due to the high complexity of the Additive Manufacturing processes, the effect of individual

10 deposition parameters on integrity and mechanical performance of the 3D-printed part is not

11 easily assessable. An energy density-based approach is often adopted to overcome such

12 complexity, being the Volumetric Energy Density (VED) one of the widely used parameters.

13 However, previous works on the AlSi10Mg alloy suggest that fully dense parts could be achieved

14 with a well defined VED range. This study investigates different combinations of deposition

15 parameters to reproduce part of this range and assess its effect on porosity, microstructure and

16 tensile properties of the AlSi10Mg alloy. It was found that different combinations of deposition

17 parameters in the employed VED range did not alter the microstructural features significantly.

18 Conversely, they severely affected porosity, showing that VED is not a reliable parameter to

19 predict the final density of the additively manufactured components. Moreover, specimens

20 produced with a similar VED but different hatch spacing exhibit significantly different tensile

21 properties. This fact suggests that the effect of deposition parameters should not be solely related

1   to the amount of energy imparted to the material. Finally, systematically higher tensile properties

2   characterise our specimens compared to results available in the literature, due to the

3   microstructure obtained with the adopted combination of deposition parameters. The energy

4   density-based approach lacks in considering the effect of process parameters on the

5   microstructural features other than porosity, which could identify their actual proper combination

6   misleadingly. Our results suggest that new approaches to define the optimum choice of process

7   parameters need to be evaluated to maximise the final performance of AM components.

8

9   Keywords: Additive manufacturing, AlSi10Mg, process parameters, VED, microstructure,

10   mechanical characterisation

11

12   1. Introduction

13   Among the manufacturing processes, Additive Manufacturing (AM) represents one of the most

14   attracting technologies of the last decade in both academic and industrial fields. The unlimited

15   flexibility in the design of complex-shaped components, the increasing need for optimised use of

16   raw materials and minimisation of machining operations and scraps, have led to an increasing

17   interest in such a topic. Currently, among the AM process, the Direct Metal Laser Solidification

18   (DMLS) laser powder bed fusion technology is one of the most successfully employed for metal

19   components production.

20   The Al alloys represent one of the most widely employed metallic materials in the AM

21   processes. They are particularly suitable for structural components which require a reasonable

22   compromise between mechanical performances and light-weighting as well as for heat-

23   controllable parts which need for high thermal conductivity, such as heat exchangers and heat

24   sinks. In light of this, Al alloys are considered very promising for aerospace and automotive

1   applications. Among all, the AlSi alloys offer the best processability, especially at Si contents near

2   to the eutectic composition. In recent years, the AlSi10Mg alloy has been extensively investigated

3   [1,2]. Thijs *et al*. [3] correlated the fine cellular microstructure with a preferred crystallographic

4   texture with the high mechanical response. Wu *et al*. [4] performed in-situ TEM compression tests

5   to elucidate the reasons for such strength. Maeshima *et al*. [5] proved the existence of a large

6   amount of Si retained in solid solution in the Al phase as additional contributions to the strength of

7   the alloy. Liu *et al*. [6] highlighted that the texture of the alloy could be modified through the

8   building parameters. Despite the broadening of Al alloys palette for the AM technologies is a

9   recent research topic [7–9], the AlSi10Mg alloy is still one of the most employed.

10   Although the strength of the Al alloys produced with the AM techniques, their mechanical

11   performances can be severely affected by the presence of process-related defects, with porosity

12   being the most deleterious one [10–12]. It descends from a non-optimised process, and hence a

13   proper choice of the building parameters is mandatory to achieve a near fully dense final

14   component. Even though the high number of parameters that take part at the overall AM process,

15   just a few of them are commonly investigated, namely: the laser power, the scan speed of the

16   energy source, the hatch spacing and the layer thickness. An energy density-based approach is

17   usually adopted in the literature to account for such process parameters, with the minimum

18   porosity achieved within a specific window of energy density. Inside the optimum window, an

19   almost entirely dense part is obtained, whereas too low or too high energy density leads to lack-of-

20   fusion and keyhole porosities, respectively [13–15]. Kempen *et al*. [16] optimised the deposition of

21   an AlSi10Mg alloy based on an energy per unit length parameter E' [J/mm], defined as the ratio of

22   laser power and scan speed. A superficial energy density E_d [J/mm^2] was employed by Krishnan *et*

23   *al*. [17] to minimise the porosity for the same alloy. However, a volumetric energy density (VED)

1 parameter is often used to define a proper process window [11,18–20], with the VED [J/mm³]

2 defined as equation (1):

$$VED = \frac{P}{v \cdot h \cdot t} \qquad (1)$$

3 where P [W] is the laser power, v [mm/s] is the scan speed, h [mm] is the hatch spacing, and t

4 [mm] is the layer thickness. Read *et al*. [20] found that ~60 J/mm³ of VED was suitable to achieve a

5 dense AlSi10Mg alloy. A similar optimum value was determined by Kimura *et al*. [19] processing

6 an AlSi7Mg alloy. Despite such agreement, data in the literature are somewhat scattered. Figure 1

7 depicts the optimum density obtained for several Al-Si alloys (Si ≥ 7%) and the correspondent VED

8 range or value [2,10,12,16–25]. For works not directly referring to the energy density, the VED was

9 calculated using the optimised building parameters accordingly to equation (1). VEDs in the range

10 30-110 J/mm³ were found to be suitable to achieve fully dense parts (relative density in the range

11 98-99.9%), with the majority of the data lying between 40 and 70 J/mm³. It is worth to notice that

12 the works reported in Fig. 1 majorly deal with the effect of the energy density on porosity of the

13 alloys, with little or no attention to the other microstructural features and their effect on the

14 resulting mechanical properties.

15 The present work starts from an actual industrial need to determine the proper combination of

16 process parameters for the AlSi10Mg alloy in light of the final tensile performance and based on

17 data available in the literature. It aims at verifying the reliability of the energy density-based

18 approach commonly adopted in the literature, with particular reference to the VED, in defining the

19 optimum process window of deposition parameters. Additionally, it seeks to assess the

20 reproducibility of tensile properties for specimens produced within a given energy density range.

21 Therefore, we investigated the effect of VED in the above-mentioned range (40-70 J/mm³) on

22 porosity, microstructure and mechanical performance of the AlSi10Mg alloy and to validate its

1 effectiveness as a proper design parameter. Our results highlight some severe limitations of such

2 an approach and elucidate the need for further research in this field.
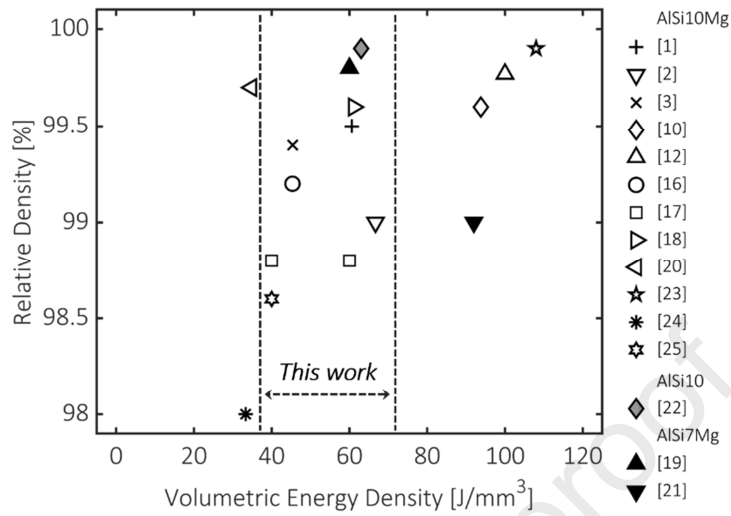

Figure 1. Comparison of literature data for different Al-Si alloys: VED ranges/values that achieved fully

dense components (relative density in the range 98-99.9%).

3

4 2. Experimental

5   2.1 Additively manufactured samples

6   The samples were produced with an EOS M290 system using the Direct Metal Laser

7 Solidification (DMLS) laser powder bed fusion technology. A commercially available AlSi10Mg

8 powder was processed in a protective argon atmosphere on a cold-building platform. All the

9 specimens were realised at constant laser power (P) and layer thickness (t). Different combinations

10 of scan speed (v) and hatch spacing (h) are reported in Table 1, together with the corresponding

11 VED. Absolute values of process parameters cannot be disclosed, as reported in other scientific

12 researches performed in collaboration with industry [26]. Hence, in Tab. 1 they are reported

13 normalised to the corresponding minimum value.

14

Table 1. Combinations of building parameters - scan speed and hatch spacing - and corresponding VED

v [mm/s]	v	$1.18 \cdot v$	$1.36 \cdot v$	$1.55 \cdot v$	v	$1.18 \cdot v$	$1.36 \cdot v$	$1.55 \cdot v$
h [mm]	h	h	h	h	$1.12 \cdot h$	$1.12 \cdot h$	$1.12 \cdot h$	$1.12 \cdot h$
VED [J/mm³]	66	56	48	42	59	50	43	38

1

2 For each condition, three cylindrical tensile specimens were produced vertically to the building

3 platform and then machined to the final size (diameter d_0 = 4 mm, gage length L_0 = 20 mm). An

4 additional 10 x 10 x 25 mm³ cuboid sample was realised with the long side parallel to the building

5 platform for each condition. Microstructural and mechanical characterisations were performed on

6 specimens in the as-built state.

7

8 2.2 Tensile tests, Brinell hardness and density measurements

9 Tensile tests were performed using an Instron 5982 testing machine, according to the ISO 6892-

10 1: 2016 standard. Density measurements were conducted on the cuboid samples with the

11 hydrostatic weighing method using a METTLER AE240 analytical scale. Samples were firstly

12 weighed in air and then in distilled water. Absolute density values were obtained as the average of

13 five measurements. Relative densities were calculated from the absolute ones as the ratio with the

14 nominal alloy density, considered as 2.67 g/cm³ according to [27]. Brinell hardness tests were

15 carried out on the lateral sides of the cuboid samples. The measurements were conducted using an

16 ERNST AT130 DR hardness tester, with a load of 62.5 kg_f and a 2.5 mm diameter spherical

17 indenter, according to the UNI EN ISO 6505-1: 2015 standard.

