1 X-ray computed tomography inspection in metal additive manufacturing: the role of witness 2 specimens

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14 Abstract

15 This work highlights the capabilities for high resolution X-ray computed tomography (CT) inspection of 16 witness specimens, built alongside a complex part, in metal additive manufacturing. Such witness 17 specimens, which can be standardized in their dimensions (fixed diameter 15 mm with cylindrical 18 shape built in a vertical orientation), allow X-ray CT inspections with fixed and reproducible workflows. 19 The detection of improper process parameters of the additive manufacturing system is possible as is 20 demonstrated in this paper. It is also demonstrated how the presence of inclusions/contamination in 21 the powder feedstock can be detected in the witness specimen. A series of Ti6Al4V witness specimens 22 with varying porosity distributions are presented, which were part of a previous study of builds of the 23 same set of parts on different laser powder bed fusion systems. This demonstrates how various process 24 parameter errors are highlighted and proven to be detectable in witness specimens using standardized 25 CT procedures. More importantly, it also allows the potential to detect layered flaws which can occur 26 horizontally in the build plane. Such layered flaws may originate from reduced laser power, improper 27 powder spreading or due to complete shut-down and restart of a build. A complex bracket and witness 28 specimen cylinder were built and a layered flaw artificially induced by shutting down the system and 29 restarting it. The positive detection of the flaw by CT in the witness rod is demonstrated. This witness 30 rod was recently part of a round robin test and the layered flaw was successfully identified by all 10 31 participants in the round robin test. The witness rod and complex part were subsequently sectioned 32 and optical microscopy reported here. This approach is especially useful for inspection of larger parts, 33 which cannot be inspected using X-ray CT at highest possible resolution due to part size and associated 34 CT scanning time limits.

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Keywords: X-ray tomography; microCT; non-destructive testing; witness specimen; metal additive
 manufacturing; laser powder bed fusion; stop-start flaw; porosity

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40 **1. Introduction**

41 Additive manufacturing (AM) is an emerging technique used to manufacture custom and complex parts 42 for a variety of commercial applications [1–3]. One major industrial interest is the production of metal 43 parts, which is possible for a variety of alloys with excellent mechanical properties: one popular alloy 44 is Ti6Al4V which is used in biomedical and aerospace applications [4,5]. Laser-powder bed fusion (L-45 PBF) is the most widely adopted metal AM technique, which allows the manufacturing of relatively large parts with intricate, complex designs by melting layer-by-layer in a powder bed, using a laser 46 47 beam. For parts built using Ti6Al4V alloy by L-PBF, the mechanical performance can be superior to 48 conventionally manufactured cast and wrought parts [6].

However, despite the huge potential of AM, various manufacturing imperfections can occur which lead to compromised mechanical properties. Of the many types of AM imperfections possible in L-PBF parts, the most technologically important is the presence of porosity. Different forms of porosity can originate from improper process parameters [7,8], changes in the powder morphology (for example due to changing from virgin to used powder [9]), the separation of the part from the support structures during processing, and redistribution of loose powder in the form of funnels [10] as well as other causes that cannot always be predicted.

56 Stop-start flaws are of particular interest in this work. These flaws can be created when the system 57 stops and restarts, for example, due to power failure. The formation of these flaws in the build plane 58 (horizontal in plane of powder bed) is due to the shrinkage during cooling of the solidified part below 59 the powder level during the "off-time" of the laser, creating a thicker powder layer than previous layers and which is then not entirely melted on the next layer when the laser restarts. There is also a thermal 60 61 mismatch which could contribute to the observed porosity formation. A similar effect can occur if the 62 laser power unexpectedly drops creating one or more layers which are imperfectly melted, in this case 63 imperfect melting occurs over a large area, creating a similar flaw type. These horizontal flaws are 64 particularly important as they can potentially extend across the entire part. Even when the extent is not large, the layered flat shape makes this kind of flaw a strong stress concentrator at its (side) edges 65 66 when subjected to loading conditions. The grain evolution during solidification depends on heat flow, 67 so stop/start flaws and other types of porosity can influence the microstructural grain growth in the 68 vicinity of the flaw. The interaction of the pore morphology and microstructural features results in 69 different stress distributions during loading, thus leading to unexpected damage behaviour with 70 different types of pore shapes [11].

