Whitepaper

This Whitepaper Gives Answers to:

- What is the metallurgical background of HIP parameter optimization?
- How do different HIP parameters affect the material properties?
- What are the optimized HIP parameters for LPBF Ti64?

Hot Isostatic Pressing for Laser Powder Bed Fusion Ti64

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Optimized HIP Parameters to Improve Static and Dynamic Properties

Additive Manufacturing (AM) of titanium Ti-6Al-4V (Ti64) by Laser Powder Bed Fusion (LPBF) results in a metastable microstructure that differs significantly from that of conventionally manufactured titanium. The high cooling rates in laser melting allow the formation of very fine martensitic microstructure. Because of this different as-built microstructure, it seems likely that the conventional post treatments, including Hot Isostatic Pressing (HIP), could be optimized for improved part properties.

The aim of the study was to investigate possibilities to adapt current standard titanium alloy HIP treatment (920 °C, 100 MPa, 2 h) to better fit LPBF Ti64. This was done by investigating the microstructure of LPBF Ti64 after different HIP treatments and linking the results to the static and dynamic mechanical properties.

The results of the study indicate that the HIP process can be further optimized to gain better results in terms of strength and fatigue properties for LPBF Ti64. It was observed that, with a lower temperature and higher pressure, it is possible to achieve a finer microstructure, leading to higher strength and improved fatigue performance. The results indicate a need for LPBF-specific post-treatments.

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Introduction

Hot Isostatic Pressing (HIP) is a manufacturing and post-treatment method commonly used for densification of powders and solid parts. Heat treatment can be combined with HIPping. HIP post-treatment is also commonly used as a risk mitigation measure for aerospace and medical components manufactured with Laser Powder Bed Fusion (LPBF).

HIP is a process in which components are exposed to a high temperature (up to 2 000 °C) and high isostatic gas pressure (up to 200 MPa) at the same time, enabling densification of the material and modification of the microstructure (Figure 1.). Typical defect types in LPBF (pore, lack-of-fusion (LoF)) can be closed during HIP. This has an especially positive effect on the fatigue properties of the components[1].

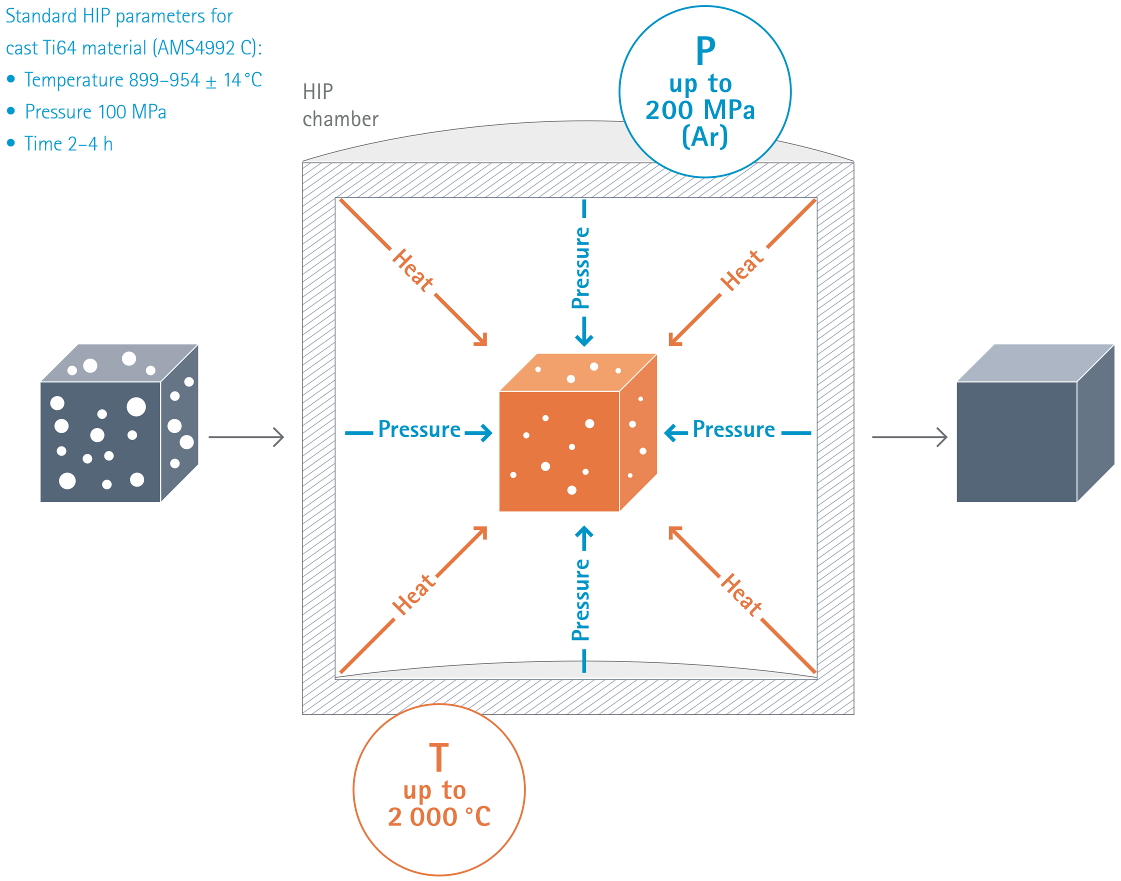


Figure 1. Principle of material densification by Hot Isostatic Pressing (HIP)

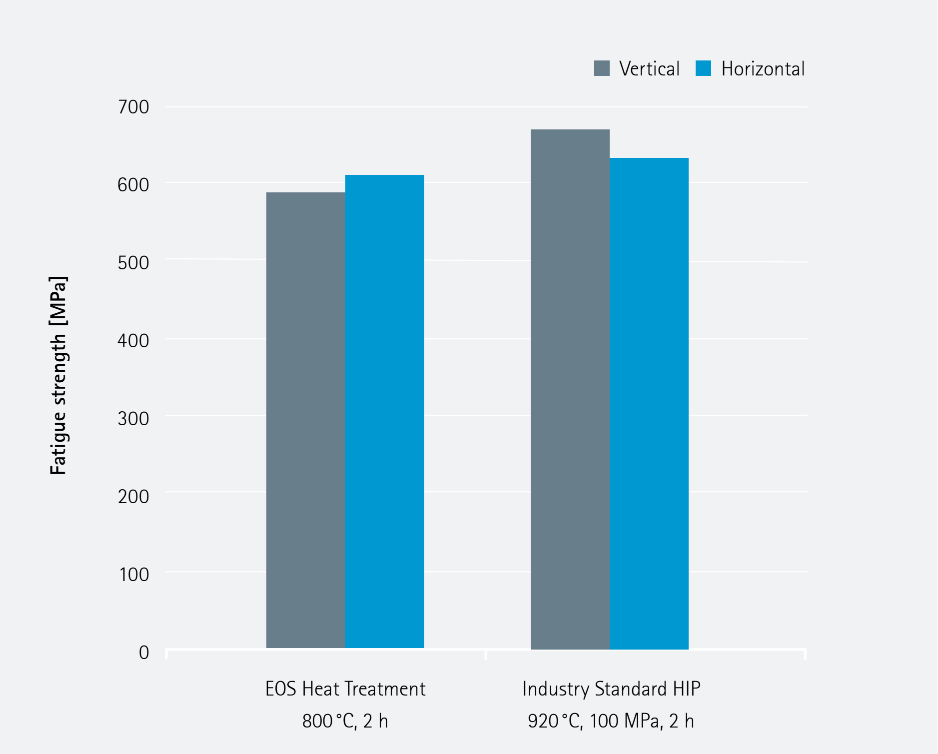


Figure 2. Fatigue strength (10^7 cycles) of EOS Ti64 HiPer 40 µm material in heat treated state and HIPped with industry standard parameters show only a minor difference

Laser Powder Bed Fusion (LPBF) of titanium Ti64 results in metastable microstructure that differs significantly from conventionally manufactured titanium. The high cooling rates in layer wise laser melting allow the formation of very fine martensitic microstructure. The current standard HIP treatment for Ti64 (AMS4992C: Temperature 899–954 ± 14 °C, Pressure 100 MPa, Time 2–4 h) was originally developed for cast material with a different initial microstructure. This HIP cycle is often commercially available with parameters 920 °C, 100 MPa and 2 h. Recent developments in materials, processes and hardware have shown that the difference in material properties between the heat treated and HIPped LPBF material is not as significant as in the past (Figure 2).

This indicates a need to adapt current standard HIP treatment to further improve material properties, especially in industries that still require HIP as a post-process step for risk mitigation.

The aim of the study was to investigate possibilities to adapt current standard HIP treatment to be better suited for LPBF Ti64 material. The effects of individual HIP parameters (temperature, pressure, time) on the static and dynamic mechanical properties and microstructure of the material were studied. As a result of the study, optimized HIP parameters for laser-melted material were introduced and tested with the EOS Titanium Ti64 Grade 5 powder and the 80 µm process.

Experimental Methods

Effect of individual HIP parameters

Test material for the study of individual HIP parameters was built on an EOS M 290 with EOS Titanium Ti64 powder and process (Figure 3). For defect analysis and microscopy, cubes with dimensions of 15 x 15 x 15 mm were manufactured. For tensile tests, horizontal blanks (ø 11.5 mm, length 80 mm) for machining and testing were manufactured according to the ISO6892-1 and ASTM E8 standards.

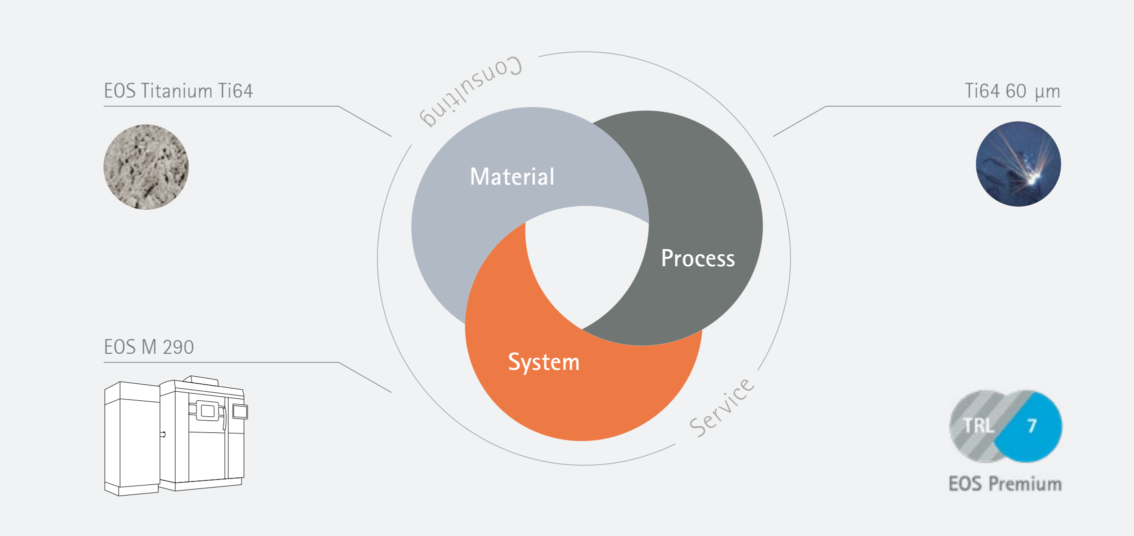


Figure 3: Set-up with EOS M 290, EOS Titanium Ti64 and Ti64 60 µm process

The EOS recommended stress relieving heat treatment for DMLS® Ti64 is 800 °C, 2 h in a vacuum. With this heat treatment, residual stresses of the material are relaxed and at the same time microstructure is modified for better mechanical properties. The fine-grained microstructure of as-manufactured Ti64 is prone to microstructure coarsening at higher temperatures with a consequent decrease in strength. Standard HIP treatment parameters for titanium alloys are 920 °C, 100 MPa, 2 h. This high temperature HIP treatment densifies DMLS material, but also causes microstructure coarsening and a decrease in mechanical properties.

The above temperatures set the boundaries for HIP temperature optimization in this study between 800–920 °C. HIP temperature is the most important parameter to optimize as it has to be high enough to allow diffusion-based densification of the material, but low enough to prevent excessive microstructure coarsening. A lower HIP temperature can be partly compensated by increasing HIP pressure. Modern HIP units work at a maximum pressure of 200 MPa. However, industrial-scale HIP units at commercial service providers are often restricted to pressures of up to 100–140 MPa. This practical point of view sets the limits of HIP pressure. Extending the HIP time can also compensate for a lower temperature. However, for economic reasons it is unreasonable to lengthen the HIP time unnecessarily. Longer HIP time also promotes microstructure coarsening. A summary of tested HIP cycles is shown in Table 1.

Test	Purpose	Temperature T [°C]	Pressure P [MPa]	Time t [h]
Heat Treatment	EOS standard heat treatment for Ti64	800	-	2
HIP - standard	Standard HIPping for titanium alloys	920	100	2
HIP - Test 1	HIP temperature optimization	880	100	2
HIP - Test 2	HIP temperature and pressure optimization	800	100	2
HIP - Test 3	HIP time optimization	800	200	2
HIP - Test 4	HIP time optimization	800	200	4
HIP - Test 5	HIP pressure optimization	800	200	2

Table 1: Summary of tests for individual HIP parameters and reference Heat Treatment

The microstructure and defect content of test materials were studied using optical microscopy. Defect sizes were classified using an image-based defect analysis method. Static mechanical properties of test materials were tested according to standard tensile test methods.