18

19 2.3 Microstructural Characterization

1   Thin sections were cut from the cuboid samples and subjected to standard metallographic

2   preparations. To further support the density measurements, the porosity of unetched specimens

3   was investigated using image analysis with a Leica DMI 5000M optical microscope (OM). The

4   relative density was calculated as the percentage of the analysed surface not occupied by the

5   porosity. Size and morphology of the pores were evaluated respectively through the Equivalent

6   Diameter (ED) [μm] and Circularity (C) parameters, accordingly to the following expressions:

$$ED = \left(\frac{4 \cdot A}{\pi}\right)^{1/2} \qquad\qquad (2)$$

$$C = \frac{4 \cdot \pi \cdot A}{P^2} \qquad\qquad (3)$$

7   where A [μm^2] is the area of the pores and P [μm] their perimeter. Samples were subsequently

8   etched for 10 s with a 1% HF distilled water solution. Microstructural and fractographic

9   observations were performed with a Zeiss EVO MA 15 scanning electron microscope (SEM) on

10   cuboid and fractured tensile samples, respectively. Size and spacing of microstructural and

11   fractographic features were measured with the software ImageJ. The width of grains and Al cells

12   and the ED of Si particles and pores were assumed as representative size parameters, whereas the

13   spacing of Si particles and dimples was measured with the 3-Nearest Neighbours Distance

14   (3NND) method. X-ray diffraction (XRD) tests were performed on the polished top surface of

15   cuboid samples in a Bragg-Brentano geometry with a Bruker D8 Advance diffractometer equipped

16   with a Cu filament (Kα, 1.5406 Å). Data were acquired in the 2ϑ range from 20 ° to 110 °, with a

17   step-size of 0.02 ° and a step-time of 1 s. Quantitative measurements were computed with the

18   Rietveld refinement method using the software Maud [28]. A 3-parameter harmonic function was

19   used to account for the presence of texture and reduce the error in quantitative measurements.

20

21   3. Results and Analyses

3.1 Level of porosity and density

Figures 2a-b-c depict representative OM micrographs of samples produced with low,

intermediate and high VED, respectively. At 43 J/mm^3 (Fig. 2a) the sample exhibits a low number

of pores, most of which being a few tens of μm in size. As VED increases to 56 J/mm^3, large circular

pores, several tens of μm wide, appear. Mostly large pores up to 100-200 μm in size and mainly

located at the bottom of melt pools characterises the sample processed with 66 J/mm^3. In addition

to keyhole pores, other defects were sometimes detected in the as-built material, like Mg-based

oxides and small shrinkage voids. They were found in a small number in all the investigated

samples, with a size generally smaller than large keyhole pores. Accordingly, their effect on the

final properties was assumed to be negligible and will not be discussed furtherly.

Image analysis was carried out to determine ED and C of pores, and Fig. 2d reports the

Probability Density Functions (PDF) of such parameters of samples in Figs. 2a-b-c. At low VED,

the ED statistical distribution is narrow and located in the range of a few tens of μm. As VED

raises, the distributions gradually flatten and broaden to higher ED values, which mirrors the

appearance of larger pores in Figs. 2b-c. Conversely to ED, the C statistical distributions slightly

narrow as VED increases. Most of the pores in each sample range between 0.7-0.9 of C, indicating a
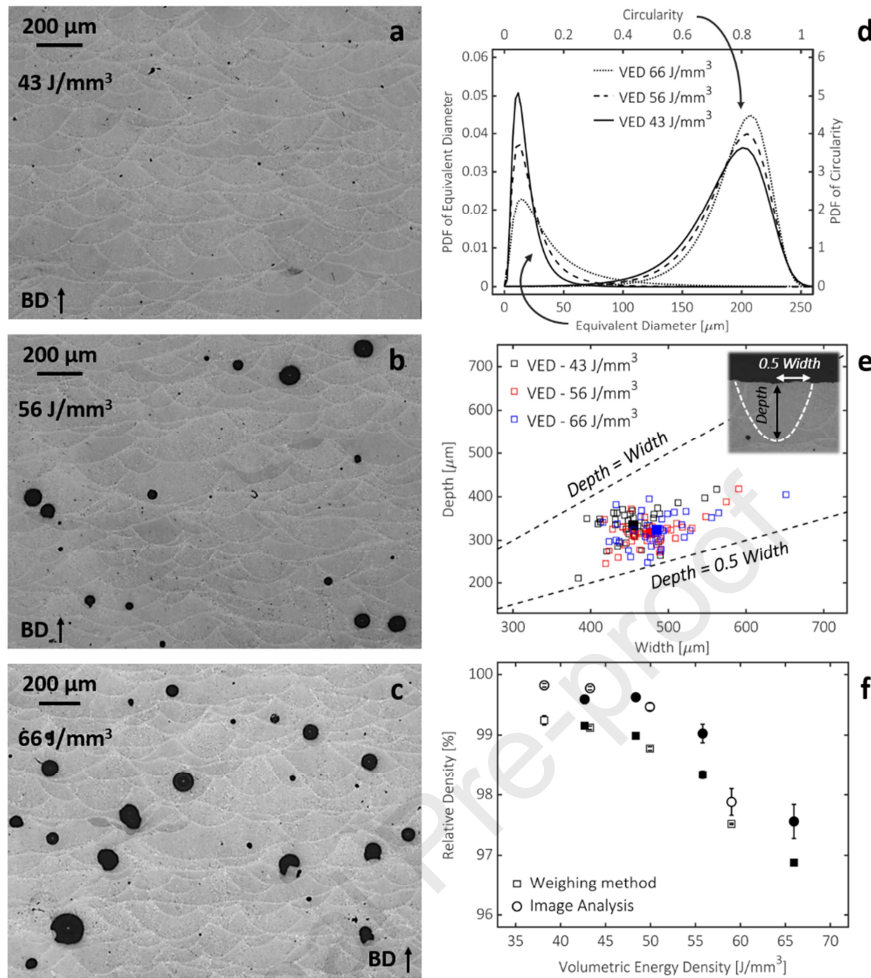
nearly circular morphology.

Figure 2. OM micrographs of samples produced with different VED: (a) 43 J/mm³, (b) 56 J/mm³, (c) 66

J/mm³, vertical arrows point out the building direction (BD). (d) ED and C statistical distributions of pores

of samples in (a), (b) and (c). (e) Aspect ratio of the melt pools. (f) Relationship between VED and relative

density, filled and open markers correspond to hatch spacing h and 1.12 h, respectively. Standard

deviation is reported as error bar.

1

2   King *et al.* [29] proposed a geometrical criterion to assess the heat transfer mode operating

3   during the deposition of an AISI 316L alloy. A depth-to-width ratio of the melt pool of 0.5 is the

4   threshold between conduction (< 0.5) and keyhole (> 0.5) modes. The same criterion was also

5   adopted for Al alloys [30]. The depth-to-width ratio of melt pools of the top layer was evaluated to

6   avoid multiple overlaps and accurately measure their size. Figure 2e shows that all data of samples

1   previously mentioned range between 0.5 and 1 of depth-to-width ratio, with no significant effect of

2   VED, suggesting a keyhole heat conduction mode.

3   Figure 2f reports the relationship between VED and the relative density. Measurements by the

4   weighing method are always ~0.5% lower than by image analysis. A lower size threshold of 70 μm^2

5   (ED ~10 μm) was applied to pores which suffer from limited resolution during image analysis.

6   Additionally, the corresponding relative density stems from an area measurement, while the

7   weighing method measures the whole sample. These observations could justify the difference

8   mentioned above. However, both methods provide the same trend, with the relative density

9   progressively reducing as VED increases. The highest value of 99.8-99.3% was reached with a VED

10   of 38 J/mm^3, and it falls to 96.5-97.5% as the energy density reached 66 J/mm^3. The distinction

11   between filled and open markers in Fig. 2f is related to the two employed h. It appears that the

12   progressively deteriorating effect of building parameters (v and h) on the relative density can be

13   described with a univocal function of the energy density.

14

15   3.2 Microstructural and X-ray diffraction analyses

16   Although VED in the analysed range considerably affects porosity, its effect on the

17   microstructure was much less evident. Accordingly, Fig. 3 depicts typical features of the sample

18   processed with parameters 1.55v-h, taken as the reference. The AlSi10Mg alloy exhibits a

19   hierarchical microstructure, with the melt pools that occupy the outer level (Fig. 3a). They are

20   delimited by melt pool boundaries, coarse zones produced with a low local cooling rate during the

21   solidification process, highlighted by black arrows in Fig. 3a. Inside the melt pools, the channelling

22   contrast of back-scattered electrons reveals large columnar grains elongated toward the building

23   direction (Fig. 3b). These grains are 5-20 μm wide and up to 200 μm long, and often epitaxially

24   grow across several melt pools. Each grain is made up of a continuous network of fibrous eutectic

1   Si that surrounds small α-Al cells, marked by black and white arrows in Fig. 3c, respectively. These

2   micrometric cells are about one order of magnitude smaller than grains in Fig. 3b, with which they

3   share the same elongated axis. The amount of eutectic phase, calculated as ~20 ± 4% by image

4   analysis, is far distant from the one expected for an AlSi10 alloy (~75%). Nano-sized Si precipitates,

5   hosted inside the α-Al cells, occupy the inner level of the hierarchical microstructures. They exhibit

6   both globular and acicular morphology, as highlighted respectively by white and black arrows in

7   Fig. 3d, and are tens of nm in size. They could either result from a reduced solubility of Si inside

8   the α-Al cells during the cooling process or precipitation-induced phenomena caused by the

9   neighbouring laser scans. The lack of Mg-containing phases suggests the element to be retained in