71 One of the best-suited methods to analyse AM parts for porosity or other flaw types and to optimize 72 AM processes for porosity minimization is X-ray micro computed tomography (microCT). A recent 73 comprehensive review of the capabilities of present day microCT for the analysis of additively 74 manufactured parts highlights the importance of this type of non-destructive testing for process 75 optimization and final product inspection [12]. The use of microCT is not new in the field of materials 76 science in general [13], and in additive manufacturing in particular [14-18]. However, its wider 77 acceptance and adoption has been limited in the AM community, mainly due to the high costs and 78 complexity of analysis, which varies for each part.

Although the capabilities of microCT are now starting to be appreciated more widely in the AM community, there is a need for standardization of microCT inspections. This is particularly true for measurement of AM part porosity and dimensional metrology of AM parts, as mentioned in [14], in order to improve the interpretation and ultimately the proper usage of the technique as discussed in [19].

84 To this end we have developed a number of simplified and standardized methods for characterisation 85 of porosity, density, and surface roughness of small coupons of 1 cm³ cubes [20-22], and for 86 characterisation of powder feedstock [23]. These methods include prescribed scanning parameters 87 and subsequent image analysis steps, in order to enhance reproducibility of these analyses across 88 different microCT systems and users. Ultimately, the hope is that these methods will be adopted by 89 industry and formally promulgated in voluntary consensus standards published by standards 90 development organisations such as the American Society for Testing and Materials and the 91 International Organization for Standardization [24,25]. These methods can be used to optimize 92 processing conditions prior to building critical parts.

93 In this paper, we demonstrate a similar proposed method of analysing cylindrical witness specimens and highlight the potential for standardization – fixed cylinder sizes allow recipes for CT scanning and 94 95 image analysis improving the reliability of flaw detection. We demonstrate how the process-specific 96 porosity from different pore formation mechanisms are present in both the witness specimen and the 97 complex part built alongside it, for a series of different sets of samples. This confirms the ability to 98 detect these types of flaws in witness specimens with the proposed scan and image analysis steps. The 99 idea is that the witness specimen analysis will always take place with the same resolution and other 100 scan settings, despite having a potentially larger complex part. Additionally, a witness specimen with 101 an artificially induced stop-start flaw is analysed here in detail, including subsequent physical cross 102 sectioning and imaging by optical microscopy. In this example, the machine was stopped and restarted 103 12 hours later, to artificially induce a stop-start flaw. This type of layer defect has been previously 104 detected by microCT scans of a complex part as reported in [26]. The concept of a witness specimen is 105 not new, and their characterization by microCT was reported previously in [27]. Witness specimens 106 are now specified for all Class A and B metal parts fabricated using PBF and Directed Energy Deposition 107 (DED) [28]. These parts are used in critical and semi-critical applications, whose failure would cause 108 significant danger to personnel, loss of control, loss of a system, loss of a major component, an 109 operating penalty, or loss of intended function. The aim of this present work is to demonstrate the 110 suggested fixed scan parameters and a step-by-step workflow to improve the reproducibility of 111 microCT inspections of such witness specimens. The ultimate aim of this is to allow easier usage of the 112 microCT technique for routine quality inspections, thus improving the quality and reliability of AM 113 parts.

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115 **2. Methods**

For the series of witness specimens (rods and cubes) and complex parts (brackets) having different 116 117 process porosity types, the data were taken from previously reported round robin testing 118 encompassing a variety of L-PBF systems and different pore distributions [29]. These sets of samples 119 were produced on a variety of different L-PBF systems with the "optimal" process parameters of each 120 system. All samples were nearly fully-dense at >99.87% density, but the (unexpected) porosity 121 distributions were different and are further described in [29]. More recent work on 5 mm cubes of 122 Ti6Al4V studied the effects of varying process parameters on one system, creating a variety of porosity 123 distributions artificially [8].

