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Analysis of the Mechanical Properties of Cold Spray Additively Manufactured Titanium: Effects of Thermal Treatments

by Leo M. D. Driggers, Kendrick H. Tran, Santiago Flores-Pereira, Clinton Strosser, Braden L. Miller, Joshua Cline, Matthew K. Dunstan, Isaac Nault, and Brady G. Butler

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14. ABSTRACT
This study explores using cold spray additive manufacturing to produce high-performance components from commercially pure titanium. The as-sprayed material initially exhibited low mechanical performance with limited strength and ductility. However, heat treatments, including vacuum annealing and a thermochemical hydrogen-based process, significantly enhanced the material's properties. Posttreatment samples demonstrated yield strengths exceeding 700 MPa, tensile strengths above 800 MPa, and elastic modulus values greater than 120 GPa, highlighting the potential of cold spray to produce strong, corrosion-resistant components from unalloyed titanium. While both heat treatments effectively improved mechanical performance, differences between the methods were minimal in properties such as toughness and hardness. The results demonstrate that combined with appropriate heat treatments, cold spray is a promising method for manufacturing titanium components with excellent strength and durability. This approach offers a cost-effective and sustainable alternative to traditional titanium manufacturing, with opportunities for further optimization to meet the demands of advanced engineering applications.

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1. Introduction

Titanium (Ti) alloys are indispensable in high-performance engineering applications due to their high strength-to-density ratio, excellent corrosion resistance, and exceptional biocompatibility. Despite being the fourth most abundant structural metal in the earth's crust,[1] Ti's strong affinity for oxygen delayed commercial production until the development of the Kroll process in the mid-20th century.[1,2] Remarkably, the fundamental methods for producing Ti alloys have changed little in the past 80 years, leaving ample opportunity to improve the cost-to-performance ratio of Ti and its alloys through novel manufacturing and processing techniques.

Commercially pure Ti (CP-Ti) stands out for its superior corrosion resistance, making it indispensable in industries such as aerospace, biomedical, and chemical processing, where high strength is typically not required.[3] However, broader adoption of CP-Ti depends on enhancing its mechanical properties to rival those of Ti alloys. Addressing this challenge requires rethinking manufacturing approaches to balance performance with cost-effectiveness.

Powder-based manufacturing methods, particularly additive manufacturing techniques, offer a promising avenue to achieve this balance.[4] Among these, cold spray additive manufacturing (CSAM) has emerged as a transformative technique for manufacturing and repairing components. By accelerating metal powders to supersonic velocities, CSAM enables solid-state bonding upon impact, producing dense, near-net-shape components without melting the feedstock material.[5] This low-temperature process minimizes oxidation and sidesteps common issues like evaporation, melting, and recrystallization, making it particularly suitable for oxygen-sensitive materials such as Ti.[6,7]

CSAM is distinguished by its high deposition rates, compatibility with irregularly shaped particles, and potential for efficient powder recycling. Moreover, its ability to create near-net-shape components or repair existing parts positions it as a sustainable and versatile manufacturing solution.[8] However, optimizing CSAM for practical applications hinges on improving the mechanical properties of its products through postprocessing, particularly heat treatments.

This study investigates the mechanical performance of CP-Ti produced via CSAM after various heat-treatment conditions. Beyond conventional vacuum annealing, a novel thermochemical treatment in a hydrogen atmosphere is explored. Hydrogen processing has previously been shown effective in refining microstructure and improving the mechanical performance of Ti alloys,[9] and has demonstrated general capabilities for reducing porosity during the sintering of group IV transition metals.[10] When applied as a posttreatment for consolidated products, this process—thermohydrogen refinement of microstructure (THRM)—has been shown to refine microstructure in Ti-6Al-4V alloys manufactured by laser powder bed fusion[11] and other additive manufacturing technologies.[12] THRM may have substantial implications for CP-Ti, as this class of Ti is not generally strengthened with conventional heat treatments.[3] This work aims to optimize CSAM processes for CP-Ti, paving the way for broader applications and driving innovation in Ti manufacturing.

2. Materials and Methods

Samples of cold sprayed CP-Ti were prepared by Titomic Ltd. using their TKF1000 system. The feedstock consisted of irregularly shaped particles produced by hydride–dehydride processing, which are advantageous for cold spray deposition. Posttreatment, the billets were machined into long cylindrical blanks of 12.7-mm diameter using electrical discharge machining (EDM). The samples were provided as-sprayed (AS) billets and were tested in three different conditions: AS, vacuum annealed (VAC), and THRM.

- AS samples: Delivered in their AS state without any further treatment, serving as the control group for comparison with the heat-treated samples.
- VAC samples: Placed in a vacuum-sealed chamber and heat-treated at 1100 °C for 1 h, followed by controlled cooling. A vacuum environment of less than 10^{-5} torr was maintained to prevent contamination from oxygen and other reactive gases.
- THRM samples: Heated in a furnace under an argon atmosphere to 1100 °C before introducing hydrogen gas and holding at this temperature for 1 h. The temperature was then reduced to 650 °C to allow a dwell period, after which the hydrogen flow was stopped and the temperature was increased to 750 °C under vacuum to remove dissolved hydrogen before cooling.

Metallographic specimens were prepared by mounting approximately 2-mm-thick sections of each rod in a hot-mount phenyl formaldehyde thermosetting powder loaded with graphite for electrical conductivity. Samples were ground with successively finer grades of silicon carbide papers and polished using 9-µm and 3-µm diamond suspensions. Final polishing was performed using an attack polish composed of a 3:1 ratio of 0.04-µm colloidal silica with a 30% hydrogen peroxide solution. Scanning electron microscopy (SEM) was carried out on a Phenom XL scanning electron microscope (Thermo Fisher Scientific) using a backscattered electron imaging mode.

Tensile samples were machined from the 12.7-mm-diameter EDM blanks. Tensile rods conformed to ASTM E8[13] cylindrical standards with a 6.35-mm-diameter gage. Samples were patterned with an airbrush using matte acrylic paint with a coarse black spray over a white background. Patterning was essential for imaging strain fields with 3D digital image correlation. Samples were tested on an MTS Systems 10/GL load frame with strain measurement from an Epsilon Technology Corp. Model 3542 axial extensometer and concurrent measurement with a Correlated Solutions VIC-3D EDU camera system. Testing was performed with a prescribed strain rate of 10^{-3} s^{-1z}. Each sample was tested until failure and fracture surfaces were analyzed by SEM to elucidate failure mechanisms.

Vickers hardness indentations were made using a ZwickRoell hardness tester. A minimum of 10 indents were acquired at a load of 1 kgf in a line across the middle of each sample. This ensured that the entire cross section of the sample could be tested. Optical microscopy was used to verify automated testing results and determine the influence of local porosity on hardness measurements. Density was measured using complementary methods of helium pycnometry and immersion-based (Archimedes) methods with a Quantachrome Ultrapyc 1200e automatic gas pycnometer and a custom-built immersion density apparatus.

3. Results and Discussion

3.1 Microstructural Analysis

Metallographic analysis reveals stark differences in microstructure among the processed materials. Figure 1 presents SEM images of the polished samples, highlighting key features such as grain size, porosity, and overall microstructural variations. Each sample is shown at magnifications of 500× and 5000×, providing a detailed view of the material’s internal structure.

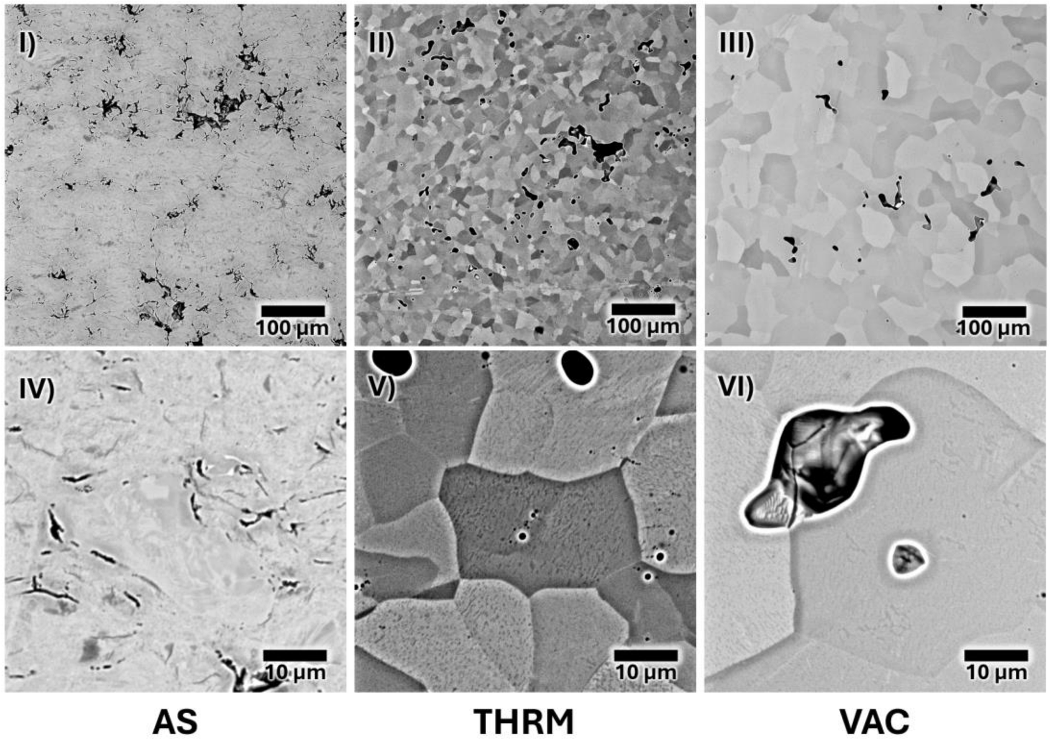

Figure 1. Microstructure of AS (I, IV), THRM (II, V), and VAC (III, VI) samples after metallographic polishing. SEM images were taken in backscatter imaging mode and processed to enhance local contrast to reveal grain structure more effectively.

From visual observation, the AS samples have the highest porosity but also the smallest grain size. The VAC sample has the largest grain size but lowest amount of residual porosity. This can be expected; the AS samples do not receive heat treatment, limiting grain growth and densification. The residual porosity in the AS condition conforms to prior particle boundaries and shows high aspect ratios. For THRM samples, the hydrogen enhances diffusion in the system and pores appear more equiaxed, but significant colonies of pores remain within the region sampled.

These images are then analyzed with the ImageJ image analysis program to compile a dataset of grain sizes for each sample type. Twenty-five random grains are selected, and two measurements are made across the diameter at various angles for each grain and for two separate images, resulting in 100 measurements per sample. The average diameters and standard deviations are reported in Figure 2.

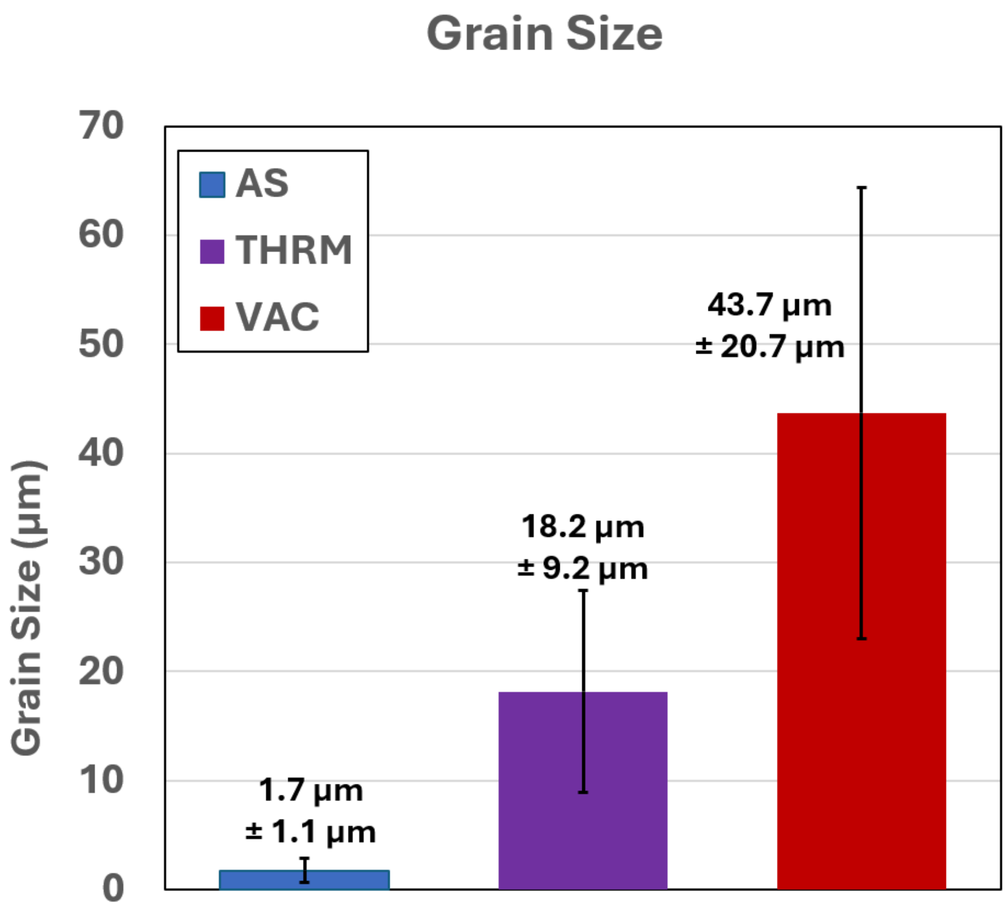

Figure 2. **Average grain size measurements for AS, THRM, and VAC samples.**

The THRM process effectively reduces grain size compared to the AS material, indicating its potential for mitigating grain growth. However, achieving a comparable density to VAC samples may require higher processing temperatures. While THRM reduces porosity relative to AS samples, the pores that remain are more clustered and exhibit distinct morphologies. This contrasts with the VAC samples, where pores are more dispersed throughout the microstructure.

3.2 Density Measurements

Density is a critical factor influencing the mechanical performance of structural alloys, particularly concerning ductility and fracture resistance. These properties are severely affected by the quantity, size, and distribution of porosity. The nominal density of CP-Ti is 4.51 g/cm³.[14] In this study, density measurements were made using both wet-immersion (Archimedes) and gas-based (pycnometer) volume measurement methods, with results presented in Figure 3. The comparison between these two techniques reveals that the results are highly consistent, with overlapping error bands for each sample. This consistency suggests that the porosity within the samples is largely closed, meaning that the pores are not interconnected. The AS sample did not exhibit significant mass or bubble formation increases during the immersion density measurements, further corroborating evidence for closed porosity. Additionally, the pycnometer method demonstrated a lower standard deviation compared to the Archimedes method.

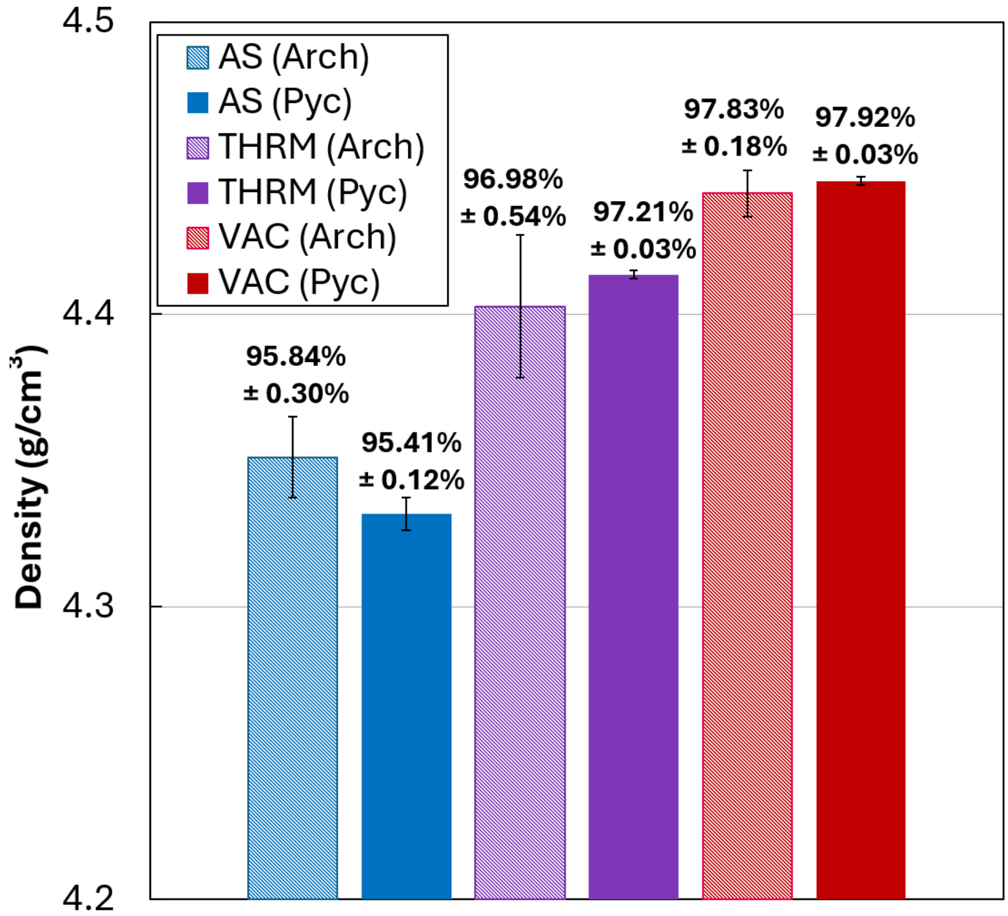

Figure 3. Density of AS, THRM, and VAC samples measured using the Archimedes method and helium pycnometry.

The VAC material showed a greater density than the THRM sample, and both heat-treated samples exhibited considerably greater densities than the AS material. This difference in density between the AS and heat-treated samples reflects the impact of the heat treatments on reducing the overall porosity, which has significant implications for enhancing mechanical performance.

3.3 Hardness

Vickers microhardness testing showed that heat treatment significantly improved the overall hardness and reduced variability in the CSAM CP-Ti samples. As shown in Figure 4, both the THRM and VAC samples exhibited greater mean hardness values and a lower standard deviation than the AS material. Despite having a larger grain size, the VAC material showed a slightly greater mean hardness than the THRM-processed material. However, the overlapping error bars indicated that these differences are not statistically significant.

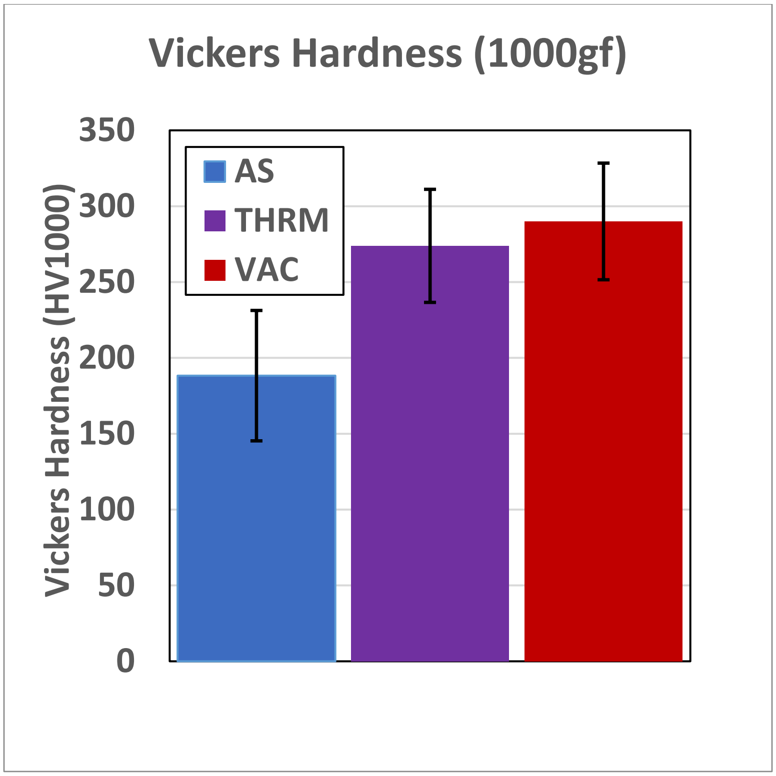

Figure 4. Bar chart of Vickers hardness values for CSAM Ti samples acquired with a load of 1000 gf.

Variability in hardness measurements was substantial in the AS material, primarily due to the influence of local porosity in the sample, as shown in Figure 5. These regions of high local porosity allowed the indenter to penetrate more readily, resulting in larger indentations and more variability in hardness measurements. In contrast, the VAC and THRM samples displayed smaller, more uniform indentations corresponding to greater hardness values. Microscopic images of these heat-treated samples also revealed reduced porosity, which contributed to the more consistent hardness measurements and fewer outliers in the data.

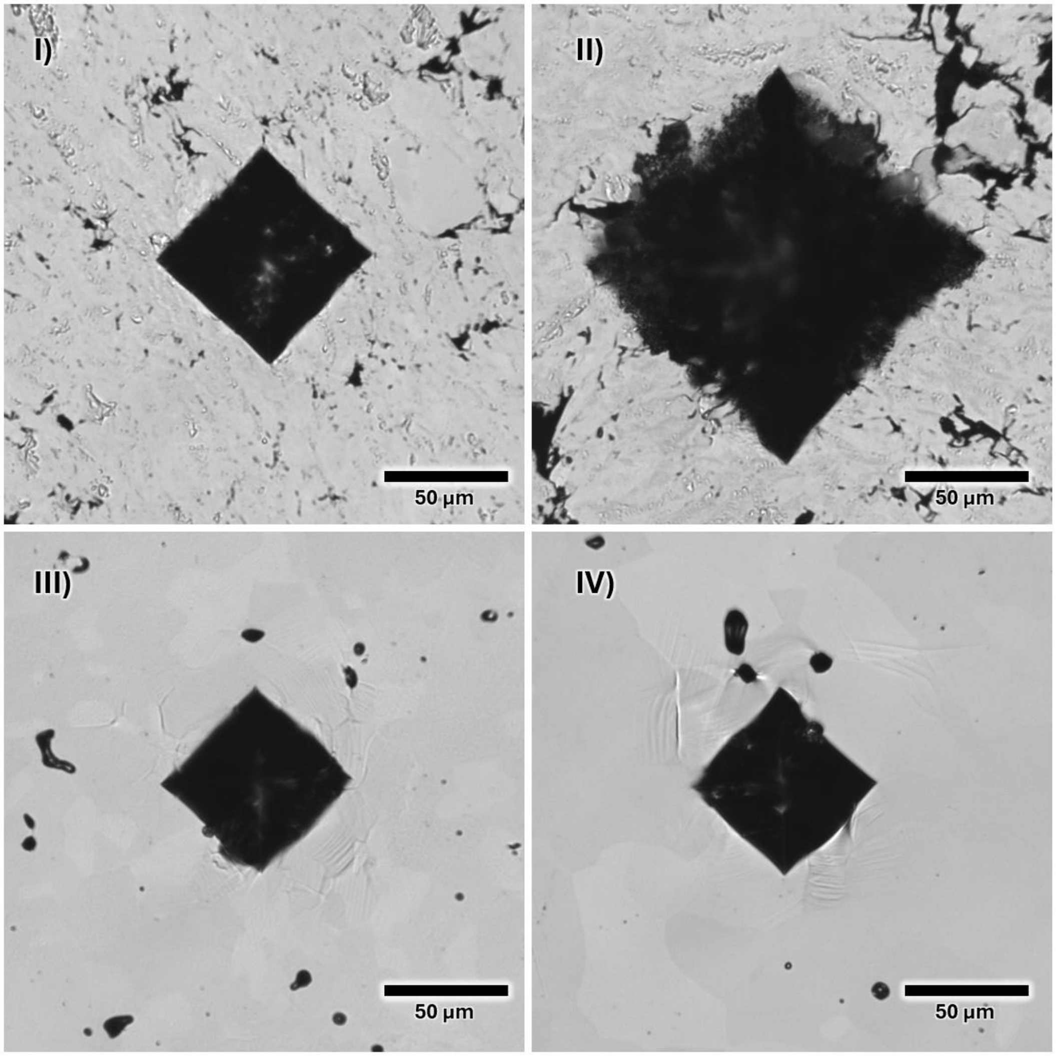

Figure 5. Indentations on AS (I and II), THRM (III), and VAC (IV) samples.

3.4 Tensile Testing

Tensile results showed stark differences between the AS and heat-treated samples. The tensile curves and extracted property data—shown in Table 1 and Figure 6—highlight differences in the ultimate tensile strength, yield stress, elongation, toughness, and Young's modulus. The AS samples showed comparatively poor mechanical performance in all the above categories, and the tensile test results were very close between THRM and VAC. THRM samples showed greater average yield strength, tensile strength, elongation, and toughness than the VAC material. However, the differences were not statistically significant according to a Welch's t-test for the differences between means, assuming an alpha value of 0.05. The P value, a figure of merit representing the probability of observing a difference in a test even though there is no real difference between samples, shows that observed differences between THRM and VAC samples are most likely to be genuine for yield strength and ultimate tensile strength measurements, with P values of 0.156 and 0.366, respectively. A small P value suggests a real difference; a large one means the difference could be due to chance. The limited sample size and high variability of results for the VAC sample may have contributed to the limited ability to detect differences in properties and additional testing should be performed.

Table 1. Tensile properties of CSAM Ti in the AS state and heat treated using THRM and VAC.

Sample	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	Young's modulus (GPa)	Toughness (MJ/m^3)
AS	Fractured before yield	345 ± 2	0.44 ± 0.01	95 ± 2	0.9 ± 0.0
THRM	776 ± 7	829 ± 10	6.84 ± 0.85	121 ± 3	52.8 ± 7.4
VAC	714 ± 24	802 ± 26	6.80 ± 0.85	127 ± 6	50.7 ± 8.3

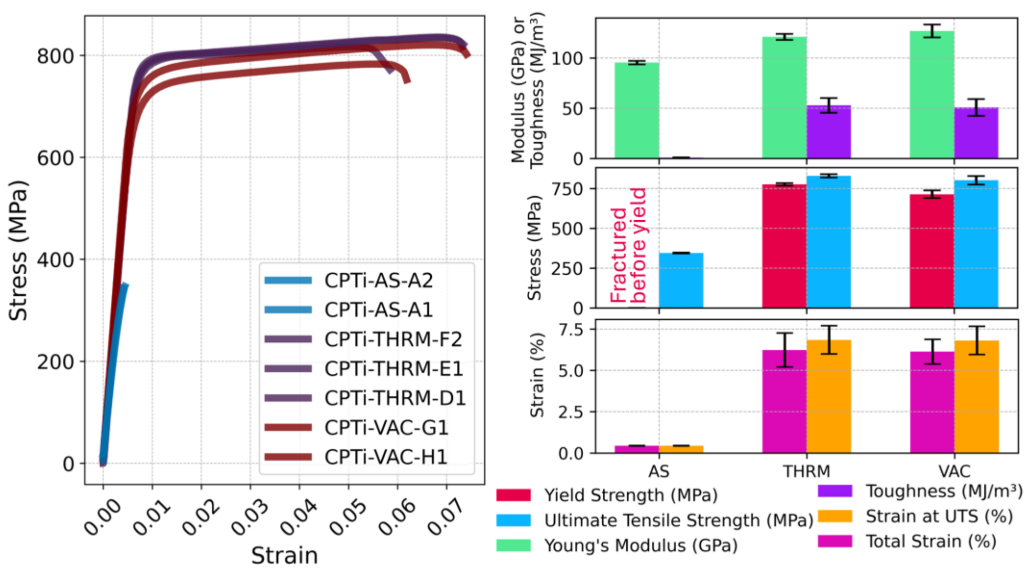

Figure 6. Stress–strain curves (left) and bar charts (right) comparing tensile properties of CSAM Ti.

Cold sprayed Ti does not meet professional standards without heat treatment. It remains too porous and weak in the AS state and consistently underperformed across all tests, exhibiting excessive porosity and insufficient strength and ductility to endure the typical service conditions where high-performance Ti alloys are commonly employed. While the cold spray process shows promise for part fabrication, mechanical performance of cold sprayed Ti only improves significantly after heat treatment. Differences between the THRM and VAC treatments are not statistically significant; however, THRM slightly outperforms in elongation, toughness, yield stress, and tensile strength.

3.5 Strain Maps

The stress–strain curves reveal a limited degree of necking in all heat-treated samples, indicating that the deformation was relatively uniform across the specimens. The 3D strain maps (Figure 7) show a highly uniform strain distribution in the heat-treated samples, ranging from a strain of 0.065–0.08 in the gage and lower relative gradients in strain. This suggests consistent material properties and strong cohesion throughout the specimens. In contrast, the AS sample exhibited significantly lower overall strain and a less uniform distribution of strain. The nonuniform strain pattern in the AS sample indicates the presence of weak points in the material, likely due to incomplete bonding between particles during the cold spray process. These weak points contribute to the reduced strength and ductility observed in the AS sample, as they act as stress concentrators and promote early failure during tensile testing.

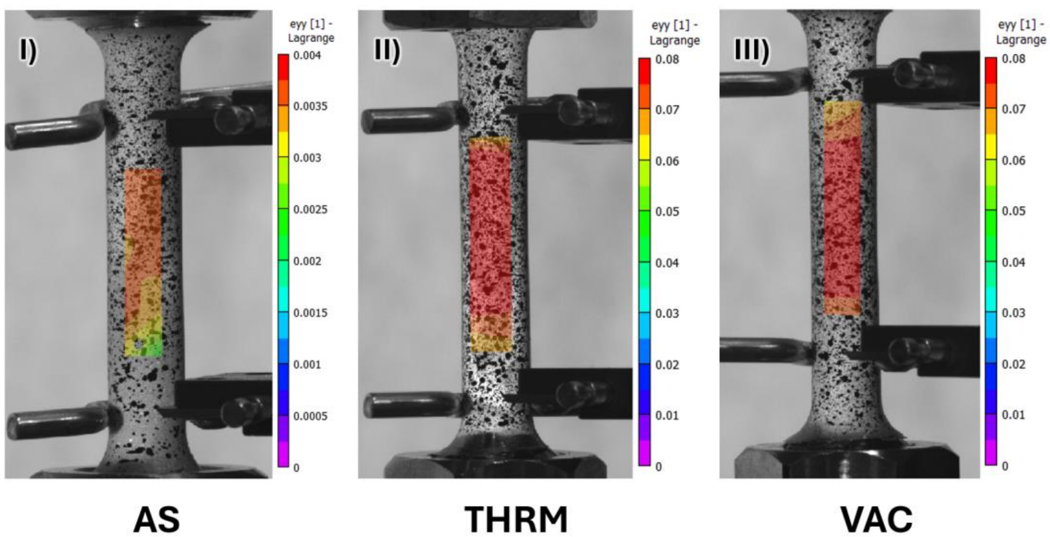

Figure 7. Characteristic strain maps from three representative tensile tests showing the last image before fracture.

3.6 Fracture Surfaces

The fracture surfaces show considerable differences in deformation behavior (Figure 8); AS samples fractured more readily than the heat-treated samples, which is reflected in the flat and faceted surface. Increased porosity and interparticle fracture resulted in a smoother fracture surface in the AS material. In contrast, the heat-treated samples showed considerable dimpling, rough fracture surfaces, and equiaxed pores. The local material response is highly ductile even though there was limited necking before fracture.

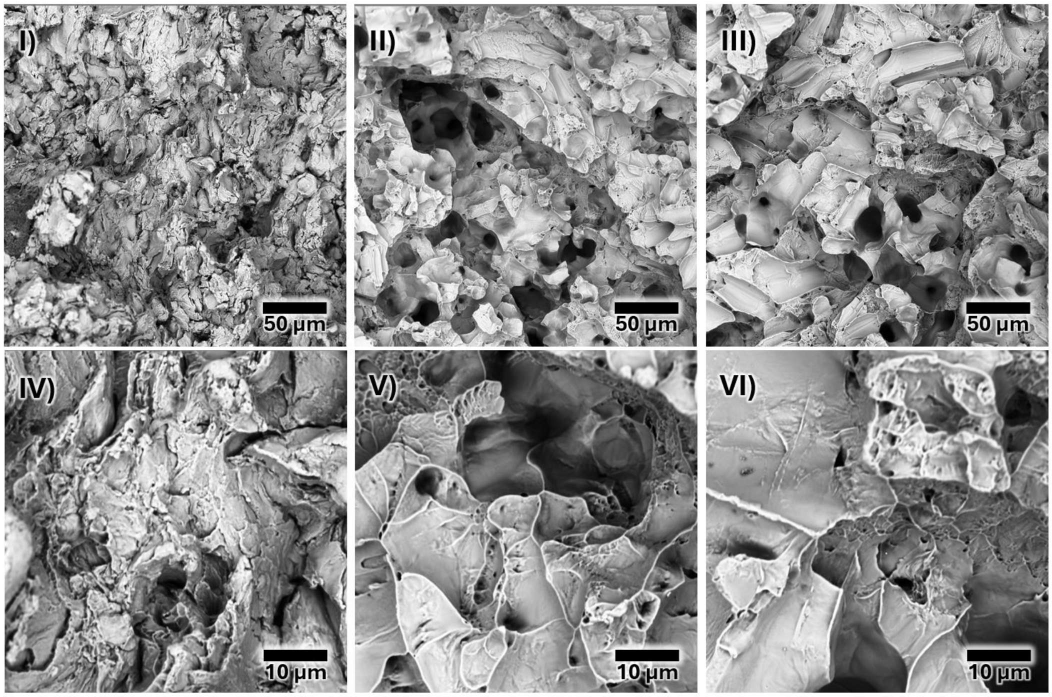

Figure 8. Fracture surfaces of AS (I, IV), THRM (II, V), and VAC (III, VI) tensile samples imaged at 1000× (top) and 5000× (bottom).

3.7 Comparison with Commercial Alloys

Cold spray processes present unique opportunities for the repair and direct manufacturing of Ti components. However, the success of this alternative manufacturing method hinges on the properties achieved in the final product. ASTM standards serve as industry benchmarks for evaluating the capabilities of traditional Ti alloys, providing a valuable reference for assessing new materials and processes. In this context, CP-Ti can be directly compared with commonly used Ti alloys. Among these, the ASTM-designated Grades 4 and 5 Ti[15] alloys are the most comparable to the CSAM Ti examined in this study, as illustrated in Figure 9.

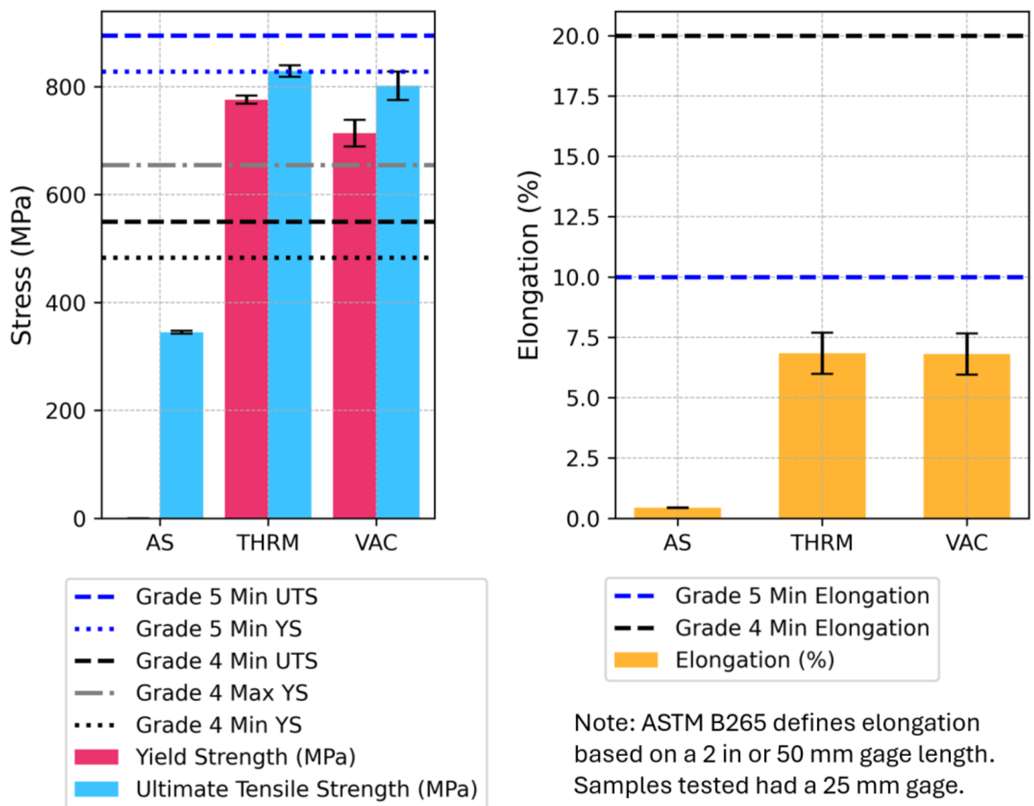

Figure 9. Comparison of properties from CSAM Ti and the ASTM Grades 4 and 5 designations.

Future research should explore increasing annealing temperatures to reduce residual porosity and improve the overall ductility of cold sprayed Ti. Given the wide margin available to soften the material while remaining within ASTM Grade 4 performance metrics, such adjustments could significantly enhance mechanical behavior. Additionally, it is crucial to measure the interstitial oxygen and nitrogen content of these materials to confirm compliance with Grade 4 Ti specifications. The current strength and modulus values suggest that interstitial levels may exceed allowable limits, warranting detailed chemical analysis.

Another key area for investigation is the spatial distribution of porosity inherent to the cold spray process. This study focused on density measurements and metallography from the periphery of the sprayed samples to allow for full-scale tensile testing of specimens. This region is likely to exhibit greater porosity than bulk material. Future work should incorporate density and microstructural evaluations across the entire cross section of the samples to provide a more comprehensive understanding of material uniformity. Addressing these factors will be critical for optimizing CSAM and ensuring the material meets rigorous standards for defense applications.

4. Conclusion

This study demonstrates the potential of CSAM for fabricating CP-Ti components with properties that rival those of traditional Ti alloys. The AS Ti samples exhibited brittleness well below the requirements for Grade 4 Ti with tensile strengths of approximately 350 MPa. However, the mechanical performance improved drastically after heat treatment. Both VAC and THRM samples achieved yield strengths exceeding 700 MPa, tensile strengths over 800 MPa, and modulus values greater than 120 GPa. These values significantly exceed the minimum strength requirements for Grade 4 Ti while falling short of the minimum required strength of Grade 5 alloys.

The strength achieved by the CSAM material is impressive, as it was obtained without the alloying additions typical of Ti alloys. This highlights the potential of CP-Ti as a structural material, particularly given its superior corrosion resistance and low-temperature ductility. However, the ductility of the heat-treated CSAM material remains below the minimum requirements for Grade 5 Ti, although still substantially greater than those for Grade 4. The yield strength of the CSAM material is also considerably greater than the maximum allowable yield strength for Grade 4 components, further emphasizing its enhanced performance.

Another goal of this study was to compare the effectiveness of hydrogen treatment to conventional vacuum annealing in enhancing the properties of Ti. While THRM samples showed modest improvements in strength (likely due to a slight reduction in grain size), no statistically significant enhancements in density or ductility were observed compared with VAC-treated samples. Differences between the two heat treatments, such as the greater toughness of THRM samples and the greater hardness of VAC samples, were minimal and may be considered negligible compared to manufacturing cost and process considerations.

Despite these findings, the mechanical properties reported here are based on basic annealing treatments and represent a starting point for further optimization. With tailored heat treatments or additional postprocessing, there is significant potential to further enhance the strength, ductility, and overall performance of CSAM Ti components.

This research underscores the promise of CSAM as a versatile and cost-effective method for fabricating high-performance Ti parts. The ability to produce strong, corrosion-resistant components from CP-Ti and the potential for further process optimization opens exciting avenues for future engineering applications. CSAM's capability to fabricate and heat-treat components to exceed Grade 4 Ti's mechanical standards demonstrates its potential to disrupt traditional manufacturing approaches and meet demanding application requirements. As this technology evolves, it could address longstanding challenges in Ti machining, including high material costs, while offering unique advantages for additive manufacturing.

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List of Symbols, Abbreviations, and Acronyms

3D	three-dimensional
AS	as-sprayed
CP-Ti	commercially pure titanium
CSAM	cold spray additive manufacturing
EDM	electrical discharge machining
SEM	scanning electron microscopy
THRM	thermohydrogen refinement of microstructure
Ti	titanium
VAC	vacuum annealed

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