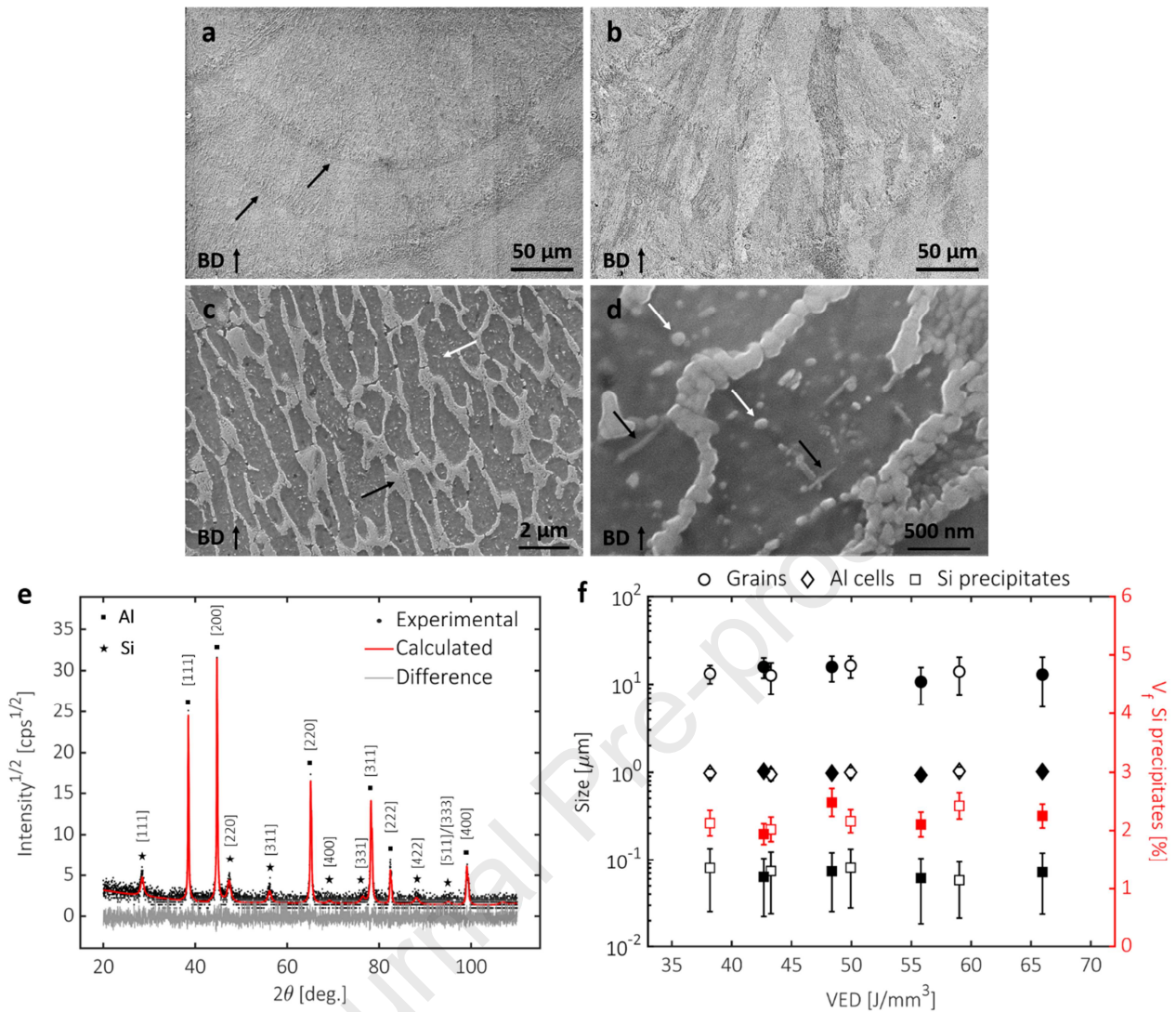10   solid solution inside the α-Al phase.

11

Figure 3. SEM micrographs of the AlSi10Mg alloy, vertical arrows point out the building direction (BD). (a) Melt pool boundaries highlighted by black arrows. (b) Same region as in (a), epitaxial columnar grains. (c) Al cells (white arrow) and eutectic network (black arrow). (d) Nano-sized Si precipitates with globular (white arrow) and acicular (black arrow) morphology. (e) Experimental and calculated XRD patterns. (f) Microstructural features with different VED values. Filled and open markers correspond to hatch spacing h and 1.12h, respectively. Standard deviation is reported as error bar. The reduced standard deviation is not visible in size measurements of Al cells.

1

2   XRD analyses furtherly confirmed the existence of a non-equilibrium microstructure suggested

3   by SEM investigations. As an example, Fig. 3e reports the experimental (dotted) and simulated

1   (solid) diffraction patterns of the 1.55v-h sample. Following SEM observations, none of the

2   experimental peaks matched with an Mg-bearing constituent. Therefore, the simulated pattern

3   accounts only for the Al and Si phases. The difference curve lies around the zero value and shows

4   that experimental and simulated patterns match well. Instead of the Al [111] being the strongest

5   peak in the diffraction pattern, a pronounced crystallographic texture is present, with the Al [200]

6   reflection being the most intense. Quantitative calculations pointed out some deviations from the

7   equilibrium conditions. A lattice parameter of 4.0498 ± 0.0040 Å was measured for the α-Al phase,

8   which is quite lower than the equilibrium one (4.0515 Å) of the AlSi10Mg alloy [31]. Additionally,

9   Si reflections barely stand out from the background in Fig. 3e, suggesting a reduced amount of

10   crystalline Si with a nanometric size. Indeed, a weigh fraction of Si of ~8.0 ± 0.3% was calculated by

11   the Rietveld method. From the mass balance of the alloy with a calculated amount of Si of 9.6 wt.%

12   by optical emission spectroscopy, the residual Si retained in solid solution in the α-Al phase was

13   estimated as ~1.85 wt.%.

14   Microstructural features were quantitatively evaluated by image analysis to confirm the

15   negligible effect of VED, and results are reported in Fig. 3f. The logarithmic Y-axis shows that the

16   size of microstructural features extends over two orders of magnitude, but it does not appreciably

17   change with VED. The mean width of elongated grains and cells is 14 ± 2 μm and 1 ± 0.05 μm,

18   respectively. The mean ED and volumetric fraction of Si precipitates are 72 ± 8 nm and 2.2 ± 0.2 %,

19   respectively, similarly to [32].

20

21   3.3 Mechanical properties and fracture surfaces

22   Figure 4 depicts the relationship between VED and Brinell hardness, which mirrors the one in

23   Fig. 2f. As the energy density increases, porosity significantly raises and negatively affects

24   hardness. Data in Fig. 4 are reported into two series (filled and open markers) as a function of *h*.

1 Both series show that, for a given h, hardness decreases with a reduction of v. Similarly, at constant

2 v, hardness is lower using a smaller h. As in Fig.2f, the separate effect of deposition parameters can

3 be described incorporating them in a single energy parameter, that is in a univocal relationship
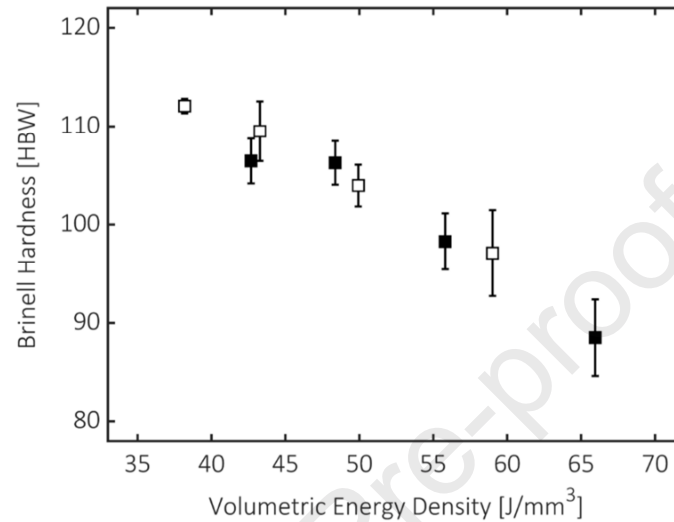
4 with the hardness.

Figure 4. Brinell hardness of samples produced with different VED values. Filled and open markers

correspond to hatch spacing h and 1.12h, respectively. Standard deviation is reported as error bar.

5

6   Figure 5 depicts the engineering stress-strain curves of each investigated condition. Specimens

7 produced with low v and h (v-h, v-1.12h), and thus with the highest energy density, display the

8 worst mechanical properties. As the energy density reduces, both strength and ductility

9 progressively raise, reaching the outstanding tensile strength of ~500-550 MPa, and a reasonable

10 elongation of ~6%, at the highest values of v and h (1.55v-h, 1.55v-1.12h). Similar performances

11 were achieved just for specifically designed high-strength Al alloys [7,8,32], while the resistance of

12 the AlSi10Mg alloy ranges between 350-450 MPa in the literature [10,16,20,27].

13   Despite the different final properties, all the stress-strain curves display a similar shape. The

14 applied stress reaches a maximum, and then the alloy abruptly fails during the strain-hardening

15 stage, without any evidence of stress reduction. The necking stage is absent, and thus the alloy fails

14

1   before the potential UTS is reached. True stress (σ) and strain hardening rate (Θ) are plotted as a

2   function of the true strain (ε) in the insets in Fig. 5 for each investigated condition to assess such

3   behaviour furtherly. According to Considère's criterion [33], necking starts when $\Theta = d\sigma/d\varepsilon = \sigma$, at

4   the intersection of σ and Θ curves. However, insets in Fig. 5 show that, in most cases, such a

5   condition is not satisfied, even if σ and Θ values at failure are generally quite close. Similar

6   behaviour is often reported in the literature of additively manufactured parts [21,34,35], and it is

7   ascribed to the existence of a high density of microstructural defects, like tangled dislocations,

8   residual stresses and sub-micrometric pores. However, in the current study, abrupt failure is more

9   likely determined by keyhole pores, which overtake the effect of features mentioned above.
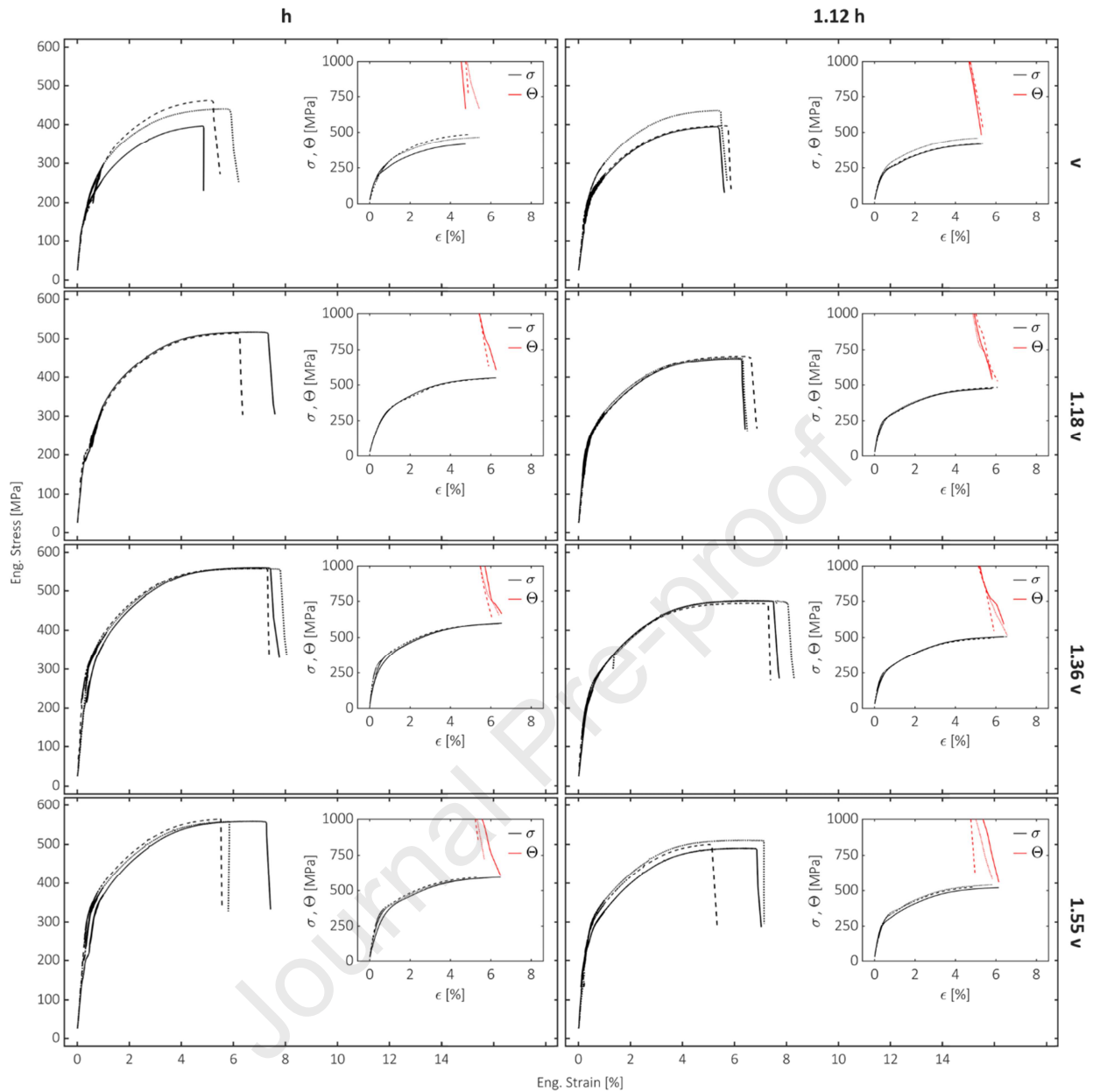
Figure 5. Engineering stress-strain curves of samples produced with different VED values. Insets depict the

