Good Practice Guide

For the correct choice of characterisation technique depending of the level of accuracy needed and the type of measurement required.

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1 Introduction

With the ability to produce patient specific medical devices on demand, the Additive Manufacturing (AM) technology is of growing interest for the medical sector. As the technology developed at a much faster pace than regulations or quality control, there is currently a lack of validated techniques to verify the finished parts. The intention of the MetAMMI project is to diminish these gaps and to increase confidence of the medical device industry in the AM technology.

In the frame of the MetAMMI project, various dense and lattice parts have been manufactured using different AM technologies and different materials. The parts have been characterised and typical geometrical deviations have been identified as well as their origin in the process chain. Based on these information, validated techniques for the identification of typical deviations have been investigated.

The present Good Practice Guide summarises the results of the investigations. It first provides a short overview of the characterisation techniques investigated with a brief description of the measurement principle, as well as the advantages, disadvantages, accuracy, sample requirements, and efficiency. Then, for each type of measurements required (mechanical testing, microstructural characterisation, and defect detection), recommendations are given based on the results from MetAMMI. Finally, the findings for defect detection are summarized in an overview table allowing for characterisation technique selection based on the required measurement type and the needed level of accuracy.

2 Characterisation techniques investigated

This section introduces the characterisation techniques employed throughout the MetAMMI project. For each, a brief description of the measurement principle is given, as well as a short overview of the advantages, disadvantages, accuracy, sample requirements, and efficiency. The methods are grouped by the type of analysis they can deliver, *i.e.*, volumetric, surface, density & porosimetry, and others.

2.1 Volumetric methods

2.1.1 XCT

Computed tomography is a well-known X-ray-based imaging technique able to acquire and display the complete 3D measured object with one single scan [DEC14]. The volumetric image is obtained by an image reconstruction of several 2D images (*i.e.* projections) acquired from several angular positions of the object. The 2D images represents the attenuation of the X-rays by the object. The reconstructed volume model is composed by image elements called voxels (*i.e.* volumetric pixels), each voxel has a grey value representing the local X-ray attenuation of the object.
A CT system consists of three main components: X-ray source, manipulation system and the X-ray detector. In contrast to the medical CT systems, in the industrial and metrological CT systems the object is rotated by a rotary stage instead of the X-ray source and detector. This configuration provides better mechanical precision; therefore, it delivers better metrological results.
For the data analysis, *e.g.* nominal/actual comparison, the object surface must be determined. The surface determination routines implemented in the state-of-the-art CT data analysis software are based on the local grey level gradient of the voxels, which are close to the surface. The determined surface is the basis for the nominal/actual comparison. The nominal/actual algorithm searches the closest actual surface (surface determined by CT) perpendicular to the nominal surface (from the CAD data or stl) and measures the distance between the nominal and actual surface. An alignment of the object (translations and rotations of the object in the 3D space), to guarantee that both nominal and actual object are in the same position and orientation is necessary. An alignment procedure commonly used is called best-fit registration.
Advantages: Full 3D characterization with one CT scan, non-destructive technique, precise measurements when compared to the medical CT scans, big flexibility regarding object materials, shape and surface quality. Disadvantages: long data processing times, big datasets, potentially harmful radiation – shielding required (not relevant for mechanical parts), measurements not fully traceable to the meter, measurements not as precise as the tactile measurement systems.
Resolution/ Accuracy: minimum spot size around 3 µm. Sample sizes in a range of 500 µm up to 20 cm possible, lattice and dense structures can be measured and any material. Limitations only for metals. E.g. maximum sample thickness to measure steel is about 20 mm. For parts with sizes as fabricated within MetAMMI the mean measurement time is around 1,5 hours; costs are around 350 € per hour.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
- Full 3D characterization - Non-destructive technique - Flexible technology	- Large datasets - not traceable measurements - Long scanning time	Simple measurement task, *e.g.* sphere center distance measurement error of below 5 µm.	Big range of materials from plastics to approx. 20 mm of steel	Flexible concerning object geometry	Average of scan time is around 1,5 h	Approx. 350 €/h

2.1.2 Terahertz computed tomography

Terahertz waves are electromagnetic waves in the frequency range from 0.1 to 10 THz, with a wavelength from 30 µm to 3 mm, they are between microwaves and infrared. Terahertz waves penetrate various dielectric materials, including plastics, ceramics, crystals, and concrete, allowing terahertz transmission and reflection images to be considered as a new imaging tool complementary to X-rays or infrared. Terahertz imaging is a well-established technique in various laboratory and industrial applications. However, these images are often two-dimensional. Three-dimensional, transmission-mode imaging is limited to thin samples, due to the absorption of the sample accumulated in the propagation direction. A tomographic imaging procedure can be used to acquire and to render three-dimensional

images in the terahertz frequency range, as in the optical, infrared or X-ray regions of the electromagnetic spectrum.
Terahertz computed tomography presents new potential because it can measure amplitude as well as spectral phase information in comparison to X-ray imaging. With Terahertz CT, it is not utopist to identify or compare different substances and localize them in a nondestructive manner. [GUI14]
Terahertz techniques are mostly suitable for polymer and ceramic parts, but not for metallic parts, as metals reflect THz waves. The analysis can be performed either in 2D or in 3D depending on the experimental set-up. Depending on the frequency range, resolution of single points (defects) with 1mm accuracy is possible.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Low cost Non ionising Sensitive to low density material	Data processing is complex	10 µm up to 20 mm	No metal Non conducting materials No polar molecules	Plane parts are better Few mm up to 30 cm	2 h – few days	500 € per measurement

