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MANUELA
Additive Manufacturing
using Metal Pilot Line

Deliverable D4.4

Material testing (1)

WP 4

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Terms and definitions

Acronym	Description
AM	Additive Manufacturing
LPBF	Laser powder bed fusion
EBM	Electron beam melting
SEM	Scanning electron microscope
EDX	Energy dispersive X-ray spectroscopy
EBSD	Electron backscatter diffraction
FIB	Focused ion beam
TEM	Transmission electron microscope
Det.	Determination
Hcp	Hexagonal close-packed
VED	Volume energy density
FWHM	Full width at half maximum
CHALM	Chalmers University of Technology, Sweden
POLITO	Politecnico di Torino, Italy
FAU	Friedrich-Alexander Universität Erlangen-Nürnberg, Germany
METAS	Federal Institute of Metrology, Switzerland
IVF	RISE IVF, Sweden

Executive Summary

MANUELA will provide an AM pilot line including many material testing methods. These methods will support the AM process development and the on-line monitoring development by covering the subject areas:

- microstructure analysis
- mechanical characterisation
- porosity determination
- controlling dimensional accuracy

Table 1 shows all the available methods with additional information about their goals, the testing interval and the sample characteristics. The testing interval provides a compromise between generating results and knowledge and increasing manufacturing costs by the testing effort.

Furthermore, the three printing facilities, namely CHALM, POLITO and FAU, defined benchmark samples. Thus, results from the different facilities are comparable.

1 Introduction

MANUELA will improve the two powder bed based AM processes LPBF and EBM. In addition, new on-line monitoring systems will be included. This way, MANUELA aims to set up a TRL 7 metal AM pilot line.

To achieve this goal, material testing is essential. The available material testing methods provided by the different facilities within MANUELA are summarized in this report.

Note, that the information about the material testing in MANUELA will be updated by a 2nd report in April 2021.

2 Summary of available material testing methods

Table 1 summarizes the available material testing methods in the MANUELA pilot line and provides information if the method is destructive or non-destructive, which facility can or will perform the testing, how often samples will be tested, which samples are feasible and the goal of the tests. To show this information clearly it is categorized.

Testing interval:

1. Per build job
2. Per process parameter
3. Per powder batch
4. Per material
5. Exceptional cases

Sample feasibility:

1. Use cases
2. Use case similar samples
3. Specific samples

Material testing goal:

1. Use case check
2. Process robustness check
3. Material / powder check

In these three categories, all options are ranked, indicated by their numbering. Superordinate options can include the subordinate options. For example, Table 1 shows, that the hardness tests will be performed after every build job on specific samples to ensure the process robustness and repeatability. Therefore, the hardness test can or will be used to check material properties (rank 3; lower rank than ‘use case check’) in dependence of different process parameters (rank 2; lower rank than ‘per build job’) but only using specific samples (rank 3; lowest rank in its category).

Table 1 shows the current state of the available testing methods. If e.g. special materials require more extensive analyses, these analyses will be performed and the table will be updated.

By using all these material testing methods, the results will heavily enhance the knowledge about the robust manufacturing of high quality parts using the LPBF or EBM process.

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Table 1: Material testing methods used in the MANUELA pilot line