corresponding true stress-true strain (σ) and strain hardening-true strain (Θ) curves.

1

2   Figure 6 reports tensile parameters as a function of investigated *v-h* combinations and the

3   corresponding VED. A maximum value of 78 ± 9 GPa of elastic modulus (E) is reached at the

4   lowest energy density of 38 J/mm³. Similar E values were reported for a fully dense AlSi10Mg alloy

5   [27]. The elastic modulus falls almost linearly to 57 ± 6 GPa as the VED increases to the maximum

6   value of 66 J/mm³. Other tensile parameters, yield strength (YS), ultimate tensile strength (UTS)

16

1 and elongation at failure (E_f), also approximately decrease with the energy density, and each one is

2 reduced by ~20-25% of its maximum value. YS falls from 300-320 MPa down to 220-240 MPa and

3 UTS from 500-550 MPa to 410-430 MPa, whereas E_f decreases from 6-6.5% to ~5%.

4   Figure 6 shows that, conversely to elastic modulus and hardness, specimens produced at

5 constant v with the lowest h (h) exhibit better performances than their counterparts (1.12 h). UTS

6 more clearly shows such an effect, because of the high deviation that YS and E_f suffer. A further

7 comparison concerns samples produced with very close VED values. The energy density of 1.55v-h

8 and 1.36v-1.12h specimens is almost the same (42 and 43 J/mm³ respectively), but the former

9 display better mechanical properties as compared to the latter. The same observation rules for the

10 1.36v-h and 1.18v-1.12h specimens, realised with a VED of 48 and 50 J/mm³, respectively.
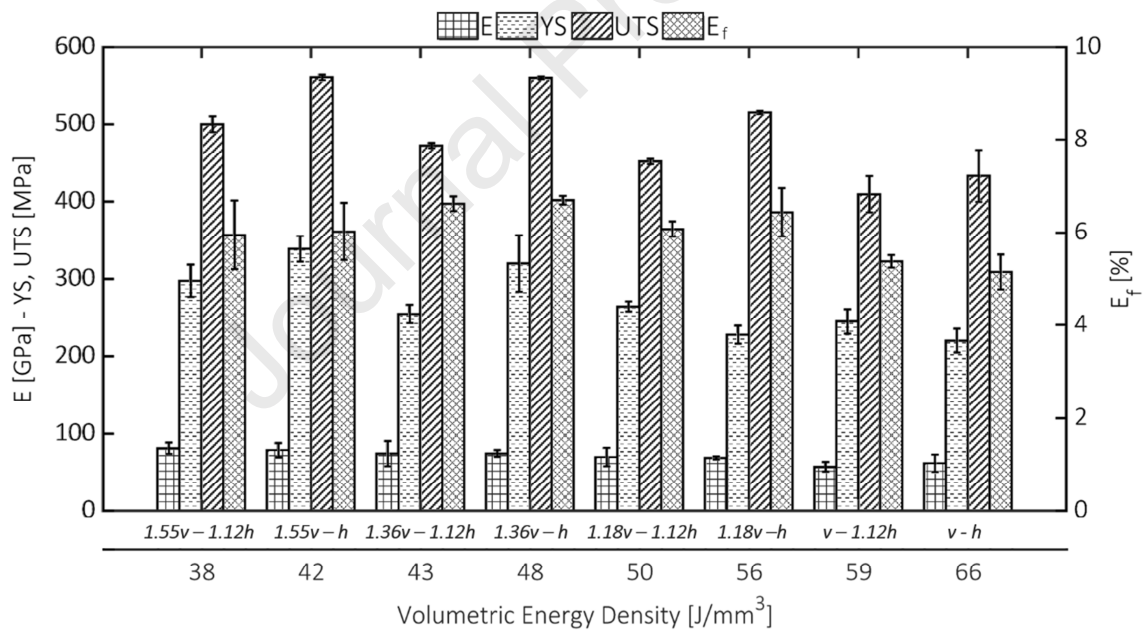

Figure 6. Tensile parameters of samples produced with different VED values. Standard deviation is
reported as error bar.

11

12   Results of image analysis in Fig. 1f showed that the porosity increases as VED raises. A

13 complementary analysis was performed on the fracture surface of tensile specimens, and results

17

1   are reported in Fig. 7. Probability histograms depict the size distribution of pores on the fracture

2   surfaces and confirm results in Fig. 1d: at low VED, most of the pores are a few tens of μm wide,

3   with the probability of finding large pores (ED > 100 μm) being almost zero. At intermediate VED,

4   the appearance of pores several tens of μm wide became more and more probable. These latter are

5   the most probable at high VED, together with large pores ranging between 100-200 μm.

Figure 7. ED probability histograms of pores on the fracture surfaces of samples produced with different deposition parameters.

1

2   A comparison between Fig. 7 and Fig. 1d shows that the size range of pores on fracture surfaces

3   and metallographic sections closely correspond at each VED value. This result suggests that tensile

4   specimens experienced limited plasticity before failure, with pores being otherwise somewhat

5   deformed. In turn, this observation matches the tensile behaviour and premature failure shown in

6   Fig. 6.

7   A more in-depth analysis of the fracture surfaces is proposed in Fig. 8, considering specimens

8   produced with the lowest (Figs. 8a-b) and highest (Figs. 8c-d) energy densities, respectively.

9   Samples processed with a low VED show a limited number of small pores and several elongated

10   features, these latter marked by a dashed circular line in Fig. 8a. Figure 8b clarifies these features to

11   be traces of tracks produced by the laser scan. Other works report the coarse and heat-affected

12   microstructure of melt pool boundaries to be a weak microstructural feature and an easy fracture

13   path [36], which explains the presence of scan tracks on the fracture surface. The inset of Fig. 8b

14   points out that, on a smaller scale, the fracture surface is made up of fine dimples.
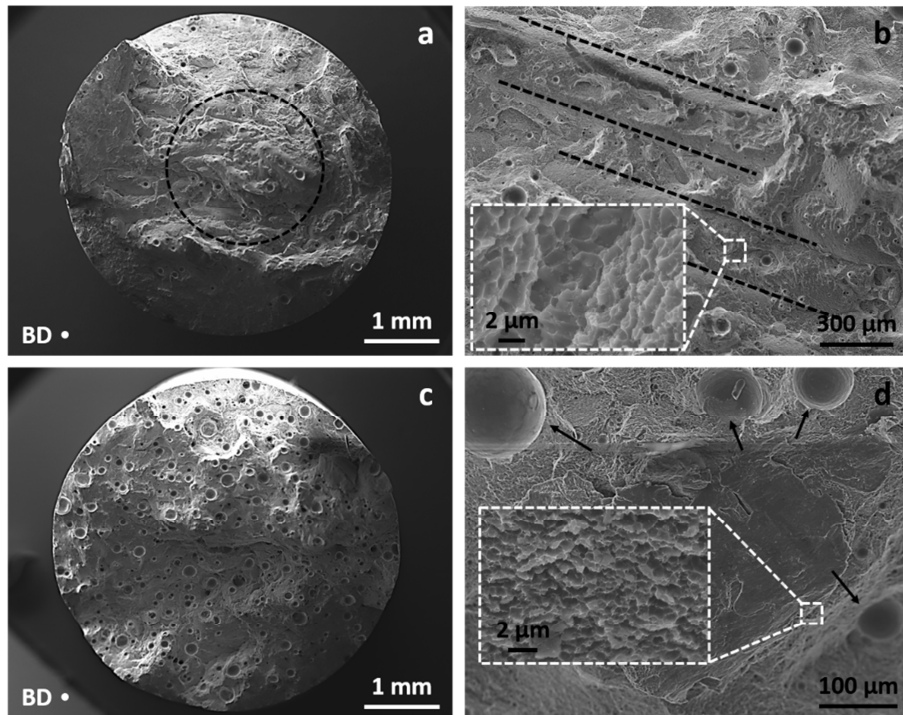
Figure 8. Fracture surfaces of samples produced with (a-b) low and (c-d) high energy density. (a) Macro-fracture, the circled area highlights elongated features. (b) Particular of the scan tracks marked with dotted lines; the inset depicts fine dimples. (c) Macro-fracture. (d) Particular of coarse pores pointed by black arrows; the inset depicts fine dimples. In (a-c), the dot on the bottom left corner highlights the building direction (BD).