2.1.3 Infrared thermography

Infrared thermography is a well-known method for contactless temperature measurement. The principle is based on the property of every object to emit electromagnetic radiations at temperatures above the absolute zero point at 0 Kelvin. The emitted radiation is based on the mechanical movement of atoms and molecules and is invisible to the human eye (below certain temperatures) but can be measured via infrared sensitive detectors. For non-destructive evaluation, it is necessary to destabilise the object thermal state through heating or cooling, the technique is then referred to as *active thermography*. The presence of an internal defect reveals itself on the surface as a temperature perturbation above this defect.
The main advantages from the technique are the fact that it is a well-known technology and often used for in-process control, in real time. The technology can be applied to both small and large components, it is relatively unaffected by the object's geometry, and well adapted for the inspection of large surfaces. There are many different types of thermography set-ups and advanced signal processing techniques which can yield a wider range of information about the sample. The main disadvantages from thermography are its sensitivity to the object's surface condition and thickness, to water and dirt. The response time of the detector has to be adapted to the thermal conductivity of the part material, *e.g.*, for metals, the thermal conductivity is high, which requires fast acquisition times. The thermography results will be sensitive to the heat source, its type, the duration of heating, and its location from the sample.
The detectable deviations include dimensional, geometrical variations (distortion, warping, shrinkage), and bulk defects (cavities, delamination). However, quantitative results are only in specific and individually calibrated cases possible. Bulk defects can only be detected if they are close enough to the surface and have a sufficient size (roughly, depth in the sample must not be larger than the defect size).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Very fast Non-intrusive Non-contact	Quantitative results only in specific cases possible	Depending on material and geometry. Well known and homogeneous thermal material parameters and needed	Any, but surface finish must be appropriate, coating might be needed for optical heating sources	Any, but geometry will limit the amount of results	Depending on material, <1s for thin thermally well conducting materials to >10min for thick, thermally low conducting materials	Depending on material and part geometry (choice of camera), few k€ (thermally low conducting materials, low spatial resolution needed) to >100k€ for thermally well conducting materials

2.1.4 Ultrasonic testing

Ultrasonic testing (US testing) is based on the propagation of ultrasonic waves in the object or material that is being tested. An ultrasound transducer is connected to the surface of the sample by a couplant (water, oil or gel) and generate ultrasonic waves that propagate through the sample. When reaching a flaw or a surface, the propagation of the ultrasonic waves is perturbed, *i.e.*, the waves are reflected, and this results in a signal being detected back by the ultrasound transducer. As acoustic waves propagate in different media with different velocities, US testing is especially suitable for homogeneous materials. The distance to the defect from the sample surface can be estimated from the propagation velocity and time. Ultrasonic testing allows the detection also of small defects below 1 mm, and the shape of the defect can be determined by scanning the object under different angles.
The advantages of US testing include high penetrating power, high sensitivity, capability of estimating the size, orientation, shape and nature of defects, and the results are immediate.
The disadvantages of US testing are due to the fact that parts that are rough, irregular in shape, very small or thin, or not homogeneous are difficult to inspect. Couplant is needed to provide an effective transfer of ultrasonic waves between the transducers and the part being inspected. Finally, extensive technical knowledge is required for the development of inspection procedures.
The detectable deviations include dimensional variations and bulk defects (cavities, delamination).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Results are available immediately	Requires couplant Requires good surface condition	Depends on frequency and penetration depth. Can reach 50 µm	Any	Dense and flat Good surface condition is required	> 1 hr	150 €/hr

2.1.5 Resonant acoustic testing

Resonant acoustic testing is a global volumetric comparative method. It consists in measuring the component's mechanical resonances in air and to compare them to the mechanical resonances of a reference components defined as a "good part". A shift in frequency will be observed in the mechanical resonances of a "bad part". The method enables to detect the component's structural characteristics: flaws, process variations in manufacturing of the component, etc., anything that will cause a shift in the mass, stiffness, and damping of a component can cause a shift in the resonant frequencies. The method is dedicated to serial production.
In the method, the excitation of the part, to make it vibrate, is done by an impact hammer and the resulting acoustic frequency data is recorded with a microphone. The output of the microphone is converted into a frequency spectrum using a high-speed analog to digital converter performing a fast Fourier transform to determine resonant peaks. Finally, a statistical analysis conducted with a software reveals if the test parts fall within the defined criteria or not.
The detectable deviations include dimensional and geometrical variations (distortion, warping, shrinkage) and defects (void, cracks, missing features, porosity).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Very simple, and fast method Enable inspection of any shape (lattice) and size parts	No localization of defects Statistical method which require reference parts (the more , the best)	Pass/fail test. The sensitivity of the method can be probably increased using signal processing algorithms	any	any	Few minutes	40k€-90k€ for a set-up