Method	NDT / DT	Facility					Testing interval	Test sample	Reason
		CHALM	POLITO	FAU	METAS	IVF			
Computer tomography	NDT	No	no	no	Yes	no	per material	use case similar samples	material / powder check
3D scan	NDT	No	possible	possible	No	yes	per build job	use cases	use case check
Light microscopy	DT	Yes	yes	yes	No	no	per process parameter	use case similar samples	material / powder check
SEM	DT	Yes	yes	yes	No	no	exceptional cases	specific samples	material / powder check
EDX	DT	Yes	yes	yes	No	no	exceptional cases	specific samples	material / powder check
EBSD	DT	Yes	possible	possible	No	no	exceptional cases	specific samples	material / powder check
FIB	DT	Possible	possible	possible	No	no	exceptional cases	specific samples	material / powder check
TEM	DT	Possible	no	possible	No	no	exceptional cases	specific samples	material / powder check
X-ray diffraction	DT	Possible	yes	possible	No	no	exceptional cases	specific samples	material / powder check
Laser flash analysis	NDT	No	possible	yes	No	no	per process parameter	specific samples	material / powder check
Porosity det. by sample cross section analysis	DT	Yes	yes	possible	No	no	per build job	specific samples	process robustness check
Porosity det. by on-line monitoring	NDT	Yes	no	yes	No	no	per build job	use cases	use case check
Hardness test	DT	Yes	yes	yes	No	no	per build job	specific samples	process robustness check
Tensile test	DT	Yes	yes	yes	No	no	per powder batch	specific samples	material / powder check
Impact test	DT	Yes	yes	yes	No	no	per powder batch	specific samples	material / powder check
Resonance frequency	NDT	No	yes	yes	No	no	exceptional cases	specific samples	material / powder check

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3 Exemplary results

In the following section, we present three examples of material testing using methods available in the MANUELA pilot line.

3.1 Investigation of Ti6246

The Politecnico di Torino (POLITO) provided an investigation of the Titanium alloy Ti6246 using X-ray diffraction and light optical microscopy.

This analysis shows that the microstructure of as-built Ti6246 samples appears mainly martensitic, see Figure 1. This is quite frequent in α+β alloys that undergo intense cooling from high temperatures, as in LPBF. While in the more common Ti-6Al-4V martensite is almost certainly α' (hcp), Ti6246 contains a higher amount of β-stabilizing elements, hence α" (orthorhombic) formation is also likely. It is impossible to distinguish by means of optical microscopy between the two forms of martensite. Because of that, more in-depth investigations using different instruments (e.g. SEM, EBSD) will be performed in the future. The comparison of the images shows that the resulting microstructure of the sample built with a higher Volumetric Energy Density (VED), see Figure 1 (a), appears as thicker martensite needles, although a complete characterization is still lacking.

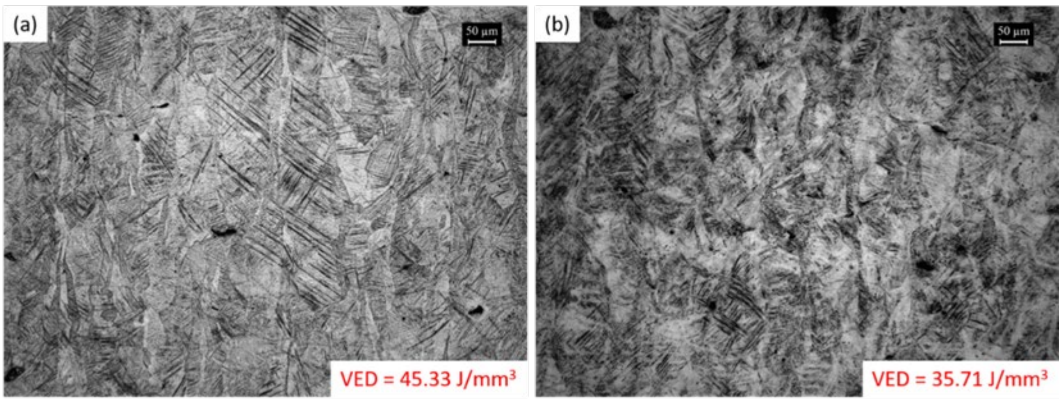

Figure 1 Light micrographs of Ti6246 samples characterized by a high value of VED (a) and an intermediate value of VED (B) (XZ plane).

X-ray Diffraction (XRD) measurements have been used to study the microstructure of AM powders and samples including the effect of the building parameters on the phase content.