1

2   Samples produced with a high energy density show copious coarse pores on the surface (Fig.

3 8c). Conversely to Figs. 8a-b, traces of the scan tracks are not distinguishable. Pores represent a

4 weaker feature than melt pool boundaries, and they mainly guide the fracture at a macroscopic

5 level, making the scan tracks no longer detectable. On the contrary, the inset of Fig. 8d depicts

6 small dimples as in Fig. 8b, elucidating a similar fracture mechanism at the microscale. The

7 fracture behaviour at the microscale was deepened analysing and comparing the spacing of fine

8 dimples in Figs. 8b-d and smallest microstructural features, Al cells and Si precipitates, in Figs. 3c-

9 d. Results were averaged among all the investigated conditions since no significant differences of

10 microstructural features were detected (Fig. 3f). From Fig. 3f, the spacing of Al cells matches their

1   width, and it is ~1 μm, whereas the mutual distance of Si precipitates was measured as ~210 ± 75

2   nm. For the ease of analysis, no distinction was made between globular and acicular precipitates,

3   which accounts for the scatter of measurements. The mean spacing of dimples on the fracture

4   surfaces was measured as ~270 ± 65 nm, which more closely resembles that of Si particles rather

5   than Al cells, suggesting the formers to mainly rule the deformation behaviour at the microscale.

6

7   4. Discussion

8   4.1 Process parameters, VED and porosity

9   OM observations in Figs. 2a-b-c highlight that, a decrease of either v or h promotes the

10   formation of large round pores up to 100-200 μm wide. Consequently, the relative density falls

11   from 99% of the almost dense material to ~96%, as reported in Fig. 2f.

12   The AM process of Al components could suffer from different kinds of porosity, usually

13   classified in lack-of-fusion and keyhole porosity. The former depends on the incomplete powder

14   melting, caused by a poor overlap of melting pools or by insufficient laser power. Due to their

15   inherent nature, lack-of-fusion pores are usually very irregularly-shaped [15], and do not fit with

16   the current OM observation. Conversely, the keyhole porosity is determined by an excess of

17   energy that the laser imparts to the powder. The resulting overheating, metal vaporisation and

18   recoil pressure, drive the melt pool surface deep into the material to form a keyhole depression.

19   Due to its inherent instability, such depression easily collapses and traps the inert shielding gas,

20   giving rise to the pore formation within the substrate. The resulting pore is almost spherical and

21   located at the bottom of the melt pool [13,14]. Accordingly, the circularity of pores in Figs. 2a-b-c,

22   and their location in the low part of melt pools, strongly suggest them to be keyhole pores.

23   Additionally, Fig. 2e shows that the depth-to-width ratio of all measured melt pools ranges

1 between 0.5 and 1, regardless of VED, confirming that samples were produced under a keyhole

2 heat conduction mode, following previously published works [29,30].

3

4 4.2 Process parameters, VED and microstructural features

5   SEM observation shows the hierarchical microstructure of the L-PBF produced AlSi10Mg alloy

6 (Fig. 3) [3,4,6]. Columnar grains grow radially and epitaxially through the melt pools (Fig. 3b),

7 with their cubic {100} orientation along the building direction (Fig. 3e). Epitaxy and strong

8 crystallographic texture can be ascribed to the strong thermal gradients that develop during the L-

9 PBF process [3]. Inside the grains, a fibrous Si network surrounds micrometric Al cellular dendrites

10 (Fig. 3c). An estimated high cooling rate of ~10^3-10^5 K/s [32] accounts for their micrometric size,

11 and determines the fibrous morphology of Si, accordingly to the quench modification

12 phenomenon [37]. The high cooling rate also extends the solubility of Si inside the Al phase during

13 the solidification. XRD analyses (Fig. 3e) confirm such a statement measuring ~1.85 wt.% of Si

14 retained in solid solution [5]. As the cooling proceeds, the solubility of Si falls and it is partially

15 ejected from the Al matrix to form nanometric features (Fig. 3d), even if they also likely precipitate

16 and coarsen due to the heating produced by neighbouring laser scans.

17   Besides the peculiar microstructure, a change in the deposition parameters as reported in Tab. 1

18 does not modify the microstructural features significantly (Fig. 3f). Size and morphology of these

19 latter depend on thermal gradient G and growth rate R. The G/R ratio determines the stability of

20 the solid/liquid front and the resulting solidification mode, while the G·R product, i.e. the cooling

21 rate, rules the final size of the microstructure. In the steady-state conditions:

$$R = v \cdot \cos \vartheta \tag{4}$$

22   where v is the scan speed, and ϑ is the angle between the scan direction of the laser and the

23 growth direction of the solidifying material [3]. G depends on the alloy properties and the energy

1   imparted to the material, thus on building parameters, like lase power P and scan speed v.

2   However, numerical simulations proved that P is a slightly influencing variable, while v has a

3   neglecting effect [38]. Therefore, a constant G value of ~10^6 K/m is often reported [39]. The limited h

4   difference of 12% employed in this work reasonably just slightly modifies the total heat input from

5   neighbouring scan tracks. Therefore, in the following discussion, its effect on microstructural

6   features will be neglected, with G/R and G·R mainly depending on v of the moving source.

7   Increasing v and thus reducing G/R, planar, cellular and dendritic structures are progressively

8   expected. Nevertheless, the Al phase of samples produced at different v always exhibits a cellular

9   structure (Fig. 3c-f). Likely, the high G prevents the increasing v from changing the solidification

10   mode. Similarly, the size of microstructural features does not change significantly among the

11   investigated samples (Fig. 3f), despite its dependence on the cooling rate according to $G \cdot R = G \cdot$

12    $v \cdot cos\vartheta$. A typical relationship between cell size and cooling rate is:

$$\lambda = a \cdot \left(\dot{T}\right)^{-n} = a \cdot (G \cdot R)^{-n} \tag{5}$$

13   where λ [μm] is the cell size for cellular growth, a [μm·s/K] is a proportional constant, $\dot{T}$ [K/s] is

14   the cooling rate, and n is a dimensionless exponent ranging between 0.32-0.38, often assumed as

15   0.33 [40]. From Tab. 1, the maximum v variation in this work is 55% for samples produced with v

16   and 1.55v. The maximum expected size difference of cells is ~13% applying equation (5), which

17   reasonably falls in the experimental uncertainty of size measurement. Similar considerations also

18   apply to other microstructural features, like elongated grains and Si precipitates, and explain why

19   no significant differences were found among the investigated samples (Fig. 3f).

20   Since similar microstructural features were obtained as the deposition parameters change, their

21   contribution to mechanical performance is more or less constant. Conversely, small variations of v

22   and h remarkably affect density (Fig. 2f). As a consequence, both hardness (Fig. 4) and tensile

1   properties (Fig. 5 and Fig. 6) decrease as VED increases. However, some relevant considerations

2   hide behind this somewhat trivial result, as will be elucidated in the following discussion.

3

4   4.3 Limitations of VED as a reliable design parameter

5   In this work, a VED of ~40 J/mm^3 is suitable to minimise the porosity level. Additionally, the

6   density reduction with any variation of v and h is fully described by VED (Fig. 2f), which stems

7   from the strong dependence of the keyhole phenomenon on the energy density. In this light, the

8   energy density-based approach seems a valuable tool to define a proper process window of

9   deposition parameters. However, Fig. 9a highlights a very different situation. According to

10   literature data, the relative density of the AlSi10Mg alloy in a VED range of 30-110 J/mm^3

11   uniformly distributes between 98-100%. Conversely, we found that porosity steeply increases as

12   VED raises from 40 to 70 J/mm^3 (red markers in Fig. 9a). Even if not available, any additional

13   increment beyond 70 J/mm^3 would further deteriorate density. Thus, a very different amount of

14   porosity could be achieved at the same VED value, which severely limits its applicability to

15   determine a suitable combination of deposition parameters. Such an outcome stems from the

16   extreme simplicity of equation (1): it comprehends just a few of the assessable process parameters

17   and does not account for material properties and physical events that characterise the dynamic of

18   solidifying melt pools. Therefore, a mere energy density-based approach cannot capture the

19   complexity of the AM process, with the effect of individual deposition parameters that cannot be

20   neglected.

21   Fig. 6 shows that tensile properties roughly decrease as VED increases, according to the raised

22   amount of porosity. However, specimens produced with an almost equal VED exhibit a very close

23   porosity but a different mechanical response. For instance, comparing 1.55v-h and 1.36v-1.12h

24   specimens (42 and 43 J/mm^3, respectively) and 1.36v-h and 1.18v-1.12h specimens (48 and 50

1 J/mm³, respectively), porosity differs less than 0.2%, whereas UTS differs ~100 MPa. In both cases,

2 the specimen realised with the lowest *h* shows superior mechanical properties, in agreement with

3 [41]. Such an effect is ascribed to the higher geometrical overlapping between adjacent tracks,

4 which provides a better material consolidation. This finding suggests that process parameters exert

5 additional effects, besides energy transfer, not taken into account by the energy density-based

6 approach. For industrial purposes, this limitation possibly leads to define the best combination of

7 deposition parameters erroneously.

Figure 9. Comparison of literature data of the AlSi10Mg alloy (grey clouds) with results from this work

(red clouds) as a function of VED. (a) Relative density. (b) UTS. (c) E_f.

8

9 Figures 9b-c furtherly compare present tensile results with available literature data. In the 30-

10 110 J/mm³ range of VED, UTS of the AlSi10Mg alloy ranges between 300-400 MPa, with few

11 exceptions up to 450 MPa [34,42], whereas E_f ranges between 1-6%. Conversely, our specimens

26

1   exhibit a systematically higher UTS (400-550 MPa), preserving a considerable E_f (5-6.5%). These

2   latter values are quite surprising considering the significant porosity of our samples (up to 3.5%)

3   and their vertical building orientation. Vertically built specimens generally show worse tensile

4   properties, particularly E_f, than horizontally built ones [2,10,20], with the corresponding values

5   that occupy the bottom part of Fig. 9c.

