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Porosity Measurement for HIPped AM Ti6Al4V Samples – A Practical Comparison between Archimedes Method and Micro-CT

Eİ ile Üretilen SİP Uygulanmış Ti6Al4V Numunelerde Gözeneklilik Ölçümü – Arşimet Yöntemi ve MikroBT arasında Pratik bir Karşılaştırma

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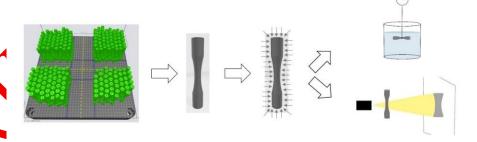
Highlights

Porosity is a critical defect that significantly influences the mechanical properties of additively manufactured (AM) Ti-6Al-4V components produced via Laser Powder Bed Fusion (LPBF). This study investigates the porosity levels of four groups of AM Ti-6Al-4V samples. Three of these groups underwent Hot Isostatic Pressing (HIP) using distinct process parameters, while the fourth group consisted of as-built specimens without any post-processing. Porosity measurements were conducted using both the Archinedes method and micro-computed tomography (Micro-CT). While Micro-CT offers detailed internal imaging, the Archinedes method—widely regarded as a conventional technique—demonstrated advantages in terms of simplicity, repeatability, and cost-effectiveness.

Graphical Abstract

Anahtar Kelimeler

Eklemeli İmalat Sıcak İzostatik Presleme Boşluk Arşimet Yöntemi Mikro-BT



Abstract

This study preents a comparative analysis of porosity measurement techniques using the Archimedes method and micro-computed tomography (Micro-CT) for additively manufactured (AM) Ti-6Al-4V components. The samples were subjected to four different post-processing conditions: three groups were processed using Hot Isostatic Pressing (HIP) with varying parameters, while one group remained in the as-built condition. While the Archimedes method offers a faster, more cost-effective approach for quantifying overall porosity levels, Micro-CT differs from the Archimedes Method in that it is also capable of detecting the locations and shapes of voids within the sample.

Özet

Bu çalışma Arşimet Yöntemi ve Mikro-BT (Bilgisayarlı Tomografi) ile boşluk/gözenek ölçümünün karşılaştırmalı bir incelemesini sunmaktadır. Eklemeli imalat (Eİ) ile üretilen Ti6Al4V numuneler 3 farklı Sıcak İzostatik Presleme (SİP) parametresiyle ardıl işleme tabi tutulmuş olup, bir grup da işlemsiz olarak bırakılmıştır. Arşimet Yöntemi boşluk seviyesini ölçmek için hızlı ve ekonomik bir yöntemdir. Mikro-BT ise numune içindeki boşlukların konumlarını ve formlarını da tespit edebilme özelliğiyle Arşimet Yöntemi'nden ayrışmaktadır.

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1. INTRODUCTION

Ti-6Al-4V, the most widely used wrought titanium alloy, is also among the most commonly utilized metals in additive manufacturing (AM) technologies. Due to its high specific strength, excellent corrosion resistance, and stable mechanical performance even at elevated temperatures, it plays a critical role in high-performance sectors such as aerospace, defense, and biomedical engineering [1–4].

In the present study, the porosity characteristics of Ti-6Al-4V manufactured via Laser Powder Bed Fusion (LPBF) are examined. Specifically, two widely adopted porosity evaluation techniques are compared: the Archimedes method [5–8] and micro-CT [9–12]. A total of 16 Ti-6Al-4V specimens produced by LPBF were analyzed to determine their porosity levels using both techniques. The aim was to assess the ability of these methods to detect density differences between HIP-processed samples and those in the as-built condition.

The results demonstrate that the Archimedes method—a conventional, cost-effective approach—is capable of detecting density variations as small as 0.0001 g/cm³ [13]. In contrast, micro-CT offers more advanced capabilities, enabling the visualization of internal pore morphology, size, and spatial distribution with high resolution [14-15].

As quality control and standardization are critical concerns in the AM industry, the choice of porosity evaluation technique becomes significant. Due to its simplicity and low operational cost, the Archimedes method may serve as a routine inspection tool for identifying deviations in part quality after each print. When

more detailed analysis is required, micro-CT can provide comprehensive insights into the internal structure of the material.

Although additive manufacturing offers significant advantages in terms of design freedom and production flexibility, it also introduces challenges in process qualification and part validation. AM processes, characterized by layer-by-layer fabrication, rapid localized heating and cooling, and the use of metal powders, inherently lead to manufacturing defects—perosity being among the most critical. Porosity has a particularly detrimental impact on mechanical properties, especially ductility and fatigue performance [16-17].