2.2 Surface methods

2.2.1 CMM

A coordinate measuring machine (CMM) is a system that measures the geometry of physical objects by recording the spatial coordinates of discrete points onto the physical object surface with a probe. In the present section, tactile CMM refers to systems using a mechanical probe, by opposition to other CMM systems using optical, laser, and white light (dealt with in sections 2.2.2 and 2.2.3). CMM systems typically specify the probe's position in terms of its displacement from a reference position in a three-dimensional Cartesian coordinate system. In addition to moving the probe along the X, Y, and Z axes of that Cartesian coordinate system, many systems also allow the probe angle to be controlled to allow measurement of surfaces that would otherwise be unreachable.
Depending on the machine, the probe position may be manually controlled by an operator or it may be computer controlled. The data collected from the part being measured needs to be aligned with either the component drawing or a computer-aided design (CAD) model. This alignment is usually carried out with reference to defined datum features on the drawing. However, for complex-shaped components, such as medical implants, a best-fit alignment may be more appropriate.
CMM are very accurate and can provide traceable measurements. On the other hand, the time required per each surface point is very long and the points to be measured (measurand) needs to be defined before the analysis is performed.
The detectable deviations include dimensional, geometrical variations (distortion, warping, shrinkage), and surface defects (uneven surface, staircase defects and point defects).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Very accurate Traceable measurements	Slow Measurands to be defined before the measurement Low point density	0,1 – 10 nm	any	Dense, 0.3 – 120 mm	> 2 h	

2.2.2 3D laser scanning

A 3D laser scanning system is technically a coordinate measuring machine using a laser source as the probe. All the general comments made in section 2.2.1 thus remains valid. 3D laser scanning is an active method, meaning that either a laser line or the object needs to move relative to one another. A thin laser line is projected on the object and the reflection is recorded by a camera where the recorded values of light intensity are transformed in 3D measurement values.
The set-up of such system is easy, the system is usually transportable and suitable for large object. The data acquisition can be fully automated and provide an absolute measurement of the outer geometry. On the other hand, the use of a laser means that mat surfaces are required, and surface with diffuse reflection, glass and blank metal are problematic. Such surfaces can be covered with a thin layer of chalk to reduce measurement uncertainties, but it is not the preferred method as it contaminates medical implants.
The detectable deviations include dimensional, geometrical variations (distortion, warping, shrinkage), and surface defects (uneven surface).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Contactless measurement Measurement of the complete surface.	Deep holes / undercuts not measurable. Surfaces may have to be pre-treated.	0.03 mm for small/ medium objects	Any, if surface is scannable	Any	< 1 h	150 €/h

2.2.3 Fringe projection

A fringe projection system consists of at least one projector which projects stripes onto the object surface and one camera which captures the reflected fringes. Often several cameras are used to avoid gaps caused by shadows on the surfaces. By determining the distance of the surface to the sensor by triangulation calculations, a point cloud of the surface can be obtained. Connecting the surface points to a surface model allows an analysis of the model such as nominal-actual-comparison can be performed.
The advantages of fringe projection method are the relatively fast and contactless measurement with a high point density. Relatively large components can be measured by combining several data sets using for example reference points. The set-up is compact and transportable. The disadvantages are identical to many other optical measurement methods: The measurement depends very much on the surface properties of the object to be measured. Highly directionally reflecting surfaces lead to overexposed areas in the camera image. In the case of transparent objects, the fringes are reflected inside the component and thus lead to a deviation of the measurement results. In addition, the system is limited by geometric features such as holes or undercuts.
By calculating the surface shape of the object, geometrical variations such as distortion, warping, shrinkage effects can be calculated. The detectable deviations also include dimensional variations, and surface defects (uneven surface).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Contactless measurement Measurement of the complete surface.	Deep holes / undercuts not measurable. Surfaces may have to be pre-treated.	Ca. 0,003 mm for small measured areas 75 µm	Any, if surface is scannable	Any	< 1 h	150 €/h

2.2.4 Focus variation

Focus variation microscopy is an optical microscope method that allow the reconstruction of the three-dimensional surface structure from sets of image plans obtained by detecting the horizontal level where the surface is in focus [ISO25178-606]. It takes advantage of a small-depth of focus and combines it with a vertical scanning of the objective to reconstruct the topography. Focus variation can be used both for measuring the 3D shape of an object as well as its surface roughness [DAN11].
The advantages of focus variation compared to confocal microscopy is that there is less shadowing effect (cavities and steep edges measurable), it is suitable for reflective surfaces with high roughness and steep edges (which are problematic to measure with most other surface technologies). It is not so sensitive to varying reflection properties of the material, and the set up can be compact and transportable. The resolution can be very high, with vertical resolution down to 10 nm within small xy range of 0.14 mm. Regarding the disadvantages, the distance between the optic and the measured object must be small (from a few millimeters to 20 mm or more), and the measurements are only relative and not absolute (calibration is needed). The fact that some parts of the set up are moving means that vibrations can be a problem. It is not suitable for samples that do not have enough variation of contrast, such as transparent and low roughness (Ra below 10 nm) samples. A trade-off between resolution and xy range has to be made. For 3D printed rough surfaces, low magnification lens has to be used, which means that vertical resolution is on the order of 100 nm. The detectable deviations include mainly surface defects (uneven surface, surface texture, staircase effects and point defects), but can also include dimensional variations.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Simple, versatile, not so expensive, suitable for steep slopes	Trade-off between vertical resolution and xy range / working distance, not for transparent and low roughness surface	Vertical resolution down to 10 nm for 0.1 mm xy range, lateral resolution is diffraction limited	Almost all material	Surface measurement from 0.1 mm to centimeters	From minutes to one hour	10 to 100 K€ for a set-up