In the present work, the fabrication of a witness rod and corresponding bracket with a stop-start flaw was accomplished with an EOS-M280 L-PBF system located at the Centre for Rapid Prototyping and Manufacturing (CRPM) at the Central University of Technology, Free State, South Africa. The powder used consisted of gas atomized Ti6Al4V extra low interstitials (ELI) from TLS Technic with mean spherical particle size of 45 μm. Standard process parameters for Ti6Al4V were used as recommended

by the L-PBF system manufacturer for layer thickness of 30 μ m. Argon was used as a protective atmosphere with oxygen content controlled to stay below 0.12 percent.

131 The parts were all scanned in a microCT system at the Stellenbosch CT facility [30] similar to the 132 complex part scans reported in [31]. The parts include a complex part (a bracket), its witness specimen, 133 and a 1 cm³ cubic coupon produced during the same build. The witness specimen is a 15-mm diameter cylinder built vertically up to total height of the complex part height (in this case roughly 40-mm high). 134 The microCT scans of the witness specimen can be done at a resolution of up to 10 μ m with typical 135 136 microCT systems, but this requires reasonably long scan times and does not allow for mounting the 137 sample at an angle. The selected voxel size is 25 µm which allows a larger field of view and faster scan 138 times, with sufficient quality and resolution to allow detection of important porosity distributions as 139 shown in this work. In the case of this work, the X-ray tomography parameters were: 200 kV, 100 μA 140 with 0.5 mm beam filter, 250 ms acquisition time per image and no averaging of images to allow fast 141 scan time of 20 minutes per sample.

Since the bracket was designed to be used in a load-bearing application, topology optimisation was
 performed to ensure optimal load-bearing capacity relative to weight, this is reported elsewhere [32].
 The bracket was scanned at 46 µm initially and then close-up sections were scanned at 23 µm, with

- 145 similar X-ray settings as above.
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147 **3.** Results and discussion

The results are split into two sections: the first section demonstrates how the porosity distributions in the cubes, witness rods and brackets correlate with one another. This was partly reported in [30], where the focus was on differences in cubes and the witness specimens were not analysed yet. The second section focuses on the detection of an artificially created stop-start flaw present in a single witness rod.

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154 **3.1 Process porosity distributions**

Several types of process-induced porosity, each with a unique mechanism responsible for its formation, are clearly distinguishable in the microCT scans of the samples made by different commercial L-PBF systems as described in [30]. In that previous work, contour pores, lack of fusion pores and keyhole pores (subsurface at the top surface only) were detected in the 1 cm³ cubes, for example. These were unexpected at the time, which highlights the potential for microCT to add value in detection of process errors and for process optimization or refinement.

The presence of these same pore types in the associated complex brackets made by the same L-PBF 161 162 system was also confirmed by microCT scans, though with less clarity in places due to poorer resolution 163 of the brackets. In this paper, microCT scans of witness rods confirm the presence of these same pore types and pore distributions and show that each distribution type can be positively detected in this 164 type of sample. Figures 1,2 and 3 show the examples of contour pores, lack of fusion pores and 165 keyhole pores (at the top surface only) respectively. Different 3D views are used to illustrate the 166 167 presence of the same signature porosity in each set of samples. The importance here is that the witness specimens can be used to check when slight power drops might create lack of fusion pores in a series 168 169 of layers. Figure 1 shows contour porosity which is just below the surface at all vertical walls and is 170 clearly seen in the top view of the witness specimen in Figure 1(d).





Figure 1: Process-induced porosity detected in both witness specimen and complex part, 173 174 demonstrated here for contour porosity at end of scan tracks. Views shown of witness specimen (a) of 175 surface view, (b) transparent angled view, (c) transparent side view and (d) transparent top view. View 176 of bracket shown of (e) surface view, (f) transparent angled view, (g) transparent side view and (h) transparent top view.

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Figure 2 shows lack of fusion porosity and in the witness specimen as seen in Figure 2(c) in a side view, 179 180 the lack of fusion porosity is not uniformly spread across the build height. More porosity is present 181 near the bottom of the witness specimen in this case. In the associated bracket, this difference is not clear (see Figure 2(f) for side view). This is presumably due to additional pore formation mechanisms 182 183 at work in the complex part. The build strategy, the presence or absence of supports, powder delivery 184 and part orientation on the plate have to be analysed as well as process parameters.



Figure 2: Process-induced porosity detected in both witness specimen and complex part, demonstrated here for lack of fusion porosity. Views shown of witness specimen (a) of surface view, (b) transparent angled view and (c) transparent side view. View of bracket shown of (d) surface view, (e) transparent angled view, (f) transparent side view and (g) transparent top view.

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Figure 3 shows an example of keyhole mode pores at the top surface only, this is best seen in the witness specimen in a side view as in Figure 3(c). It is also seen in Figure 3(f) that the same type of pores are present in the bracket at horizontal top-facing surfaces.



Figure 3: Process-induced porosity detected in both witness specimen and complex part, demonstrated here for keyhole mode porosity under top surface. Views shown of witness specimen (a) of surface view, (b) transparent angled view and (c) transparent side view. Views of bracket shown of (d) surface view, (e) transparent angled view, (f) transparent side view and (g) transparent top view.

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In Figure 4 is shown a different set of samples with less porosity but clearly the witness specimen shows layered porosity – a particularly worrying form of porosity. When inspecting the associated bracket closely at a height corresponding to the appearance of the lower layer of porosity seen in the witness rod, this layered porosity is also detected in the in-plane CT slice data (Figure 4 (b)), and was missed in out-of-plane CT slice data (Figure 4 (c), bottom). This porosity distribution might occur due to imperfect powder spreading on this particular layer and confirms the utility of the witness specimen for detecting layered flaws of this type.

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Figure 4: Layered lack of fusion porosity, presumably due to imperfect powder spreading – shown in (a) the witness specimen in 3D, and in the bracket by using carefully aligned slice images it is possible to image the pores (b)in the flaw plane and (c) out-of-plane in one arm of the bracket.

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217 In addition to pores, contamination can occur in L-PBF systems such as from previous builds with 218 different powder [33]. These inclusions can influence the melting process and act as stress 219 concentrators in final parts under loading conditions. Such inclusions have different density, size and 220 shape than the rest of the powder. Powder contamination also plays a potential role in the formation 221 of porosity in L-PBF parts, since the inclusion particles have different melting temperature. It has been 222 shown for example that during in-situ alloying with controlled amounts of different powders, the ideal 223 process parameters change [34]. One set of parts in this series contained such inclusions as shown in 224 Figure 5. This figure shows the detection of high density inclusions, which appear as white dots in both 225 the witness specimen and bracket. Thus, the manufacturing of witness samples is useful for identifying

- 226 negative features such as powder contamination, which is unacceptable in the manufacture of critical
- 227 parts. In this case it was confirmed that contamination from a previous build was likely.



Figure 5: Inclusions (powder contamination) detected in (a) witness specimen and also in (b) the associated bracket – white dots seen in slice images are denser particles.

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232 **3.2 Stop-start flaw**

233 Besides inherent process porosity, some errors can occur which create localized porosity or flaws, 234 which can extend across the entire build plane or large parts of it. Of these, one of the most important 235 is the stop-start flaw which occurs when there is a shutdown of the system and a restart later. This 236 type of flaw is caused by shrinkage of the solidified part, which creates a larger layer height of powder 237 in the next layer upon restart, which does not fully melt, thus leading to a specialized form of lack of 238 fusion. A similar effect of imperfect melting on a single layer can occur when the laser power drops 239 temporarily, or when powder spreading is uneven due to part warping or recoater damage, for 240 example. Such effects may potentially be spread across the entire build, which means it may be 241 detectable by using witness specimens. This is shown for an artificially induced stop-start flaw in a 242 witness rod in Figure 6.



Figure 6: Stop-start flaw detected in witness specimen, different views of (a) 3D surface, (b) transparent angled view, (c) transparent side view, (d) slice top view in plane of flaw, (e) with the associated slice plane indicated, (f) slice side view and (g) associated slice plane indicated.

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249 Detecting a start-stop flaw or other type of layer defect in a production part at the same height as the 250 flaw which was observed in the corresponding witness specimen (Figure 6) can be problematic for X-251 ray tomography. In this case, despite a stop-start interval of 12 hrs (the machine was stopped during 252 the build and restarted the next day), no layer defects were found inside the complex part (bracket) at 253 the 46-µm resolution of the bracket scan. A higher resolution "zoom scan" of the bracket at 23-µm 254 voxel size of the potentially problematic area corresponding to the known build height and build 255 orientation where the build was stopped and restarted is shown in Figure 7.



Figure 7: High resolution "zoom scan" of a potentially problematic area, corresponding to stop-start layer defect in a witness rod at a known z-height in the build. No layer defects were found in this case (23-µm resolution). Shown here are (a) the cross sectional (side) view and (b) top view in plane of expected location of layered defects (13 mm from top as measured from witness specimen).

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However, no layer defects were found in this area or elsewhere in the build plane corresponding to the location of the stop-start flaw. The bracket was also sectioned and no flaws were found under nominal magnification with an optical microscope. Stop-start flaws may not be present in all locations in the build plane, as also seen in the witness specimen (the defect does not cover the entire area of the cylinder), and in this case they did not extend into the bracket.

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269 The detection of planar 2D-flaws in AM parts perpendicular to the build (Z) direction is inherently 270 challenging for any NDE technique, including microCT. As pointed out in [35], AM processes tend to 271 prohibit volumetric defects with significant height in the Z direction. The major concern, therefore, is 272 for planar defects, such as aligned or chained porosity or even laminar cracks, or the stop/start defects as examined in this paper, that form along the build plane. The implications of this are: 1) planar 273 274 defects are well suited for growth, 2) planar defects generally have low contained volume, 3) the 275 orientation of defects of concern must be known before inspection, especially when detection 276 sensitivity depends on the defect orientation relative to the inspection direction, and 4) the Z-height 277 of planar defects can be demanding on incremental step inspection methods such as CT. Therefore, it 278 is important to manually assess slice images in microCT data from at least 2 orthogonal orientations 279 and it is critical that the part is scanned at an angle relative to its original build direction. Regardless, 280 when a larger production part cannot be inspected at sufficiently high resolution due to size limits, and 281 layer or planar defects are positively identified in a smaller matching witness coupon, it would be safer 282 to assume the presence of undetected layer defects in the production part and reject the part.

283 Microscopic analysis of physical cross-sections of the cylinder shows that the stop-start layered defect 284 is comprised of a chain of pores with irregular shapes. Large irregular pores were found with vertical 285 sizes ranging from 120 to 180 µm with narrow (up to 20 µm) shrinkage cavities. Usually, big irregular pores correlate with low energy input, when laser power density was not enough to fully melt the 286 287 powder layer and previously melted material (also known as lack-of-fusion). Taking into account the 288 30-µm powder layer thickness used in this experiment, and the optimal process parameters needed 289 to produce a fully dense part, the reason large stop-start defects occurred in the witness rod in this 290 case can be attributed to shrinkage of the whole system during cooling, including the powder 291 delivering system, baseplate and as-built part which had been previously melted in the first cycle prior 292 to machine stoppage. The redistribution of residual stresses, detaching from the substrate or the 293 warping of parts during cooling for several hours can lead to uneven layer thickness when the next 294 powder layer is delivered. So, interaction of all these factors can lead to random porosity in L-PBF parts 295 after a stop-start cycle, as was found in this experiment and as revealed by the presence of horizontally 296 aligned pores in the witness rod (Figs. 6 and 8). The same defects were expected in the bracket but 297 were absent (Fig. 7, 8(b) at the arrows). . In this stop-start L-PBF process, defects occurred along several 298 layers taking into account their size (Fig. 8a) but despite this, the extent was not across the entire build 299 plane and did not extend into the bracket.

As previously stated, the interaction of the microstructure and different types of porosity can be critical for the performance properties of the L-PBF part [36]. The interruption of the microstructural grain growth (in Ti6Al4V prior beta-gains grow typically vertical along the build direction) makes for possible new locations of crack initiation and growth along the inner (top and bottom) edges of the flaw along the vertical grain boundaries ending at the flaw.

Sharp edges of pores interrupted by prior-beta grains and notches coinciding with the direction of
 acicular martensitic α' phase (Fig. 8a) can influence not only the crack initiation under loading but can
 also deteriorate the fatigue performance of as-built and stress-relieved L-PBF components. Textured
 microstructure related to anisotropic structural properties usually remains even after heat treatment,
 for example, in Ti6Al4V [5,37].



Figure 8: Cross section and microstructure of cylinder with layered defects (a) and part of the bracket where layered flaw was expected but was not found at location indicated by white arrows (b).

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The advantages of the microCT inspection of witness specimens using standardized workflows has been clearly demonstrated in this work. The inherent disadvantage is that some layered flaws or irregular porosity distributions may occur in a complex part but not in the witness specimen. This means that the microCT inspection should be complimented by other inline process monitoring and post-process quality control tools. The shape and size of witness specimens, their position near the complex L-PBF component and the extent of layered flaws across the build plane justify a separate study in future.

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For denser metals the method will need some modification compared to that presented here. The work in this paper was presented for Ti6Al4V and will be suitable for less radiodense materials (e.g., Al alloys, plastics, etc.). For denser materials, a narrower/finer witness specimen may be required to allow penetration of typical laboratory microCT X-ray beams. Also, it is imperative that the orientation of defects of concern is known before inspection to maximize CT detectability of known or suspected

328 planar flaws. This means the build/print direction must be known and part angled relative to this, to 329 ensure proper detection of the layered flaws in build plane. For larger L-PBF parts, the Z-height of 330 planar defects such as the stop-start flaw examined here can be demanding on incremental step 331 inspection methods such as CT. Nevertheless, this method should be useful for routine analysis with 332 the only modification being the resolution of the scan of the complex part, which in turn depends on 333 part size. The standardization of witness specimen geometry (e.g., uniform coupon diameter) allows a 334 fixed methodology for all identically shaped additively manufactured witness specimens, regardless of 335 the AM platform used, machine-to-machine variation, or variation within a single AM machine. The 336 only limitation is that larger parts will require longer witness specimens, which will require longer scan 337 times to identify flaws with the requisite resolution (≤ 20 -µm voxel size in this paper). The advantage 338 is that only one witness specimen may be adequate for an entire batch of parts to ensure the absence 339 of unwanted layer, cross-layer, or other volumetric defect types (e.g., inclusions, trapped powder, 340 cracks, etc.).

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342 Conclusions

343 The advantages of using witness specimens and microCT scanning thereof according to fixed workflows 344 was investigated. It was shown how this approach can accurately identify process porosity signatures, 345 which can act as "witness" to in-process changes in the parameters over a single layer or multiple 346 layers. The presence of contamination of metal powders was demonstrated in one case and this was 347 accurately detected in both a witness specimen and its corresponding complex part. Lack of fusion porosity detected in a witness specimen was found to occur across multiple build layers in one case, 348 349 and analogous lack of fusion porosity was confirmed in the complex bracket associated with this 350 witness specimen. Finally, an artificially induced stop-start flaw was investigated and its detection in 351 witness specimen confirmed and analysed in detail using microCT and optical microscopy of cross-352 sections. This stop-start flaw was found to extend widely but not completely over the entire part, and 353 in this case, did not extend to the complex part built alongside it (also investigated by microCT and 354 optical microscopy). This points to the possibility that unexpected flaws including layered flaws may 355 occur in complex parts despite passing a witness specimen microCT test. Similarly, there may be 356 situations where localized power fluctuations occur or where build quality varies with location in the 357 powder bed. This means that additional complementary tools are needed for 100% quality control and 358 understanding the limits of the microCT technique to detect planar defects is therefore important. This 359 work is expected to contribute to the wider understanding and better utility of microCT as inspection 360 tool, especially with standardized workflows using witness specimens.

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