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Porosity regrowth during heat treatment of hot isostatically pressed additively manufactured titanium components

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ABSTRACT

X-ray computed tomography has been used to track the behaviour of individual pores found in selective electron beam melted additive manufactured titanium. Porosity was found to shrink below the detection limit of X-ray microtomography (<5 μm) upon hot isostatic pressing. Spherical argon containing gas pores, which have a high internal gas pressure following hot isostatic pressing, have been found to progressively reappear and grow in proportion to their original as-built size during high temperature (β-anneal) treatments, whereas larger irregular low pressure pores did not reappear. The implications of these observations in terms of additive manufacturing are discussed.

In additive manufacturing (AM), complex components can be produced directly from CAD models by the sequential deposition and consolidation of 2D layers of material [1,2]. The benefits of this new manufacturing approach have been well documented in the literature [1,2]. However, with powder bed AM, residual gas porosity and lack of fusion defects (LOFDs) are known issues [3] that, if not adequately controlled, can cause a significant knockdown in fatigue life of components [4]. In powder bed AM, irregular LOFDs arise when the melt pool is too small to fully fuse the powder, while gas pores originate from bubbles which are trapped in the melt pool on solidification and have a near spherical morphology [5]. In the powder bed AM process selective electron beam melting (SEBM) of titanium, gas pores have been shown to dominate the number fraction of the total porosity present (>95%) [5]. Although their overall volume fraction is low (<0.2% [5]), the high frequency of such gas pores means that SEBM fatigue samples often fail from cracks initiating at this type of defect [4]. To solve this problem, many aerospace companies are considering applying post-build hot isostatic pressing (HIPing) treatments to AM components. It has been reported that HIPIng of AM parts is very effective at closing porosity, which reduces internal stress concentrations, with a consequent increase in fatigue life [2,6].

Recently, the current authors used high resolution 3D X-ray computed tomography (CT) to confirm that, with SEBM, all the as-built internal pores could be removed/shrunk by a standard HIP cycle (Fig. 1b) to below the detection limit of the equipment used (~5 μm) [6]. Here, we aim to examine the effect of applying a typical β-anneal heat treatment on the porosity closed by HIPing. Since SEBM is carried out in a vacuum chamber, at a pressure <1 Pa, the gas pores found in built components mainly originate from bubbles of argon that were originally trapped in the feedstock powder particles during atomisation, some of which are subsequently unable to escape from the melt pool during SEBM. Argon has an atomic radius too large for significant solubility in titanium [7] and thus, following HIPing, some argon containing pores might be expected to survive in the material, but at a significantly smaller size and higher internal pressure. Indeed, if equilibrium is reached the pore pressure could equal the HIPing pressure of around 100 MPa.

To examine whether a typical β-annealing treatment would result in pore growth in HIPed AM components, CT scanning was performed on the same sample after manufacture by SEBM, after HIPing under a 100 MPa pressure at 920 °C for 2 h (see ref. [6]), and following annealing for different times and temperatures, so that individual pores could be tracked at each stage within the sample. The sample comprised a 10 mm high, 1.7 mm diameter, cylinder having an axis aligned with the build direction, machined from the centre edge of the face of a $10 \times 10 \times 15\ mm^3$ cuboid sample. The cuboid sample was manufactured with argon gas atomised, Ti–6Al–4V powder in an Arcam AB SEBM system using standard operating conditions (see refs. [5,6,8] for details). This consisted of first melting the perimeter of each 2D layer by 'contouring', before consolidating the internal area using a snaking 'hatching' pattern. The cylinder was deliberately machined from the contour-hatch overlap region, which prior work has shown contains a relatively high number density of LOFDs and gas pores [5].

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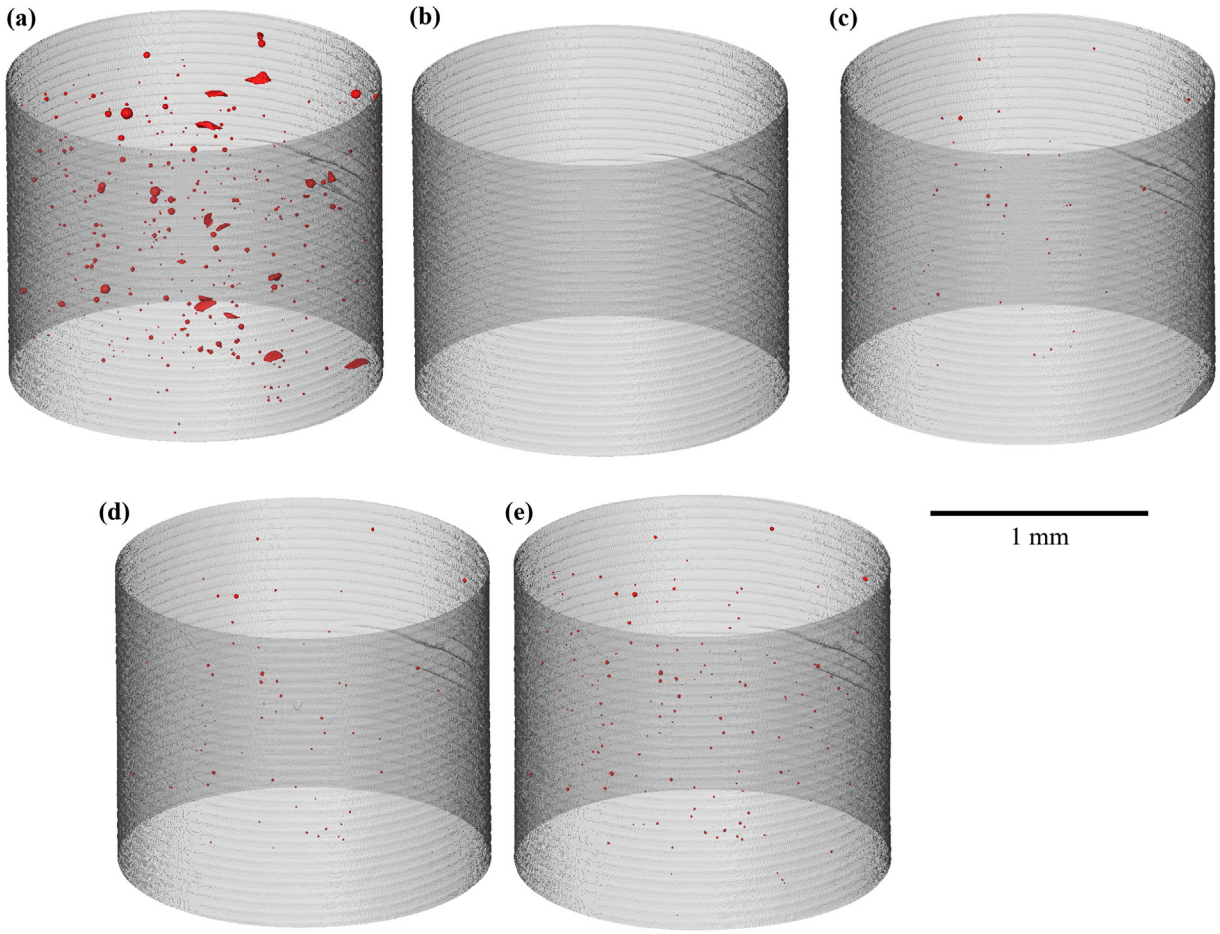

Fig. 1. 3D visualisation of the porosity (red) imaged by CT scans of the same cylindrical sample (build direction vertical) (a) as-built; (b) following HIPing; (c) 10 min at 1035 °C; (d) 10 h at 1035 °C; and (e) 10 min at 1200 °C.

X-ray CT scanning was undertaken using a Xradia Versa 500 machine on the mid-section of the (~1.5 mm height) cylindrical sample, using the procedure fully described in ref. [5]. Under these conditions, a 2 μm voxel size was achieved allowing the reliable identification of defects with an equivalent (sphere of equal volume) diameter greater than 5.2 μm. The CT data was analysed using Avizo 9.0 software.

Three heat treatments were applied sequentially to the HIPed sample in the following order: HT1, 10 min at 1035 °C (1308 K); HT2, 10 h at 1035 °C; and HT3, 10 min at 1200 °C (1473 K). The first heat treatment (HT1) was chosen to replicate a typical β-anneal employed by the aerospace industry, while HT2 and HT3 were used to study the effect of increasing the hold time and maximum temperature, respectively. To avoid oxidation, all heat treatments were performed in a vacuum furnace at <1 Pa, using a heating ramp rate of 5 K/min and allowed to cool to ambient temperature under vacuum. After each heat treatment, the same mid-section of the cylindrical sample was rescanned by CT and the data spatially realigned, to correlate between scans the behaviour of any pores that were present.

Fig. 1 shows a 3D visualisation of the pores detected in the cylindrical sample as-machined from the built cuboid, and following HIPing and the three sequential heat treatments described above. It should be noted that in the as-built condition, at this location near the edge of the sample, the volume fraction of LOFDs was high in comparison to that seen in the centre of an as-manufactured build, where LOFDs typically make up only a minor fraction of the pore population [3,5]. The contour strategy, used to melt the edge of SEBM samples, has been shown to contain a greater number density of LOFDs, in comparison to the hatching strategy used to melt the internal area [5].

From Fig. 1b it is apparent that after HIPing no pores can be detected in the scanned volume shown, and this is confirmed by the statistical data presented in Table 1. However, following the first heat treatment step (HT1), CT analysis revealed the presence of some small pores. Following the second and third heat-treatment, more and larger pores became evident. It is notable from Table 1 that increasing the hold time (HT2) led to less pore growth than increasing the hold temperature (HT3).

From visual inspection of the 3D projections given in Fig. 1 it is hard to correlate the as-manufactured pores through the HIPing and heat treatment stages. However, in Fig. 2 the evolution of a single pore is shown. Detailed analysis has confirmed that all the pores appearing during the heat treatment stages can be correlated to the locations of pores in the original build. Evidently such pores (Fig. 2a) had shrunk/disappeared during HIPing (Fig. 2b), before reappearing and growing during the subsequent heat treatment steps (Fig. 2c–e).

Table 1
Statistical data from quantification of all the pores detected in the same sample, as-built, and following HIPing and annealing.

Condition	Volume fraction (%)	Number	Mean equiv. dia. (μm)	Max. equiv. dia. (μm)
As-built	0.0397	309	13.3	53.3
HIPed	0.0000	0	–	–
HT1	0.0007	49	8.4	18.6
HT2	0.0011	63	9.2	20.0
HT3	0.0026	140	9.4	21.6