The XRD patterns of Ti6246 powder and as-built samples with different parameters are reported in Figure 2. The resulting peaks correspond to a mainly α" microstructure. The bulk as-built samples show larger peaks (higher FWHM) with respect to the powder. This suggests smaller crystallites. This can be due to a significant number of phenomena, whose in-depth investigation is currently on going. Sample 1A, characterized by the highest VED value, shows an almost complete disappearance of the 53° peak (indicated with an arrow), which is instead clearly recognizable in the other samples. This result also needs further investigation.

Porosity is evaluated by image analyses taken at 50x using the Leica DMI 5000 on cubic samples cross sections. The porosity value is then calculated for each image using the software ImageJ.

The results obtained on the Ti6246 are plotted versus the volumetric energy density values in Figure 3.

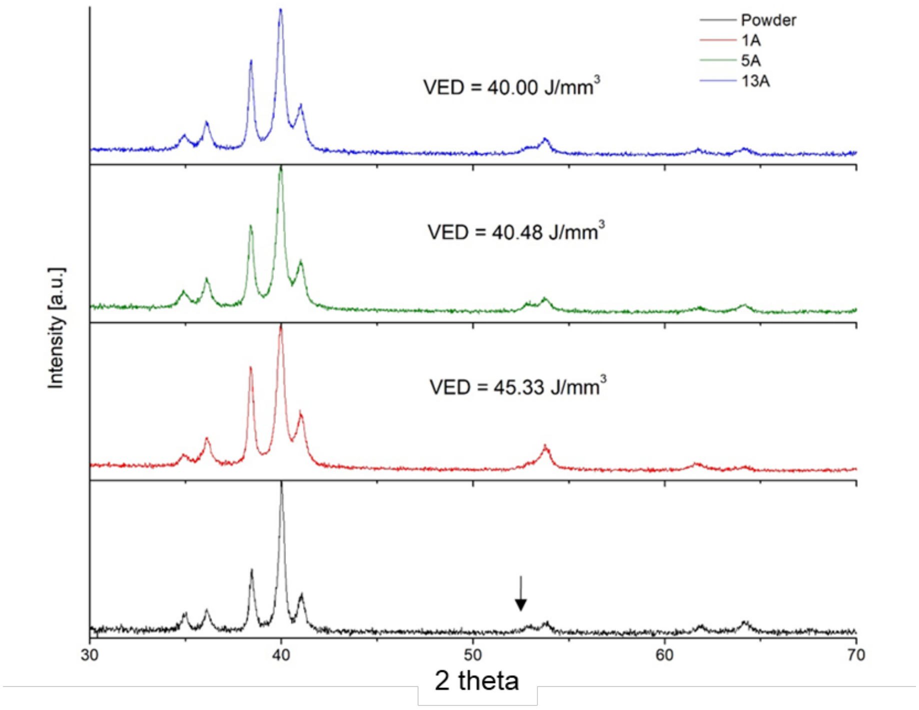

Figure 2 XRD pattern of T6246 powder and samples built with different VED values.

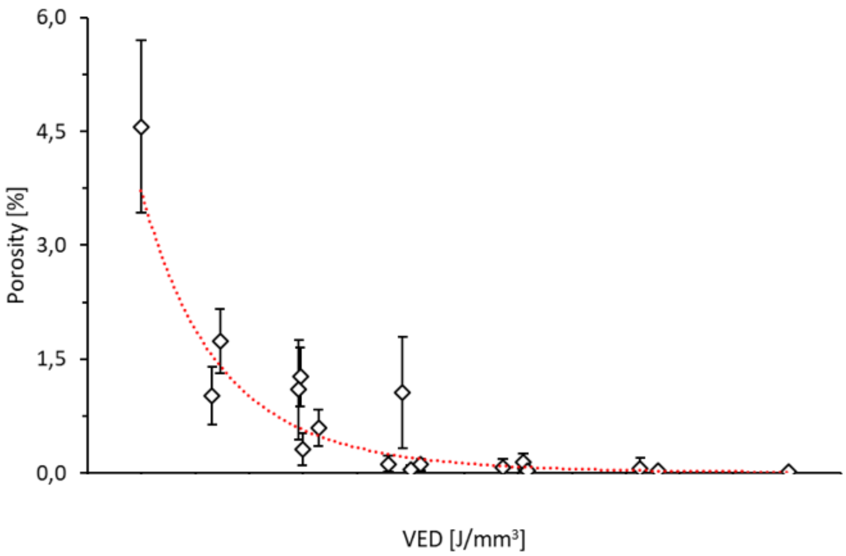

Figure 3 Porosity versus VED of Ti6246 samples.

3.2 In-situ electron optical observation

FAU uses an electron optical (ELO) observation for on-line monitoring during the EBM process. The ELO observation generates images of each layer directly after the powder melting. These images contain information about the porosity, melt surface features and the dimensional accuracy.

Figure 4 shows a test sample for the EBM process. During manufacturing a dense and a porous version of this sample, the ELO system generates images. Two of them are shown in Figure 5. The left image shows the dense sample. On the right side, there is the porous sample, apparent due to the dark spots within the sample. Therefore, the ELO observation enables in-situ pore detection.

The report D4.3 'Development and calibration of the on-line process monitoring for material of interest (1)' describes the ELO system more detailed.

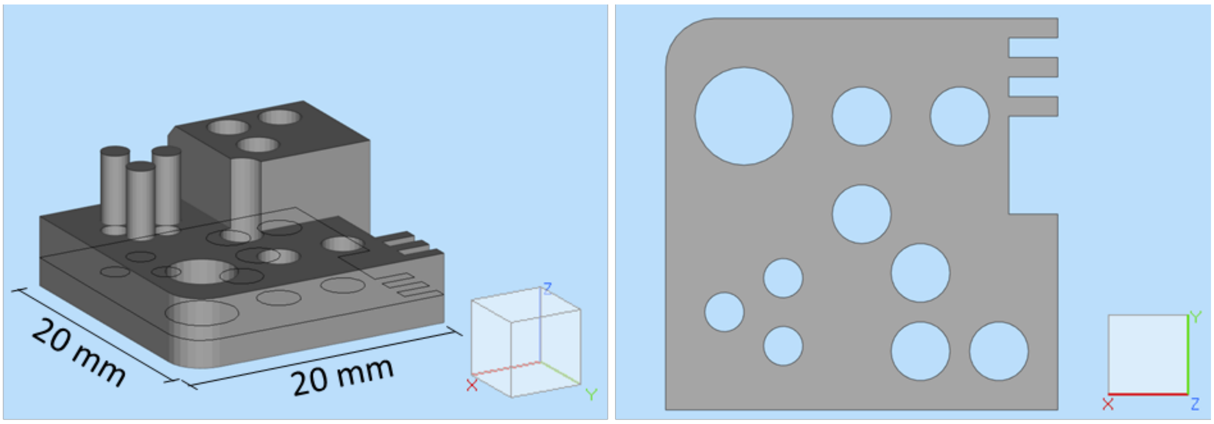

Figure 4: Test sample for the EBM process with implemented on-line monitoring using the ELO system. Right: Cross section of the test sample.

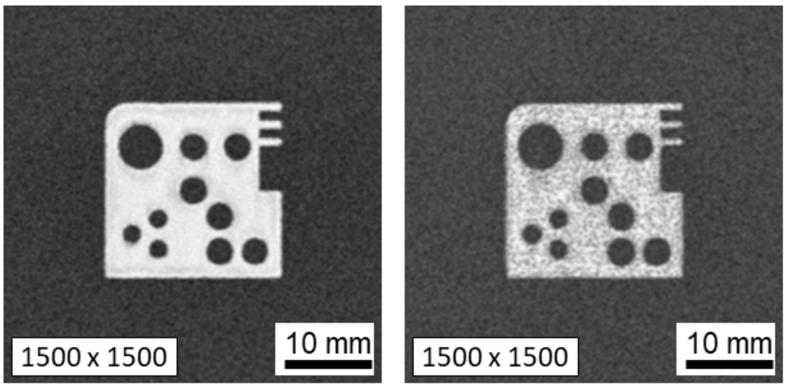

Figure 5: ELO image of a dense (left) and a porous (right) test sample. Resolution: 1500 x 1500 pixels.