6   The outstanding mechanical properties reached in this work could be ascribed to the

7   hierarchical structure of the AlSi10Mg alloy. It exploits all the strengthening mechanisms of

8   metallic materials, namely internal friction, Hall-Petch, Orowan, Taylor, solid solution and load-

9   bearing mechanisms, with the corresponding relationships reported, in this order, as follows:

$$YS_{est.} = [\sigma_0] + \left[\frac{k}{\sqrt{d_{cell}}} + \frac{k}{\sqrt{d_{grain}}}\right] + \left[\frac{\varphi Gb}{d_{Si.particle}}\left(\frac{6V_{Si.particle}}{\pi}\right)^{1/3}\right] + [\beta MGb\sqrt{\rho_d}] + [HC_{Si}^{N}] + [1.5\sigma_i V_{Si.eutectic}] \qquad (6)$$

10   where σ_0 is the internal friction (72 MPa, [43]), k (50 MPa·μm$^{0.5}$, [32]), φ (~2, [32]), β (0.16, [44]),

11   H (~13 MPa·at.%$^{-1}$, [45]), N (1, [45]) are a material constants, d_{cell} (1 ± 0.05 μm) and d_{grain} (14 ± 2

12   μm) are widths of Al cells and elongated grains, G (27000 MPa) and b (0.286 nm) are shear

13   modulus and Burger vector of the Al phase, $d_{Si.particle}$ (72 ± 8 nm) and $V_{Si.particle}$ (0.022 ± 0.002) are

14   size and volumetric fraction of Si precipitates, M is the Taylor factor of FCC crystals (3.06, [34]), ρ_d

15   is the dislocation density (3.05·10^{14} m^{-2}, [46]), C_{Si} is the solid solution Si (~1.8 ± 0.3 at.%), σ_i is the

16   bonding strength of the Al/Si interface (240 MPa, [47]), and $V_{Si.eutectic}$ is the volumetric fraction of

17   eutectic Si. This latter value is calculated as the difference between total crystalline Si (0.091 ± 0.004,

18   from XRD results) and Si precipitates. Applying (6), individual reinforcement contributions are

19   calculated as 72, 63, 75, 66, 23 and 25 MPa, which shows the relevant role of Al cells and Si

20   precipitates to the alloy strength. The estimated yield strength $YS_{est.}$ is 325 ± 10 MPa, which very

21   well matches YS of 1.55v-h and 1.55v-1.12h samples, and thus correspond to the strength of the

22   almost dense material. In this light, the contribution of porosity Φ [%] to strength loss can be

27

1   estimated as ~40 MPa· Φ^{-1} from data in Fig. 6. The harmful effect of pores is much pronounced

2   compared to the strengthening effect of microstructural features, which suggest the importance of

3   using optimised process parameters.

4   Preservation of a high E_f despite the increasing porosity stems from the microscale fracture

5   behaviour. In dense samples, MPBs represent the weakest fracture path (Figs. 8a-b), whereas

6   keyhole pores substitute them in porous specimens (Figs. 8d-e). Despite such difference at the

7   macroscale, similar fine dimples form during the deformation stage at the microscale (insets of

8   Figs. 8b-d). Quantitative analyses show that dimples are ~270 nm spaced similarly to Si

9   precipitates (~210 nm), from which they likely nucleate and grow. During the deformation stage, Si

10   precipitates provide plenty of additional sites for strain localisation other than large keyhole pores,

11   avoiding these latter to cause excessive damage accumulation and enhancing a ductile behaviour

12   [48]. As a consequence, E_f limitedly suffers from the increased porosity, and moderately falls from

13   6.5% to 5%. A further reason for the relevant ductility of our specimens is the lack of defects other

14   than keyhole pores, like oxides, unmelted powder particles and shrinkage voids, as stated in

15   section 3.1. Such features are sometimes reported in additively manufactured Al alloys [10,11,20]

16   and would remarkably contribute to tensile properties reduction. It is worth to note that, despite

17   the relevant ductility, tensile curves in Fig. 5 show that all samples prematurely fail before the

18   necking stage. Keyhole pores and possible additional defects, such as densely tangled dislocations

19   and residual stresses, likely explain such behaviour.

20   The analysis reported above shows that VED is not a reliable parameter to estimate tensile

21   properties of the additively manufactured AlSi10Mg alloy. The energy density-based approach

22   accounts for the effect of deposition parameters mainly on porosity but lacks in considering their

23   impact on the microstructural features which, as here demonstrated, significantly impact on the

24   final mechanical performance. Our work highlights that the use of VED, as currently defined, to

1 determine the proper combination of process parameters would not necessarily guarantee the

2 optimum choice. Hence, although it could help in determining an adequate range of process

3 parameters, it cannot be intended as a reliable design parameter.

4

5 5. Concluding remarks

6 In the present work, different combinations of building parameters were employed to realise

7 AlSi10Mg alloy specimens by DMLS technology within a specific VED range, based on a literature

8 survey. Metallurgical and mechanical characterisations were conducted to investigate the effect of

9 VED and deposition parameters on porosity, microstructural features and tensile properties of the

10 alloy. Results were compared with literature data to highlight some relevant limitations of the

11 energy density-based approach. The following conclusions can be drawn:

12 ▪ A limited variation of process parameters remarkably affects the porosity of the alloy but has a

13 negligible effect on the microstructural features. This is an advantage where the process is to be

14 optimised for defect reduction or productivity maximisation.

15 ▪ The VED range suggested in the literature does not necessarily lead to fully dense parts, with

16 the present specimens showing up to 3.5% of porosity. Very different amount of porosity could

17 be achieved with the same VED value. The simplified energy density-based approach just

18 roughly estimates the energy imparted to powder bed and cannot predict the final density of

19 additively manufactured components. Besides considering just a few parameters, it lacks in

20 assessing the effect of individual deposition parameters on the dynamic of the melt pool, which

21 significantly affects porosity.

22 ▪ The almost dense material shows outstanding tensile properties. The strengthening

23 contribution of each microstructural feature was estimated to assess its individual effect on

24 material strength. Results well match experimental tensile properties and show that Al cells

1   and Si precipitates provide the primary reinforcement. However, even a limited porosity

2   dramatically reduces material strength and overtakes the contribution of microstructure.

3   ▪ At equal or similar VED, specimens produced with a lower *h* show better tensile properties.

4   Additionally, our specimens exhibit systematically higher tensile performance as compared to

5   literature data. VED just roughly estimates the effect of deposition parameters on the amount

6   of energy imparted to the powder bed. Conversely, it lacks in assessing their effect on the

7   microstructural features and fails in predicting the final properties of the alloy.

8   ▪ As currently defined, the use of VED guarantees neither the selection of suitable deposition

9   parameters nor the optimisation of material properties. Hence, it cannot be intended as a

10 reliable design parameter.

11

19

20 Data availability

21 The data that support the findings of this study are available from the corresponding author on

22 reasonable request.