2.2.5 Confocal microscopy

Confocal microscopy is an optical microscope method that allow the reconstruction of the three-dimensional surface structure from sets of image plans obtained by accurately detecting the horizontal level where the backscattered light alone has a maximum intensity. To detect the backscattered light alone, the light beam emitted by a light source passes through a *light source pinhole* and is then focused on the surface with an objective lens. The beam reflected from the surface is then sent back to a detector (generally a photo-detector) through another *discrimination pinhole* which only transmits the light focused on the pinhole and not the out-of-focus light surrounding the pinhole. The detector will receive the maximum light intensity when the light beam is in focus on the surface. The optical system is thus characterized by two conjugated pinholes, therefore the name confocal microscopy [ISO25178-607].
The advantages compared to *e.g.* focus variation microscopy is a higher resolution both horizontal and vertical and from a physical point of view the working principle is more basic and well defined giving a well-defined observed height for each pixel in the image. The accuracy can be very high, with vertical resolution down to several nanometers for xy range of 0.15 mm. Both roughness and topography can be measured. Regarding the disadvantages, the distance between the optic and the measured object must be small (from a few millimeters to 20 mm or more), and the measurements are only relative and not absolute (calibration is needed). The fact that some parts of the set up are moving means that vibrations can be a problem. Confocal microscopy performs the scan point-by-point and is typically slow. Modern confocal microscopes have significantly improved scanning speed (up to 10 frames per second for conventional confocal and 100 frames per second for spinning disk). On some surfaces the sensor is not able to assess the height position on all points on the surface giving “non-measured points” (missing data) or outliers. Possible reasons for the missing data are dark surfaces, shiny surfaces or steep slope. Both resolution and maximum slope angle are dependent on the numerical aperture (NA) of the objective. To achieve high resolution or resolve steep slopes, high NA objectives, which often have small xy range and short working distance, have to be used. This brings significant practical limitations for rough surfaces generated by 3D printing. In general, low values of NA below 0.4 (equivalent to 20x lens with field of view (FOV) of 0.5 mm, lateral resolution of 300 nm, and vertical resolution of 20 nm) are not suitable for roughness measurement.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
High lateral and vertical resolution	Not suitable for steep slopes or deep holes Trade-off between resolution/slope and xy range / working distance Expensive	down to several nanometers vertically and 150 nm laterally for xy range of 0.1 mm	Almost all material	Surface measurement from 0.1 mm to centimeters	From minutes to one hour	40 to 400 K€ for a set-up

2.2.6 White light interferometry

Aka coherence scanning interferometry or vertical scanning interferometry, white light interferometry (WLI) instrumentation consists of an interference objective integrated with a microscope. Broadband light is directed to the objective lens, where the light is divided by beam splitter. One beam is directed to the sample and the other beam to a reference mirror. The two beams are recombined and directed to the camera detector. During the vertical scanning, the detector measures the light intensity as the optical path is varied. Interference maximum at each pixel is detected and the fringe envelope is used to calculate the position of the surface. The advantage is that resolution can be very high (down to sub-nanometer, although the accuracy depends on material), and low reflectance surface (well below 1 %) can be measured. Another advantage compared to confocal is that vertical resolution is not dependent on NA of the lens so objective with large FOV can be used without compromising resolution. Regarding disadvantages, it is sensitive to vibrations, and it is not suitable for all types of surfaces. Surface with high roughness and steep slopes can pose problems, similar to confocal microscopy. Dropouts and errors can occur due to scattering, low intensity, and interactions of neighboring pixels.

Advantages	Disadvantages	Accuracy	Sample		Efficiency	
			Material	Geometry	Time	Costs
High resolution, vertical resolution not dependent on NA	Not suitable for very rough and high gradient surface, dropouts or errors due to complex reasons	Vertical resolution down to subnanometer, lateral resolution is limited by Abbe criterion (up to 250 nm)	Almost all material	Surface measurement from 0.1 mm to centimeters	From minutes to hours	40 to 400 K€ for a set-up

2.2.7 Confocal chromatic probe instrument

The principle, advantages and limitations of confocal chromatic microscopy is similar to confocal microscopy discussed above. However, the resolution is not quite as good, and the price are a little lower. The operating principle is based upon the chromatic dispersion of the white light source along the optical axis, via a confocal device, and the detection of the wavelength that is focused on the surface by a spectrometer [ISO25178-602].
The advantage is that the system can be designed as remote sensor which is coupled to optical fiber for illumination and analysis optics. The flexible set up is portable and suitable for dirty or hard-to-reach areas. Another advantage is the freedom of depth discrimination is allowed by choosing a lens glass type with appropriate dispersion. The system can be built to have either small working distance with high depth resolution, or large working distance with lower resolution. Disadvantage is that chromatic sensors are limited to single point measurement, and resolution is not as good as confocal microscopy.

Advantages	Disadvantages	Accuracy	Sample		Efficiency	
			Material	Geometry	Time	Costs
Remote sensor is highly portable and flexible, working distance and resolution can be tailored	Point measurement, area scan is very slow	Down to a few nanometers	Almost all material	Not suitable	Seconds	30 to 300 K€ for a set-up

2.2.8 Stylus instrument

The stylus instrument is used for the metrological description of surfaces. Depending on the measuring section, roughness, waviness and form deviations can be measured [KEF11]. In this contacting method, the surface is scanned with the aid of a diamond tip. Due to the surface contour, the probe moves with an almost constant speed vertical to the traversing length over the surface to be tested. The motion is converted into a continuously varying electrical signal which is amplified, digitized, filtered and stored as primary profile (P-profile). For roughness measurement, the diamond tip has a conical shape with a radius of 2 µm, 5 µm or 10 µm, angle of 90° or 60° and a static measuring force of max. 0.8 mN [VDI2602]. However, the radii of the probe tip act as mechanical filters and the geometry of the probe tip determines the limit of the detectable roughness. In addition, the measurement result can be strongly influenced by vibrations. Therefore, measurements during productions are often made with a skid-mounted tracing system. This system has a smaller measurement loop which is less sensitive to vibrations. Overall, the stylus profile method is a good technique to determine the surface properties of AM parts with a high resolution over a relatively long evaluation length.

Advantages	Disadvantages	Accuracy	Sample		Efficiency	
			Material	Geometry	Time	Costs
High resolution over a relatively long evaluation length	Mechanical filtering and strongly influenced by vibrations			Any outer surface	Depending on the measuring task	< 100 € (simple profile measurements)

2.3 Density & porosity

2.3.1 Archimedes' method

Archimedes' method is global volumetric method that allows the determination of volume and density. Defects can be detected by comparing to density values of a good reference part. The method is more appropriate for serial production. The system consists of a precision balance with an inferiorly attached thin wire at the end of which a suspension device is hooked. This suspension device is immersed in a container, filled with a liquid, and placed beneath the balance. The classical principle of the hydrostatic method involves measuring the apparent mass of the sample in air, then in twice-distilled water to deduce its density. However, in the case of lattice structures, water bubbles form at the interface air-lattice preventing the water to penetrate deep inside the lattice since the surface tension of water is too high (72.8 10^{-3} $N.m^{-1}$ at 20°C). In order to avoid this phenomenon, which distorts the measurements, absolute ethanol is used.
Archimedes' method is suitable for dense and lattice geometries but requires a reference part to detect defects. Assessment of percentage of lattice cell by comparing dimensional and Archimedes' measurements of the volume; Verification of compliance with part specifications; Characterisation of AM material by comparison (internal porosity quantification); Evaluation of the repeatability/reproducibility of AM processes by comparison. Although the measurement time is quite fast (30 min per sample), the preparation time is very time consuming as it is approximately half a day.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
cheap	No localization of defect	Depend on the balance accuracy	any	any	30 min/ measurement	Less than 20k€ for a set-up

2.3.2 Gas pycnometry

Gas pycnometry is a global volumetric method that allows the determination of volume and density. A pycnometer consists of two cells: a sample cell and an expansion cell which are connected to each other via a pipe which can be closed by a valve. The principle of the density measurement with a gas pycnometer is the following:
1) The device under test is placed in the pycnometer sample cell at the initial pressure of P_{atm}. The valve is closed.
2) The pressure is increased until P_1 in the sample cell.
3) The pycnometer valve is open.
4) The pressure P_2 is measured when the equilibrium between the two cells is reached.
5) The apparent mass of the sample (m) is measured with a precision balance.
6) The density is then calculated using:

$$\rho = \frac{m}{V_{\text{sample}} + V_{\text{expansion}}\left(\frac{P_2 - P_{\text{atm}}}{P_2 - P_1}\right)}$$

In terms of advantages, the pressure measurement accuracy is high at around 0.1 %. The accuracy for density measurement depends of the size of the sample cells but is never higher 5 %. In terms of disadvantages, the technique is not suitable for big parts due to the limitation in size of the pycnometer chamber. The technique also gives only an overall density value of the part, which means that there is no information on the type of defect (pore or delamination), location and number (many small pores versus few large pores).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Fast and cheap	No localization of defect Less accurate than Archimedes Not suitable for big parts	Less than 5%	any	Small parts, less than 50 mm in diameter and less than 75 mm in depth	Fast 10 min/ measurement	15k€-20k€ for a set-up