3.3 DoE approach, LBPF process monitoring and investigation of 316L

Chalmers uses DoE approaches as well on-line process monitoring to set optimised processing parameters for laser powder bed fusion, today facilitated on the EOM290 machine. Also, the process monitoring is used for assessing manufacturability with respect product design (e.g. overhangs, geometries, etc) as well as quality assurance when manufacturing several parts of different kind or in repetition.

Further information on on-line process monitoring system is found in report on deliverable 4.6 "On-line system calibrated and tested". In any case, an important stage is always to manufacture test specimens of different kind to reassure the mechanical performance and benchmark against data for standards(e.g. ASTM A240M-18 for tensile testing), combined with range of material characterisation techniques including:

- Optical microscopy to check for porosity/relative density
- Scanning electron microscopy for assessing microstructure/melt pool characteristics
- X-ray diffraction for determining phase and their characteristics
- EBD to determine grain characteristics, texture effect, etc
- Surface chemical analysis (XPS, Auger) to check for surface chemistry of powder and fracture surfaces of tested specimens, etc

Some examples when applied to 316L stainless steel are illustrated below (Figure 6). One important notice is that is advisable to always fabricate and test specimens with as-fabricated surfaces to properly assess the mechanical properties (Fig 7). Also, It is important to assess the role of specimen thickness, since from a generic point of view a thin specimen will have different mechanical properties owing to its dimensions irrespective to how its fabricated either being additive manufacturing, sheet metal forming, or any technology.

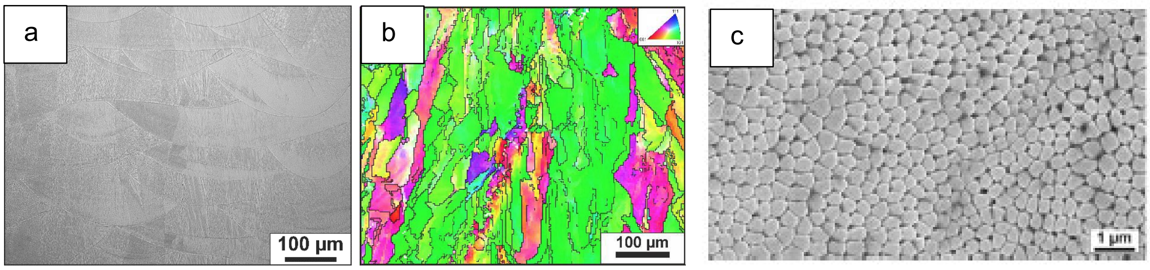

Figure 6: LPBF-processed 316L (99.99% density): a) optical image of cross-section along buld direction showing melt pools, b) EBSD image of cross-section along build direction illustrating crystallographic grains with orientation information, c) close-up SEM image showing subgrain/cell structure typical for LPBF-material, here showing cross cells view (Leicht et al, Chalmers).

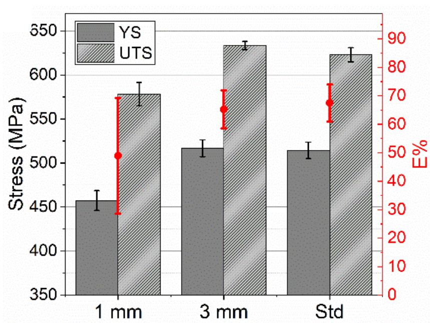

Figure 7: Illustration of mechanical properties of LPBF-fabricated 316L specimens with different thickness incl. standard specimens (ASTM A240M-18), (Leicht et al., Chalmers)

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4 Benchmark samples

To provide comparability between the results of the different manufacturing facilities using different AM processes, the benchmark samples, shown in Figure 8, will be built and analysed.