23

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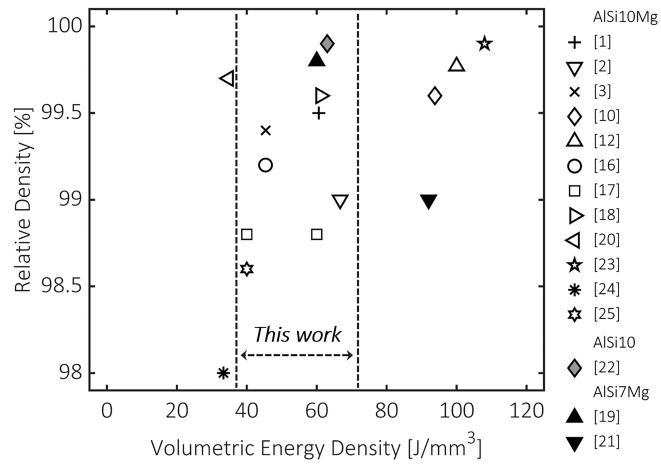
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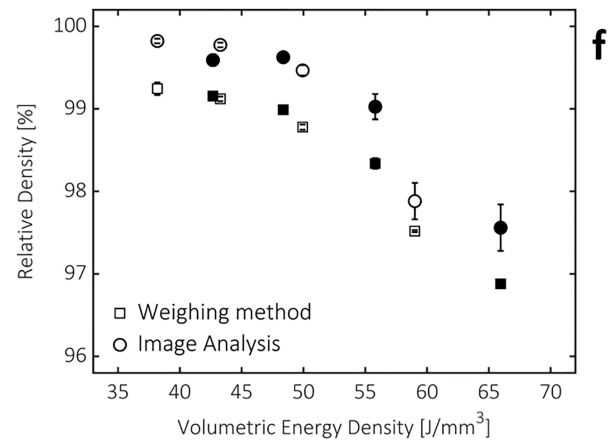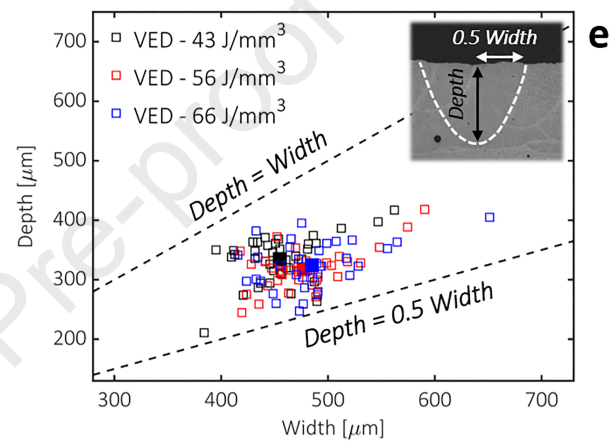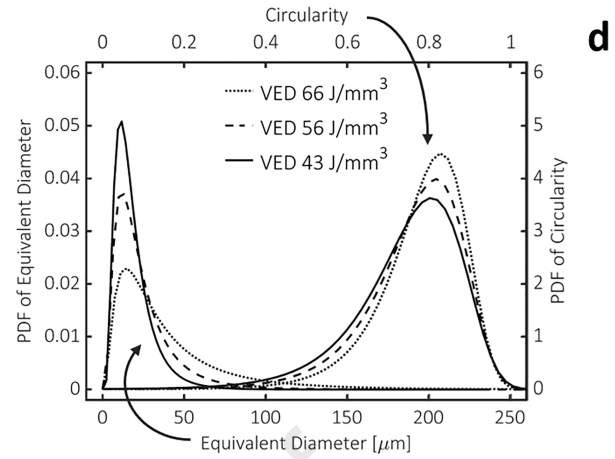[25]   T. Kimura, T. Nakamoto, M. Mizuno, H. Araki, Effect of silicon content on densification, mechanical
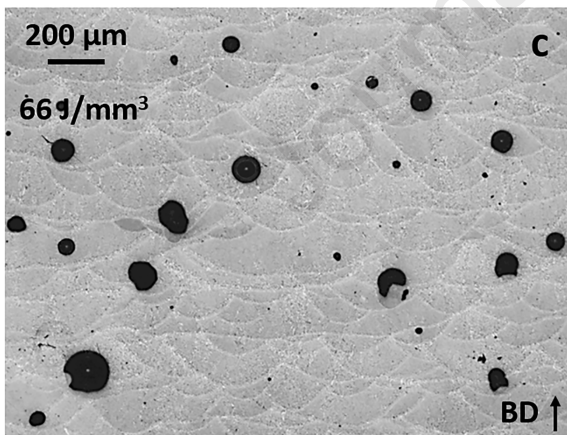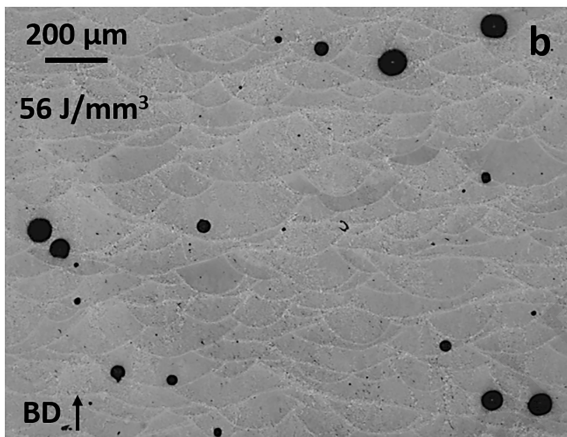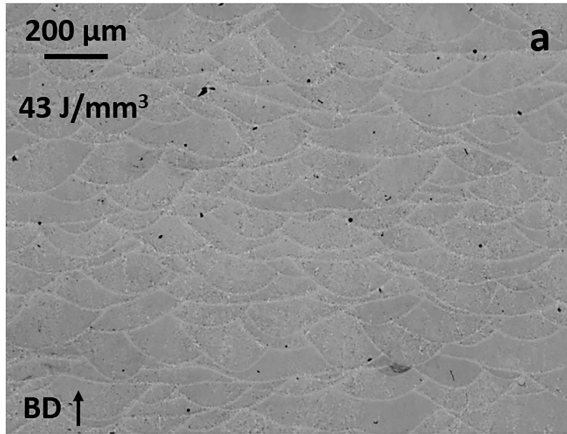
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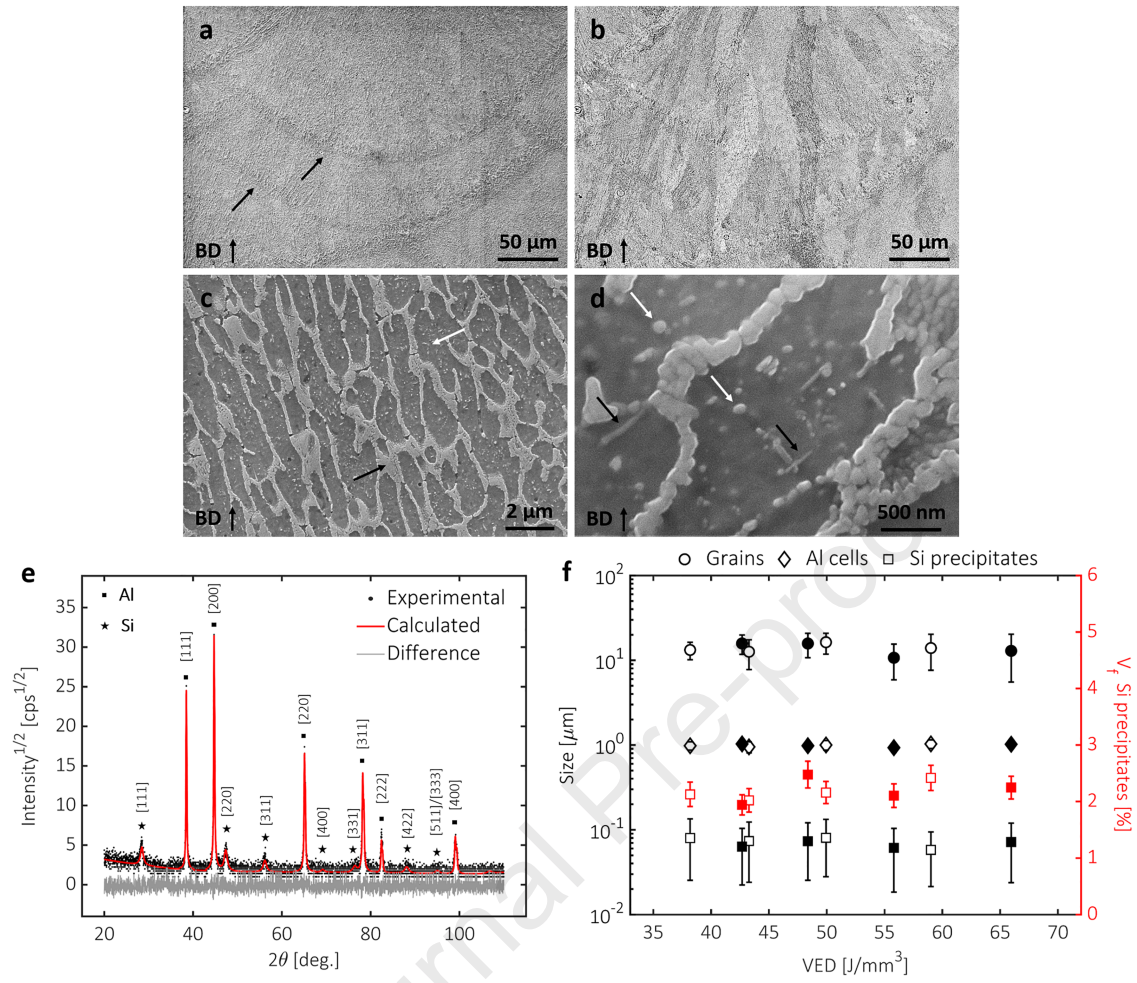24   [34]   B. Chen, S.K. Moon, X. Yao, G. Bi, J. Shen, J. Umeda, K. Kondoh, Strength and strain hardening of a

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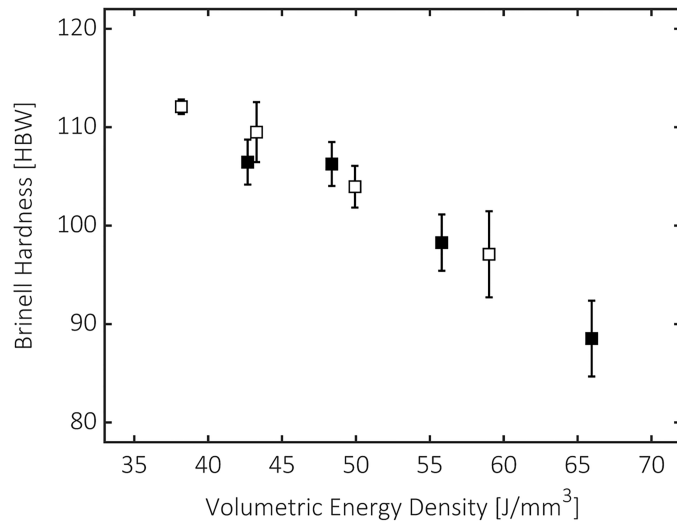alloy fabricated by selective laser melting: Microstructure evolution, mechanical properties and