2.4 Others

2.4.1 Gas permeability measurements

With a capillary flow porometer, the gas permeability of porous materials can be measured. During the measurement, an inert gas flows vertically through the porous sample at default pressure and at constant temperature. The test runs with an elevated inlet pressure relative to the outlet pressure, which is kept at atmospheric pressure. The gas flow is measured as a function of time at constant pressure. A porometer set-up, previously planned for the analysis of mineral building materials, such as cementitious mortars and natural stones has been used within the frame of MetAMMI to investigate printed lattice parts. The measured parts were printed without and with certain intended defects.
The underlying principle is the Hagen-Poiseuille relationship for laminar flow of a compressible fluid through a porous solid with small capillaries under steady-state conditions. The relationship solved for the specific gas permeability coefficient K [m²] can be written as:

$$K = \eta * \frac{Q * L}{A} * \frac{2p}{(p_e - p_a) * (p_e + p_a)}$$

with the gas viscosity η, the measured volume flow rate *Q* in m³/s, the sample thickness parallel to the flow direction *L* in m, the cross-section area of the sample *A* in m², the default absolute pressure p in N/m², at which the volume flow rate is measured, the absolute gas inlet pressure p_e in N/m² and the absolute gas outlet pressure p_a in N/m² [GRÄ86, KOL89].
The gas permeability coefficient of the samples investigated varied randomly and did not enable reliable conclusion regarding included defects. Investigations are ongoing and further parameter studies as well as an improved sample holder might lead to more reliable results.

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
			Any	Porous parts only	Fast	

2.4.2 Weight measurements

A simple measurement of the final parts mass by weighing is one approach to identify defects in manufactured parts. It is a fast and inexpensive method, easy to integrate in the manufacturing process chain for the evaluation of the quality of a printed part. The expected mass (*m*) of the part can be calculated by the product of final density of the solid material (*ρ*) and the nominal part volume (*V*) retrieved from the corresponding STL files, when taking into account typical shrinkage for the material used:

$$m = \rho * V$$

The results from weighing of the printed part can be compared to the calculated mass or to the mass of a reference part. Investigations within the project showed, that weighing allows to identify defects leading to a change in volume and mass of around 20 % compared to a reference part. Also, the detection of minor defects is possible if the process is very stable (see also **deliverable D6**).

Advantages	**Disadvantages**	**Accuracy**	**Sample**		**Efficiency**	
			Material	Geometry	Time	Costs
Fast	No localization of defect	Depends on scale	Any	Any		

3 Type of measurement required

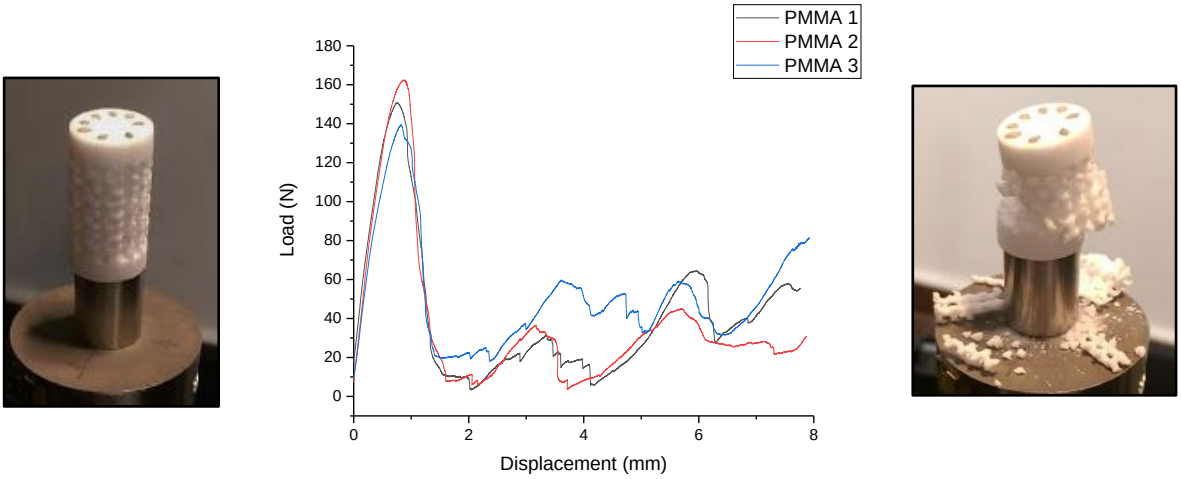
This chapter presents possible non-destructive measurement methods and their suitability to detect certain types of deviations in additively manufactured parts described before in chapter **Error! R eference source not found.**.
An analytical toolbox in chapter **Error! Reference source not found.** provides a quick overview on the d ifferent measurement methods investigated and for the detection of which defect category they are basically suitable. In addition, Table 1 allows to roughly compare possible accuracy, sample requirements and efficiency of the different measurement methods.

3.1 Mechanical testing

Mechanical testing is used to determine the mechanical properties of interest of a material or to determine the mechanical behavior of a structure under specific loading situation.

In the first case, mechanical testing is generally used to obtain geometry-independent mechanical properties. There exist a large number of tests available, many of which are standardized. For example tensile testing [ISO6892, ASTMD3039, ASTMD638, DIN50125], compressive testing, 3-point bending test, fatigue testing.
In comparison to conventional manufacturing, orientation-dependent differences in the mechanical properties of additively manufactured parts represent a process-specific property. For the tests on CoCr samples, cylinders with a diameter of 9 mm and a total length of 90 mm were used as blanks for the tensile test specimens, which were then machined according to DIN 50125. Specimens were manufactured at an angle of 45° (oblique), 0° (parallel) and 90° (perpendicular) to the base plate and tested using a Messphysik Beta 200-4/6x16 system.

In the second case, the entire structure is placed under loading and its behavior is recorded. As such test is geometry dependent by nature, there is generally no standard available. Additionally, one can combine destructive testing and non-destructive testing, by performing X-ray computed tomography whilst running a step-by-step mechanical loading test. This type of test in called *in-situ* testing and provides the 3D deformation of the structure as a function of the load and the creation of damage can also be recorded (digital image correlation, digital volume correlation). An example of such test is shown in Figure 1.



a) Sample before in-situ testing *b) Load vs displacement curve* *c) Sample after in-situ testing*

Figure 1: in-situ compression test of 3D printed PMMA cylindrical scaffold.

3.2 Microstructural characterisation

Microstructural characterisations are mainly performed for metallic and ceramic materials. They usually involve optical and/or electron microscopy to examine multiple phases within a material, to locate and

characterise defects, or to observe damaged or degraded areas in failure analysis investigations. The sample is first mounted in resin then its surface is polished to the desired level [ASTME3] before immediate examination or after etching. Chemical or electrochemical etching is performed [ASTME407] in cases where grain structure is of interest. Grain boundaries are preferentially attacked during the brief exposure to the etchant, leaving behind a surface wherein the two-dimensional grain structure can be clearly observed. Optical microscopy is generally preferred as it is faster and requires less sample preparation, but the level of accuracy is limited. Additionally, sections can be made parallel or perpendicular to the mounting direction to investigate existing pores in build direction. When a higher accuracy is required, electron microscopy (SEM) can be employed, and the sample then needs to be electrically conductive (or made conductive using a conductive coating). Elemental chemical analysis can be coupled to a microstructural analysis by SEM, as most SEM are nowadays equipped with and EDX detector that can record the X-ray spectrum emitted by the sample. This spectrum is composed by peaks corresponding to all the different elements that are present in the sample. EDX can be used for qualitative (the type of elements) as well as quantitative (the percentage of the concentration of each element of the sample) analysis.

As polymer materials are generally large molecules comprised of many repeating units, optical/electron microscopy are of a lesser interest. Polymer morphology on a mesoscale (nanometers to micrometers) is particularly important for the mechanical properties of many materials but requires more advanced characterisation techniques such as Transmission Electron Microscopy (TEM). To investigate the molecular structure of polymer materials, many spectroscopic techniques can be used: ultraviolet-visible spectroscopy, infrared spectroscopy, Raman spectroscopy. In all cases, these techniques fall outside the scope of MetAMMI and will therefore not be discussed here.

3.3 Defect detection

3.3.1 Dimension variation

To measure dimensional variations, volumetric methods (XCT, THz-CT, thermography, US testing, RAM), surface methods (CMM, 3D laser scanning, fringe projection, focus variation), density & porosity methods (Archimedes, pycnometer), and other methods (permeability, weight measurement) can be used. For volumetric methods, XCT gives good accuracy, whilst THz-CT, thermography, and US testing can be applied with some limitations with low accuracy. All these methods give local measurements, *i.e.,* the deviation can be located onto the part being tested, while RAM only gives global dimension variation measurements. With surface methods, the reference technique with high accuracy is CMM. 3D laser scanning and fringe projection can scan parts quickly but only with a good accuracy. Focus variation also has a good accuracy but can only be employed with limitations for dimension variation measurements. All the surface methods give local measurements. On the other hand, density and porosity methods only give global dimension variation measurements, as well as weight measurement.

3.3.2 Extra material

Except CMM and permeability method, the techniques that can be employed to measure dimension variations can also be used in a similar fashion to detect the presence of extra materials. The remarks made in section 3.3.1 are still valid. For surface techniques such as 3D laser scanning and fringe projection however, their application is still possible but with some limitations as they require the extra material to be visible from the detector.

3.3.3 Deformation

To measure deformation, volumetric methods such as XCT, thermography and RAM can be used. XCT will give a good accuracy regardless of the sample and provide information on the location and amplitude of the distortion. Thermography will be more limited, both in terms of sample geometry and accuracy, but can provide results in a fast manner. RAM can also be used, if the deformation is significant by regards to the reference sample used. Surface methods can measure deformation as well, in particular CMM, 3D laser scanning and fringe projection methods. The best accuracy will be obtained with CMM, but for a long scan time, whilst 3D laser scanning and fringe projection have a more limited accuracy but a significantly faster scan time. Finally, weight measurements can also provide deformation measurements, if the deformation leads to a significant variation in weight of the printed part.

3.3.4 Structure defects

Structure defects can mainly be detected using volumetric methods (XCT, thermography, US testing, RAM) and weight measurements. XCT gives a full 3D assessment of a sample with a good accuracy, while thermography, US testing, and RAM will give more limited information with a low accuracy. In addition, for RAM, the measurement will be global, similarly to weight measurements. Some surface methods can also be used for structure defect assessment, namely 3D laser scan, fringe projection, and focus variation but the measurements will be possible with good accuracy only with limitations, mainly due to the sample geometry.

3.3.5 Surface defects

Surface defects can mainly be assessed using surface methods (CMM, focus variation, confocal microscopy, white light interferometry, confocal chromic probe instrument, stylus instrument) and XCT. For most surface defects, CMM, focus variation, confocal microscopy, white light interferometry, confocal chromic probe instrument, and stylus instrument will give a high accuracy. The selection of the method will then be based on the size of the feature of interest and the field of view required to be scanned. 3D laser scanning and fringe projection can give good accuracy but are limited to uneven surface / waviness measurements. XCT can measure most surface defects with good accuracy.

3.3.6 Bulk defects

Bulk defects can be assessed by the volumetric methods (XCT, THz-CT, thermography, US testing, RAM), density and porosity methods, and weight measurements. The best accuracy will be obtained by XCT (along with a full 3D description of each individual defect), whereas the other volumetric techniques will provide results with a low accuracy. Density and porosity methods will give results with a good accuracy, but the measurements will be global and not local, *i.e.* pore volume fraction and/or overall density. A similar remark can be made for weight measurements, which will also give a global measurement.

4 Summary

This *Good Practice Guide for the correct choice of characterisation technique depending of the level of accuracy needed and the type of measurement required* was built upon the experience acquired throughout the MetAMMi project. Parts produced by binder jetting, laser-based powder-bed fusion and material extrusion have been investigated as well as parts manufactured using vat photopolymerization and lithography based ceramic manufacturing. Liquid, solid and powdery feedstocks were processed to fabricate metal polymer and ceramic parts with dense and lattice geometries. The widespread range of materials, manufacturing processes, and part geometries was matched by the equally wide range of characterisation techniques employed to characterise these produced parts. Overall, the knowledge gained is gathered and summarised in Table 1 with an overview for the correct choice of characterisation technique depending of the level of accuracy needed and the type of measurement required.
However, only a clear definition of the required measurands will lead to the selection of a well-suited technique. The material and geometry of the specimen are the main limiting factors as to which characterisation technique can be employed. Costs and time, both acquisition time and data processing time, must also be bore in mind to optimally select a characterisation technique.

Table 1: Table for characterisation technique selection as a function of required measurement and needed level of accuracy.

		Volumetric Methods					Surface Methods								Density & Porosity		Others	
		XCT	THz-CT	Thermography	Ultrasonic Testing	Resonant Acoustic Testing	CMM	3D Laser Scan	Fringe Projection	Focus Variation	Confocal Microscopy	White light interferometry	Confocal chromic probe	Stylus instrument (tactile)	Archimedes	Pycnometer	Permeability	Weight measurement
Detectable defects																		
Defect Categories	**Example**																	
Dimension Variation	Too big; too small	✓	✓	✓	✓	✓	✓	✓	✓	✓					✓	✓	✓	✓
Extra material	Residua powder; closed pores	✓	✓	✓	✓	✓		✓	✓	✓					✓	✓		✓
Deformation	Warping; overall distortion	✓		✓		✓	✓	✓	✓									
Structure Defect	Broken/ missing features	✓		✓	✓	✓		✓	✓	✓								✓
Surface Defect	Uneven surface	✓					✓	✓	✓	✓	✓	✓	✓	✓				
	Surface texture; roughness	✓					✓			✓	✓	✓	✓	✓				
	Staircase effect	✓					✓			✓	✓	✓	✓	✓				
	Point defects	✓					✓			✓	✓	✓	✓	✓				
Bulk Defect	Porosity	✓													✓	✓		
	Cavities	✓	✓	✓	✓	✓									✓	✓		✓
	Delamination	✓		✓	✓	✓												✓
Measurement Facts																		
System	Possible **A**ccuracy	A	A	A	A		A	A	A	A	A	A	A	A				
Sample	Geometry (**l**attice/ **d**ense/ **a**ny)	a	d	d	d	a	d	a	a	d	d	d	d	a	a	a	l	a
	Material (**m**etal/ **p**olymer/ **c**eramic/ **a**ny)	a	p c	a	a	a	a	a	a	a	a	a	a	a	a	a	a	a
Efficiency	Costs	€	€	€	€	€	€	€	€	€	€	€	€	€	€	€	€	€
	Time	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱	⏱
Defect detection	**L**ocal or **G**lobal	L	L	L	L	G	L	L	L	L	L	L	L	L	G	G	G	G

Legend:

✓	Possible	⏱	Fast (< 2h)	€	Cheap (< 100 €/h)	A	High (nm range)
✓	Possible with limitations	⏱	Medium (2h – 4h)	€	Medium (100 – 200 €/h)	A	Good (µm range)
		⏱	Slow (> 4h)	€	Expensive (> 200 €/h)	A	Low (mm range)

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