The influence of the scan length will be determined through the different sizes of the cuboid samples. The samples a, b and c will also show the possibilities and limits of building thin walls. The sample g will provide information about the possible cavity size and their shape fidelity. The samples h and i complete the investigation of the typical build direction dependent surfaces.

The printing facilities, will also fabricate method specific test samples.

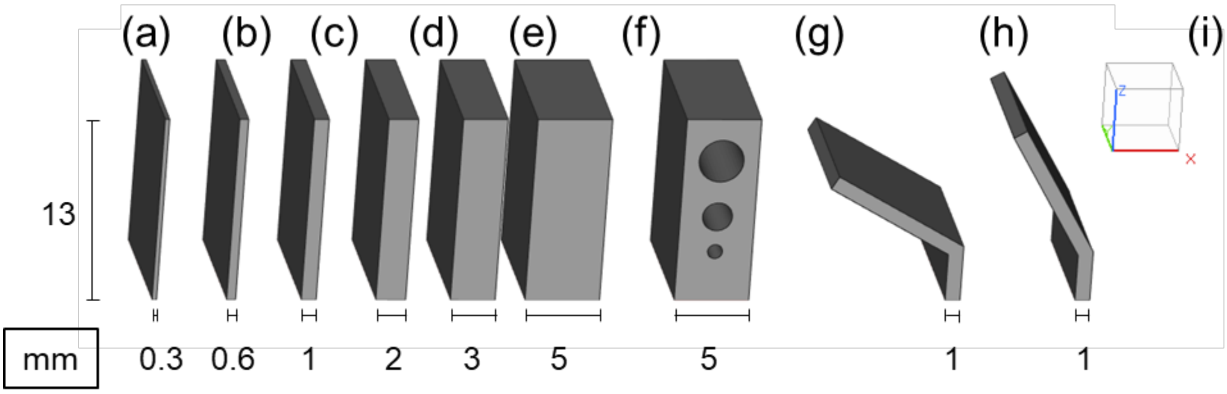

Figure 8: Benchmark samples for the MANUELA pilot line.

5 Correlation between material testing and on-line monitoring development

The material testing supports the on-line monitoring by proofing its results. Later on the on-line monitoring will replace some testing methods resulting in lower effort to check the part quality.

In case of EBM, the comparison of the ELO images with the well understood results and conclusions of the conventional methods will benefit the ELO results and their interpretation. This comparison will clarify the potential, show limits and provide a first possibility for the on-line monitoring calibration.

Information about the on-line monitoring during the LPBF process is provided by the report D4.6 ‘Online monitoring systems calibrated and tested’. With the application of monitoring systems, development of processing windows to create for example higher productivity can be done and then test samples to verify mechanical properties constitute and important part of the approach.

6 Correlation between material testing and AM process development

The available material testing methods cover many subject areas, namely microstructure analysis, mechanical characterisation, porosity determination and dimensional accuracy. Furthermore, the correlation between material testing results and on-line monitoring also supports the process development. With an usable on-line monitoring system, many process development steps can be achieved much faster. Hereby, the effort for post manufacturing characterisation will become reduced.

7 Conclusions

This report highlights the wide range of the available material testing methods. These methods will support other tasks within MANUELA by correlation with the AM process development and the on-line monitoring development. Furthermore, material testing will expand the knowledge about powder bed based AM and related material properties distinctly. Consequently, a large amount of data will be acquired and incorporated in the material property database, which is part of the MANUELA pilot line.

The exemplary results described in this repot indicate the possible improvement of processing materials already in use and the integration of new materials into the MANUELA pilot line.

Testing intervals for each testing method are determined considering the increase in manufacturing cost. Therefore, the MANUELA pilot line will deliver improved AM processes at competitive product costs.