With advances in AM process optimization, reported porosity levels in metal AM parts have significantly decreased—from initial values as high as 13% to less than 0,002% in optimized builds [18]. Various methods are available to quantify porosity in metal AM parts, including the Archimedes method, scanning electron microscopy (SEM) of polished cross-sections, optical microscopy (OM), and X-ray computed tomography [18–22].

In this study, the focus is placed on evaluating the strengths and limitations of the Archimedes method and micro-CT as viable techniques for porosity assessment. Their applicability for use in quality control protocols for metal AM parts is critically examined, with the goal of supporting process validation and standardization efforts within the industry.

2. MATERIAL AND METHODS

A total of 212 Ti-6Al-4V coupons were fabricated using a Laser Powder Bed Fusion

(LPBF) process on an EOS M400-4 machine, operating with a layer thickness of 60 µm, a volumetric energy density of 37.78 J/mm³. The process parameters were configured with a laser power of 340 W, a scanning velocity of 1250 mm/s, a hatch spacing of 0,12 mm, and a spot size of 80 µm. The fabrication was conducted in an argon atmosphere using used Ti-6Al-4V powder. A total of 16 specimens were sampled from different regions of the build chamber for subsequent porosity characterization. approach was employed to minimize potential biases location-specific associated with variations within the chamber. The chemical composition of the material is provided in Table 1.

Of the selected samples, 12 underwent Hot Isostatic Pressing (HIP) and were divided into three subgroups (n = 4 per group), each treated under different HIP conditions. The remaining four samples were retained in the as built condition. The HIP parameters applied were as follows:

HIP #1: 920 °C / 100 MPa X hours / cooling rate of 10–20 °C/min

HIP #2: 815 °C (190 NPa /2 hours / cooling rate of 10.20 °C hair

HIP #3. 920 °C 100 MPa / 2 hours / cooling via Quintus Cunform Rapid QuenchingTM, with a cooling rate of 850 °C/min [20]. The high cooling rate in HIP #3 was employed to regulate the microstructure. This approach was intended to prevent grain coarsening and the consequent reduction in mechanical strength commonly associated with the slow cooling conditions of HIP processes.

Table 1: Chemical composition (weight percent).

| Alloy | Al | V | Fe | Ti |
|---------------|----------|---------|------|---------|
| AM Ti6Al4V | 5,83 | 4,27 | 0,27 | 89,63 |
| Reference | 5,5-6,75 | 3,5-4,5 | <0,4 | 87,6-91 |

2.1 Archimedes Method

This method is based on the principle of buoyancy, as described by Archimedes' principle, which determines the density of a specimen by assessing its weight in two different media: air and distilled water. Initially, the mass of the sample is measured in air, followed by a subsequent measurement in distilled water. The difference between these two readings enables the calculation of the sample's density. The porosity percentage is then determined by comparing the experimentally measured density with the theoretical density of fully dense Ti-6Al-4V alloy.

Density measurements were performed using an A&D HR-250AZ analytical balance (Figure 1). The resulting porosity values, which ranged from 0,038% to 0,291%, are presented in Table 2. The density and percentage of the porosity for the coupons are represented in Figure 2 and Figure 3, respectively.



Figure 1: Density measurement equipment (A&D HR 50AZ) and sample orientation for Archimedes method.

Table 2: Densities and porosities with corpor numbers and subgroups with Archimedes Method.

| НІР | 4 | Sample # Density (g/cm³) – Porosity (%) | | | | | |
|------------|----------------|-----------------------------------------|----------------|----------------|----------------|--|--|
| HIP #1 - | Mean Value | #22 | #30 | #23 | #4 | | |
| | 4,4289 - 0,033 | 4,4284 - 0,043 | 4,4286 - 0,038 | 4,4281 - 0,050 | 4,4303 - 0 | | |
| HIP #2 | Mean Value | #48 | #47 | #42 | #53 | | |
| | 4,4278 0,058 | 4,4265 - 0,085 | 4,4278 - 0,056 | 4,4282 - 0,047 | 4,4285 - 0,041 | | |
| HIP #3 | Mean Value | #114 | #84 | #88 | #117 | | |
| | 4,4263 - 0,091 | 4,4267 - 0,0813 | 4,4251 - 0,117 | 4,4282 - 0,047 | 4,4251 - 0,117 | | |
| As-built - | Mean Value | #150 | #124 | #155 | #156 | | |
| | 4,4194 - 0,247 | 4,4174 - 0,291 | 4,4204 - 0,223 | 4,4207 - 0,217 | 4,4190 - 0,255 | | |

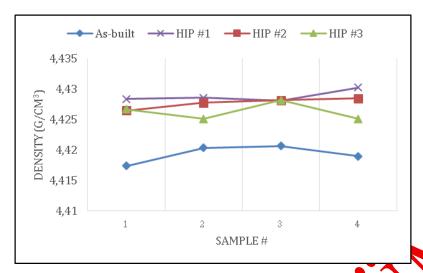


Figure 2: Density measurement using Archimedes Method.

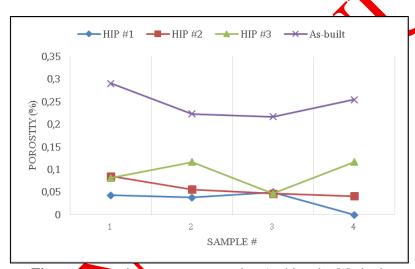


Figure 3: Porosity measurement using Archimedes Method.

2.2 Computerized Tomography (CT)

CT is a non-destructive imagine technique used to visualize the internal structure of materials in three dimensions [18, 23-27]. In this method, an X-ray source emits radiation that passes through the specimen, with the intensity of the transmitted rays decreasing as they traverse the material. Internal features such as pores, voids, unmelted powder particles, and cracks influence the attenuation of the X-rays, thereby affecting the signals received by the detector. By rotating the specimen around its axis and capturing multiple radiographic projections at various angles, a 3D voxel-based representation of the internal

structure can be reconstructed through postprocessing of the acquired data.

This technique enables the identification and analysis of internal defects, including pores, voids, and cracks, with high spatial resolution. Depending on the desired level of detail, the internal architecture of the material can be rendered in high-resolution 3D.

Compared to the Archimedes method, CT analysis requires more advanced equipment and specialized software for data reconstruction and analysis. While Archimedes method provides a relatively simple approach to estimating density and porosity, it lacks the spatial resolution and

visual detail offered by CT. Furthermore, CT is more time-consuming and costly, particularly when high-resolution scans and detailed analyses are required.

In this study, four samples manufactured using four different parameter sets were scanned using a Zeiss Xradia 510 Versa Micro-CT system (Figure 4) The segmentation threshold was set to 0,87, with a minimum detectable porosity size of 15,68 μ m. Image reconstruction was performed automatically by the software, applying a beamhardening correction of 0,05 and a center shift of

0,5. The thinner sections of each specimen were selected for scanning, and the resulting images were evaluated for internal defects and structural integrity (Figure 5). The results are compiled in Table 3. This scanned section was considered representative of the overall porosity structure of the sample due to its position along the build direction. Its location at the mid-height of the specimen further supports the assumption that it reflects the average porosity characteristics of the lower and upper sections.

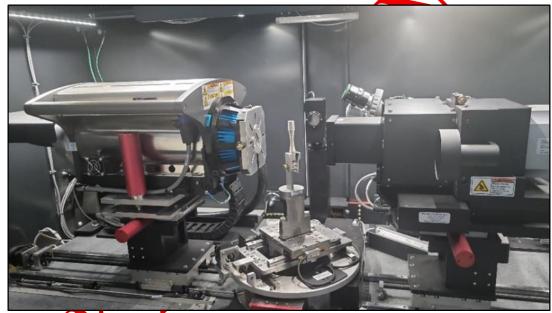


Figure 4: Micro-CT set-up for Zeiss Xradia 510 Versa.

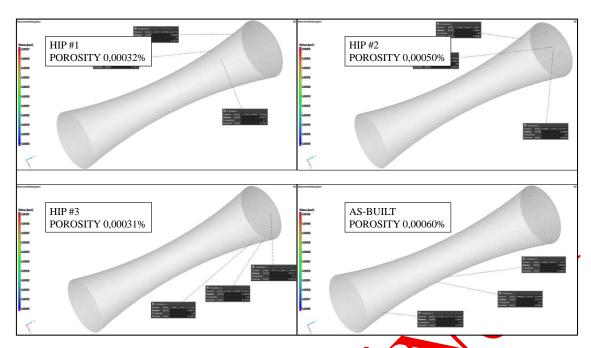


Figure 5: Micro-CT images with porosity percentage.

Table 3: Densities and porosities with coupon numbers and subgroups using Micro-CT.

| | Sample # Density (g/cm³) – Porosity (%) | | | |
|------------------------|-----------------------------------------|---------------------|---------------------|-------------------------|
| HIP Parameter Sample # | HIP #1 Sample #4 | HIP#2 Sample #53 | HIP#3 Sample #84 | As-built Sample #124 |
| Density – Porosity % | 4,43029 - 0,00032 | 4,43028 - 0,00050 | 4,43029 - 0,00031 | 4,43027 - 0,00060 |

2.3 Comparison of Porosity Characterization Methods

Both the Archimedes method and Micro-Computed Tomography (Micro-CT) offer distinct advantages and limitations in the characterization of porosity, depending on the specific requirements of the analysis.

The Archimedes method is a relatively simple, rapid, and cost-effective approach for estimating the bulk density and overall porosity of a material. It involves basic equipment and straightforward calculations, making it accessible for routine quality control or preliminary assessments. However, it provides only a single scalar value for density and porosity, without any information on the spatial distribution, shape, or

size of internal defects. In essence, the method treats the specimen as a "black box," offering no insight into the internal structure or the precise nature of the porosity.

In contrast, Micro-CT is a more advanced and highly detailed technique that enables three-dimensional visualization of a specimen's internal features, including the size, shape, and spatial distribution of pores, voids, and cracks. This method provides volumetric data with high spatial resolution, making it particularly valuable for identifying localized defects and assessing the structural integrity of materials. However, these advantages come at the expense of longer processing times, higher operational costs, and

the need for specialized software and expertise for image reconstruction and analysis.

While the Archimedes method is significantly more economical and faster in terms of processing time, Micro-CT incurs higher costs per measurement, particularly when full-volume scans at high resolution are required. Additionally, the technological complexity and resource demands of Micro-CT make it less suitable for high-throughput or cost-sensitive applications.

In summary, the choice between these methods should be guided by the specific goals of the investigation: if a rapid and general assessment of porosity is sufficient, the Archimedes method is appropriate. However, for detailed structural analysis and defect localization, Micro-CT offers unparalleled capabilities.

3. RESULTS AND DISCUSSION

The results indicate that the densities of HIPped specimens are marginally higher than those of the as-built samples. Even the as built specimens demonstrated high densitication, with an average density of approximately 4,4190 g/cm³—corresponding to 99,75% of the theoretical density [28,29]. Among the HIPped samples, one specimen (Sample #4) exhibited a density nearly equivalent to the theoretical maximum, indicating the effectiveness of the HIP process in minimizing internal porosity. No significant effect of the rapid cooling condition applied in HIP#3 on porosity was observed when compared with HIP#1 and HIP#2.

A general correlation was observed between the porosity trends identified by both methods. As expected, the as-built samples exhibited the highest porosity levels. However, a direct comparison of absolute porosity values revealed a significant discrepancy: porosity values obtained via the Archimedes method were up to 0,22% higher than those calculated using Micro-CT. This disparity can be attributed to the smaller porosities under the size of the minimum detectable porosity of the Micro-CT, CT results can be affected by imaging artefacts arising from the interaction between X-rays and the material, by the spatial resolution achieved during CT reconstruction, and by the thresholding procedure [30,31]. Particularly in higher-density materials, the porosity measurement results obtained by the methods can exhibit substantial discrepancies [32] The Archimedes method captures the total volume of open and closed pores within the entire sample. Micro-CT, particularly when scanning only a subsection, hay underestimate total porosity due to its localized focus and potential resolution limitations for detecting sub-voxel defects.

4. CONCLUSIONS

The Archimedes method offers a cost-effective and straightforward approach to porosity estimation. Its simplicity and low operational requirements make it a practical choice for routine quality control, and it is well-suited for use as a standard procedure to monitor each production batch for anomalies.

Micro-CT, on the other hand, provides a more advanced and comprehensive analysis by enabling three-dimensional visualization of internal features. This method is particularly useful for detailed investigations, such as defect morphology analysis and fatigue strength prediction. However, its high cost, longer

processing times, and limited accessibility—especially among smaller AM facilities—restrict its widespread use.

Both methods have their respective strengths and limitations. While Micro-CT offers highresolution structural insight, conventional techniques such as the Archimedes method remain valuable, especially for rapid screening and process monitoring. A balanced and informed use of all available methods, based on their capabilities and limitations, is essential for ensuring the quality and reliability of AM components. Understanding the complementary roles of these techniques will support the broader AM community in establishing robust qualification procedures.

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AUTHOR CONTRIBUTIONS

Osman Tuna GÖKGÖZ: Literature review, Analysis evaluation, Experimentation Writing.

Hüdayim BAŞAK: Methodology, Review and editing.

Olcay Ersel CANYURT: Methodology, Funding, Review and editing.

Katri KAKKO: Review and editing, Sample building.

Chad BEAMER: Review and editing, Sample processing.

CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest.

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