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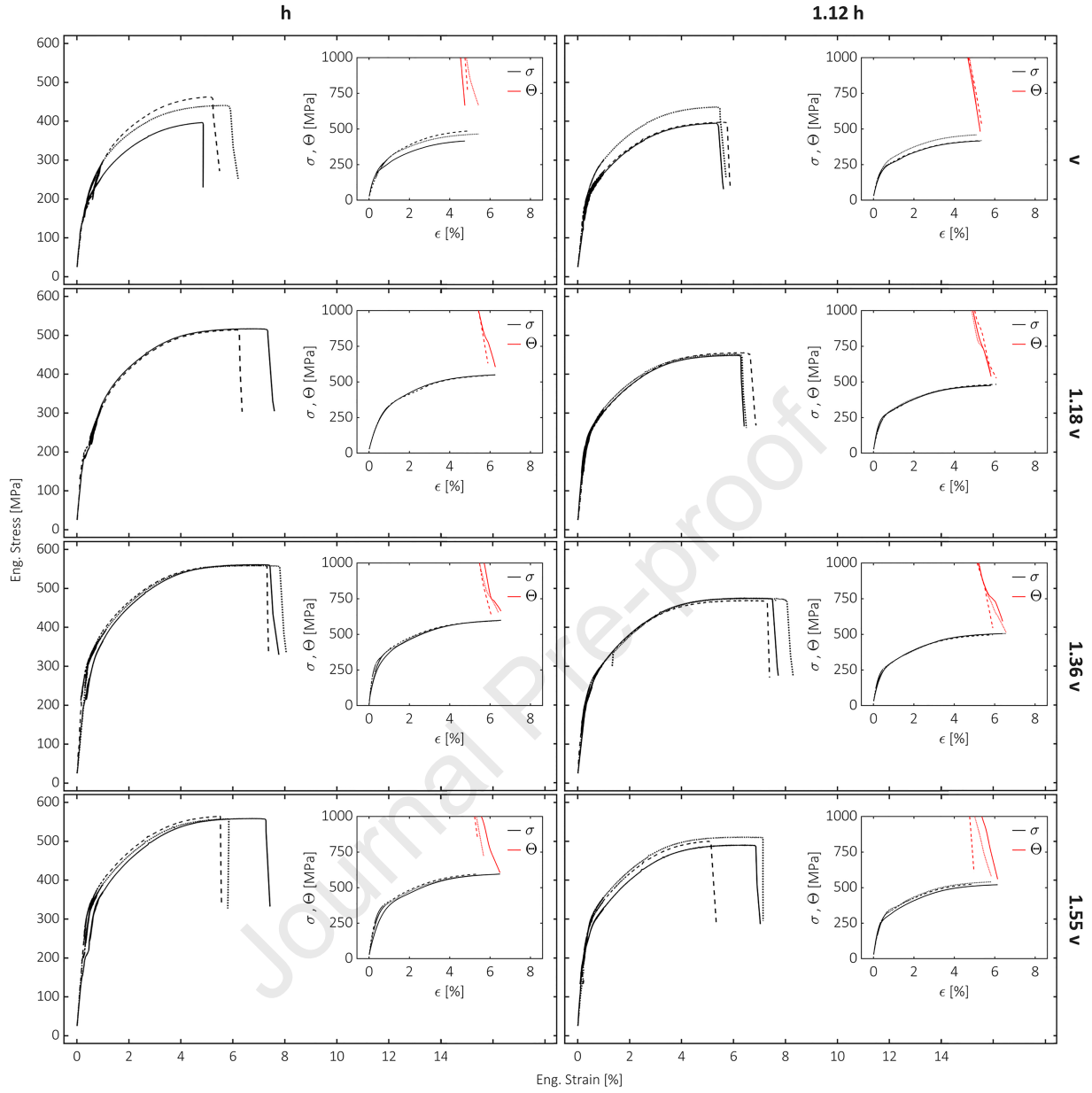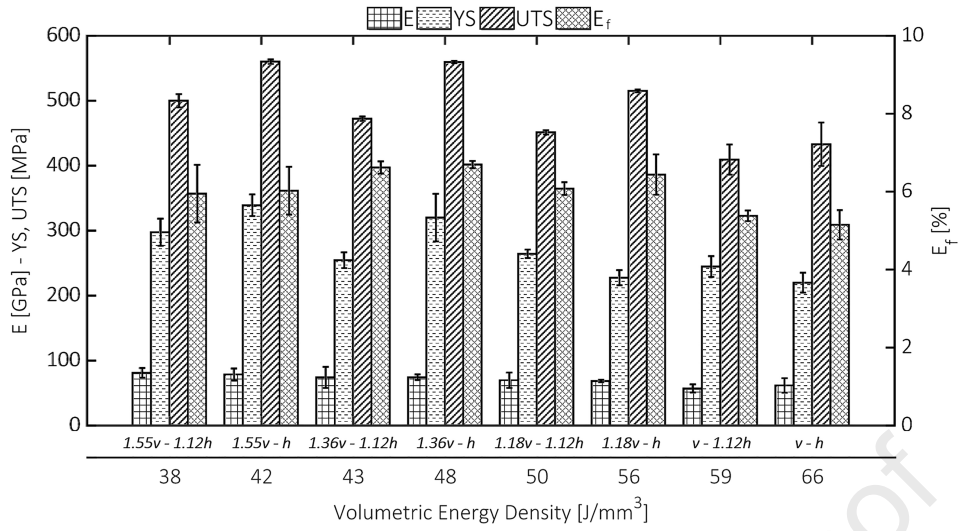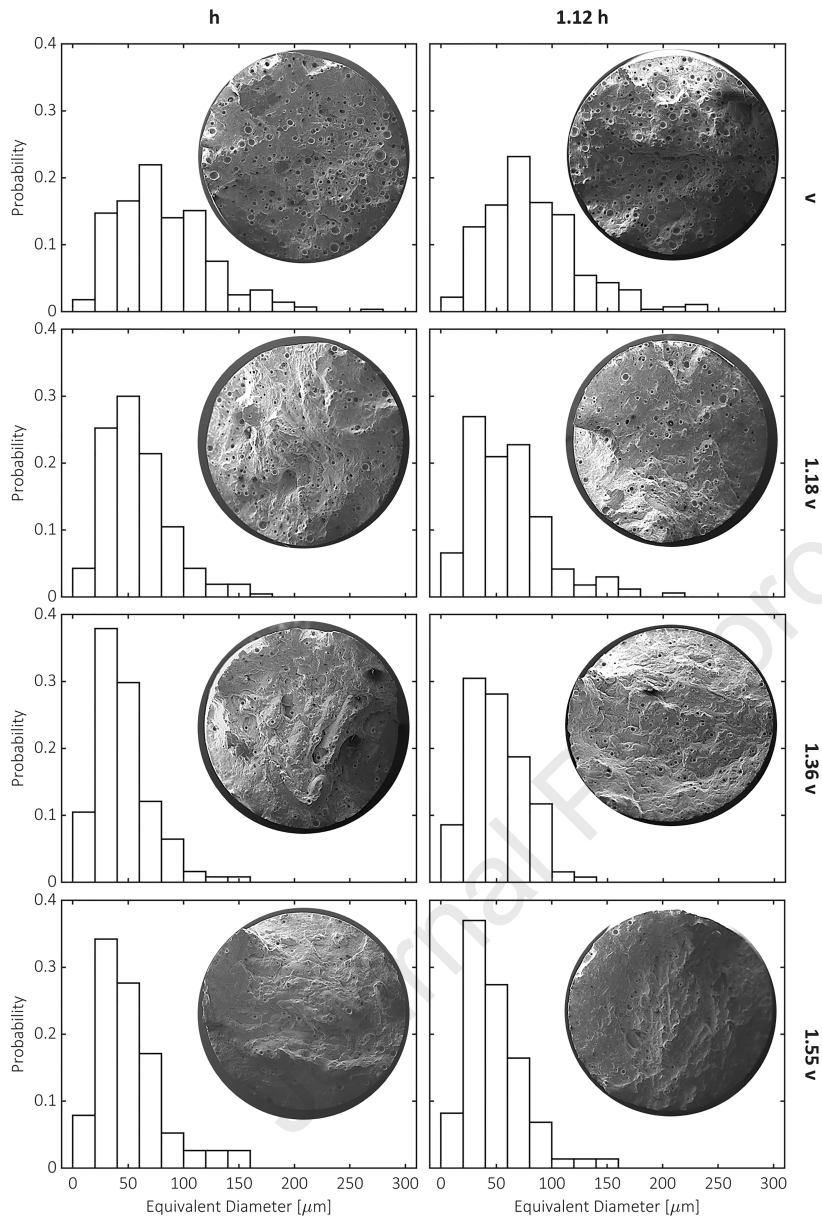18   (2019) 1–25. https://doi.org/10.1016/j.msea.2019.138210.

19

a

Relative Density [%]

Volumetric Energy Density [J/mm^3]

AlSi10Mg
+ [1]
▽ [2]
✕ [3]
◇ [10]
△ [12]
○ [16]
□ [17]
▷ [18]
◁ [20]
☆ [23]
✳ [24]
✩ [25]
▪ [34]
● [42]

AlSi10
◆ [22]

AlSi7Mg
▲ [19]
▼ [21]
■ This work

b

UTS [MPa]

Volumetric Energy Density [J/mm^3]
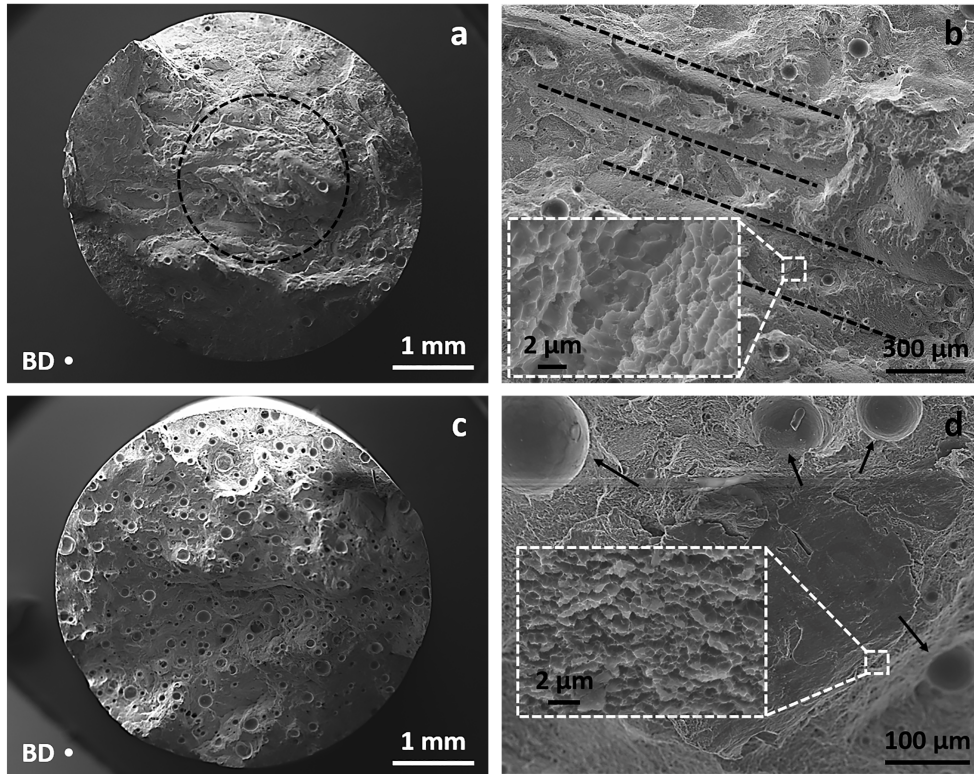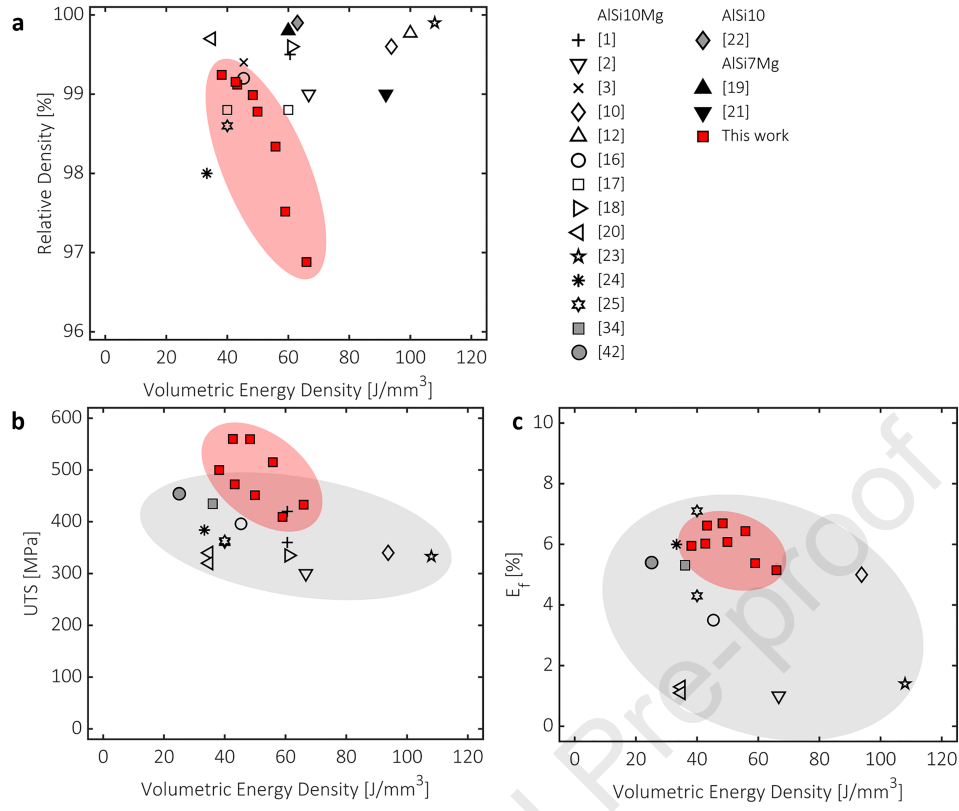
c

E$_f$ [%]

Volumetric Energy Density [J/mm^3]

Author contributions

MG performed the SEM and XRD analyses, GS performed the hardness tests and density measurements, MG and GS did the samples' preparation, OM observations and image analysis calculations. MTDG acquired the additively manufactured specimens and performed the tensile tests. MM supervised the work and acquired funding. MG wrote the first draft of the manuscript. All the authors contributed to and read the final manuscript.

Declaration of interests

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: