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<u>Improving Ti64 reusability using Powder Atomic Layer Deposition</u>

AM Material: Ti64 (Ti6Al4V)

ALD Material: Al₂O₃

Key Material Improvements: Flowability, O₂ Pickup, Yield Strength, Ultimate Tensile Strength (UTS)

Study Highlights:

Using Forge Nano Atomic Armor, coated Ti64 powder exhibited:

- 17% increase in powder flowability
- Surface enhancements with no change to bulk material properties
- Reduced O₂ pickup in extreme environments
- 7% increase in yield and ultimate tensile strength of as built and HIPed tensile bars

Ti6Al4V, commonly known as Ti64, is today's most in-demand alloy for additive manufacturing due to its high fracture toughness, corrosion resistance and biocompatibility. It is mostly sought for load-bearing applications, making robust tensile strength properties of its printed parts paramount.

Due to its popularity, manufacturers have worked to lower the cost of using Ti64 by recycling used feedstock powder back into the powder bed for later printing cycles. However, oxidation and other degradation artifacts from printing limit how much of the used feedstock can be blended with pristine powder.

In <u>additive manufacturing</u>, the surface properties of feedstock powders have large effects on the mechanical properties of printed parts so variability in powder flowability and density, for example, lead to defects in final products, reducing their strength and potentially resulting in failure points.

One strategy to improve the properties of feedstock powders to make used powder more recyclable is to use surface coatings. Ultrathin nanocoatings can be used to engineer the surface of powders with protective barriers without changing the bulk chemical composition of the powder, like with additives.

To investigate the use of nanocoatings as a potential protective mechanism to improve the reusability of Ti64, additive material supplier, Elementum 3D, partnered with Forge Nano.

In this study, we'll explore how Ti64 powder was treated using the Forge Nano <u>Atomic Armor™</u> platform, an ultrathin coating on the order of nanometers. Key powder properties, including flowability and density were investigated, and cube samples and tensile bars were manufactured to understand the effect of the Atomic Armor on the material and mechanical properties of printed parts.



The Atomic Armor™ Platform

Forge Nano's proprietary Atomic Armor is fabricated using a process called <u>atomic layer deposition</u>, or ALD, a precision coating method offering nanometer control over the material thickness and the ability to highly tune material properties.

The ALD process is known as "self-limiting," meaning the deposition reactions always go to completion and result in a film that is defect-free, dense and chemically bonded. It is an excellent candidate for improving AM feedstock powders as the process scales easily and provides excellent performance improvements of raw materials with a coating much smaller than the particle size, ensuring only the surface is affected.

Ti64 flowability and other powder improvements

Figure 1 shows the improvement in Hall flow of the coated Ti64 powder. With less than 10 nm of Al₂O₃, the Atomic Armor surface engineering platform showed a 17% increase in powder flowability. This increase in flowability improves the spreadability of the Ti64 powder in a laser bed, making a more consistent bed that is less prone to causing defects in the final printed products. Without this, the final printed parts are sure to contain non-uniform mechanical properties and potential weak points.

In addition to the flowability changes, the coated Ti64 showed a small increase in density. Ti64 is well-known for its low density so a modest increase of 0.08 g/cc shows well that Atomic Armor does not drastically change the bulk powder properties.

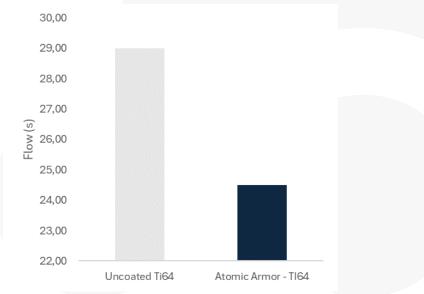


Figure 1. Hall flow time of uncoated Ti64 vs Ti64 coated with Atomic Armor.



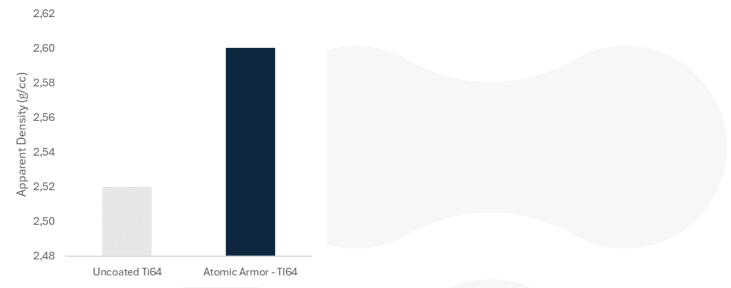


Figure 2. Density comparison of uncoated Ti64 vs Ti64 coated with Atomic Armor.

The dense, defect-free nature of Atomic Armor also functioned as a barrier, preventing additional oxygen pickup of the powder. Qualitatively, the Ti64 powder showed no color change from oxidation after exposure to air at 450 °C for 118 hours (Image 1). Uncoated powder completely oxidized after only 20 hours, evidenced by its black color.

This preliminary oxidation experiment showed that the coated Ti64 powder does not suffer from severe oxygen pickup in a stressed environment. thus, an Atomic Armor-coated Ti64 powder can be reused in the powder bed much more easily. This can decrease the amount of new Ti64 powder needed for each subsequent printing cycle, using each batch of Ti64 more efficiently.

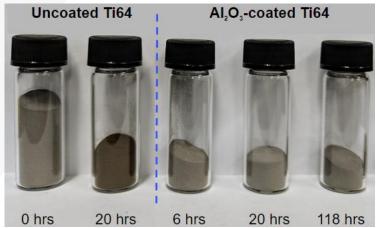


Image 1. Comparison of uncoated and Al₂O₃-coated Ti64 powder after exposure to air at 450 °C. Note that the volume of powder in each vial is arbitrary and not tied to the ALD nor oxidation processes.

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How does Atomic Armor affect bulk material properties of printed parts?

Exploring further, cube samples were printed to show the effect of Atomic Armor on the surface roughness, hardness and microstructure of printed parts. Table 1 shows a comparison of surface roughness based on the degree of printing for both parts made with uncoated and coated Ti64.

Across most orientations, there was a small surface roughness decrease with modest increases when the printing head was oriented at 45 degrees. The surface roughness decreases may be attributed to the enhanced flowability creating a more even powder bed. Improvements in surface roughness may reduce the number of post-printing steps and decrease the amount of Ti64 material wasted.

		Surface Roughness (Ra)		
Degree	Side	Uncoated	Atomic Armor	
40	Up	16.13	13.84	
40	Down	25.04	24.19	
45	Up	13.40	14.16	
45	Down	21.24	22.80	
50	Up	11.61	15.04	
50	Down	21.32	20.98	
90	Up	10.10	9.45	
90	Down	9.91	8.81	

Table 1. Comparison of cube sample surface roughness at different printed head orientations printed using uncoated and Atomic Armor-coated Ti64.

Finally, there was a small increase in hardness from 35.4 ± 1.1 to 36.6 ± 1.0 , and the microstructure of all samples was uniform with consistent electrical conductivity and $\alpha(\alpha')+\beta$ phases present. The coated Ti64 feedstock consistently created printed parts with similar or improved surface properties without changing the bulk functionality of the feedstock.

According to the data, using Forge Nano's Atomic Armor, material suppliers can offer a more consistent, longer lasting and easier-to-handle feedstock with all the same material benefits of a conventional powder.

Mechanical property enhancements of Ti64 printed parts

To test the effect of Atomic Armor on the mechanical properties of parts printed using the coated powder, tensile bars were manufactured using a laser bed powder fusion (L-PBF) method with half the bars being tested as built, and half conditioned with hot isostatic pressing (HIP).

Table 2 shows a summary comparing the mechanical properties of tensile bars manufactured using uncoated Ti64 and bars manufactured using Ti64 treated with Atomic Armor.



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Notably, parts made with the Al₂O₃-coated Ti64 showed a significant increase in strength and ultimate tensile strength for both as built and HIPed conditions. The strength properties of the bars increased about 7%. When HIPed, the bars made with Atomic Armor maintained the same percentage of strength as those made from the uncoated feedstock, showing the coating increased strength without affecting the bulk powder composition.

The treated bars exhibited a modest decrease in ductility which was more pronounced after being HIPed. However, the ductility decrease for the as built bars was within the test's margin of error. There was little effect on the modulus.

Property	Yield, 0.2% (ksi)	Ultimate Tensile Strength (ksi)	Elongation (%)	Area Reduction (%)	Modulus (Msi)		
As Built							
Uncoated	159	183	9	24	16		
Atomic Armor	171	196	8	18	16		
% Change	+7.5	+7.1	-11.1	-25.0	0		
HIPed							
Uncoated	138	153	14	42	16		
Atomic Armor	148	163	12	30	17		
% Change	+7.2	+6.5	-14.2	-28.6	+6.3		

Table 2. Comparison of mechanical properties of uncoated vs Atomic Armor-coated tensile bars. Tensile bars were tested both as built and HIPed.

For a material where strength properties are the most important, the increases in yield and UTS were exciting to measure. These tests are an excellent indicator that ALD not only preserves the key properties of the feedstock powder but can actually enhance them. These results are extremely promising for upgrading brittle materials like Ti64.

Using a more robust Ti64 powder, manufacturers get more flexibility in the design of their products, enabling the printing of parts rated for higher loads or with larger fail points. It can also enable a piece to handle increased amounts of strain-hardening.

To further make the case, Forge Nano will perform metallography and fractography, as well as planned high-fatigue tests on as built, HIPed and machined parts. More in-depth oxidation experiments and reusability studies are also planned.

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Conclusions

Ultimately, Atomic Armor was shown to protect Ti64 powder from oxidation, enhancing its reusability while maintaining all the key material benefits of the feedstock, in some cases improving them.

With a thin nanocoating applied by ALD, the following improvements to the Ti64 powder and its printed parts were shown:

- 17% increase in flowability
- Decreased surface roughness and increased hardness of cube samples with no change to bulk properties
- Reduced O₂ pickup in extreme environments
- 7% increase in yield and ultimate tensile strength of as built and HIPed tensile bars

In this study, Elementum 3D and Forge Nano have shown an excellent proof-of-concept for improving the reusability of additive manufacturing feedstock powders, but the benefits don't stop there. See what just a few nanometers of Atomic Armor can do for your feedstock or parts. Get in touch with a Forge Nano representative today.