Effect of optimized HIP parameters

Based on the results of the studies of the individual HIP parameters, an optimized HIP cycle was proposed. The performance of the optimized parameters was tested with samples built on an EOS M 290 with EOS Titanium Ti64 Grade 5 powder and process (Figure 4). Table 2 shows the HIP parameters for the proposed optimized HIP cycle.

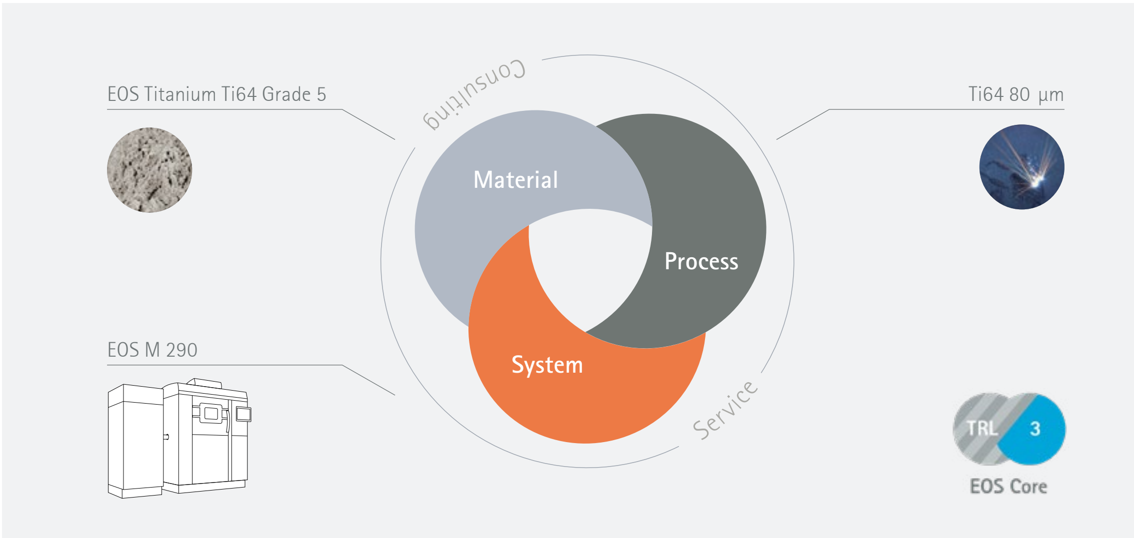


Figure 4: Set-up with EOS M 290, EOS Titanium Ti64 Grade 5 and 80 µm process

Test	Purpose	Temperature T [°C]	Pressure P [MPa]	Time t [h]
HIP - Test 6	Optimized HIP cycle	820	140	2

Table 2. Parameters of optimized HIP cycle

The properties of the samples were evaluated using tensile and fatigue testing. Fatigue testing was performed using axial fatigue testing (ASTM E466) with a stress ratio (R) of 0.1 and a test frequency of 80 Hz. The staircase test methodology (ISO 12107) was applied during tests and data analysis.

Results

The results are presented below by comparing values and micrographs of each test series to (i) EOS standard heat treatment of Ti64 (800 °C, 2 h) and (ii) standard HIP treatment for titanium alloys (920 °C, 100 MPa, 2 h). The results in the tables are averages of three samples (N=3).

Effect of HIP temperature

The HIP temperature optimization test series shows a clear microstructure coarsening effect with increasing HIP temperature (Figure 5). By using a lower HIP temperature, the lath size remains fine, resulting in higher yield strength values compared to standard HIPping (Table 3). According to the common inverse relationship, the elongation is somewhat decreased, but is still well over material standard limit (ASTM F1472).

Heat Treatment / HIP Treatment	Yield Strength $R_{p0,2}$ [MPa]	Elongation A [%]
Standard reference ASTM F1472 min.	860	10
Heat treatment 800 °C, 2 h, EOS Ti64 typical	936	14.8
HIP 920 °C, 100 MPa, 2 h	880	15.2
HIP 880 °C, 100 MPa, 2 h	914	14.9
HIP 800 °C, 100 MPa, 2 h	949	13.3

Table 3. Effect of HIP temperature on the mechanical properties of LPBF Ti64 material: Yield strength (Rp0.2) & Elongation (A)

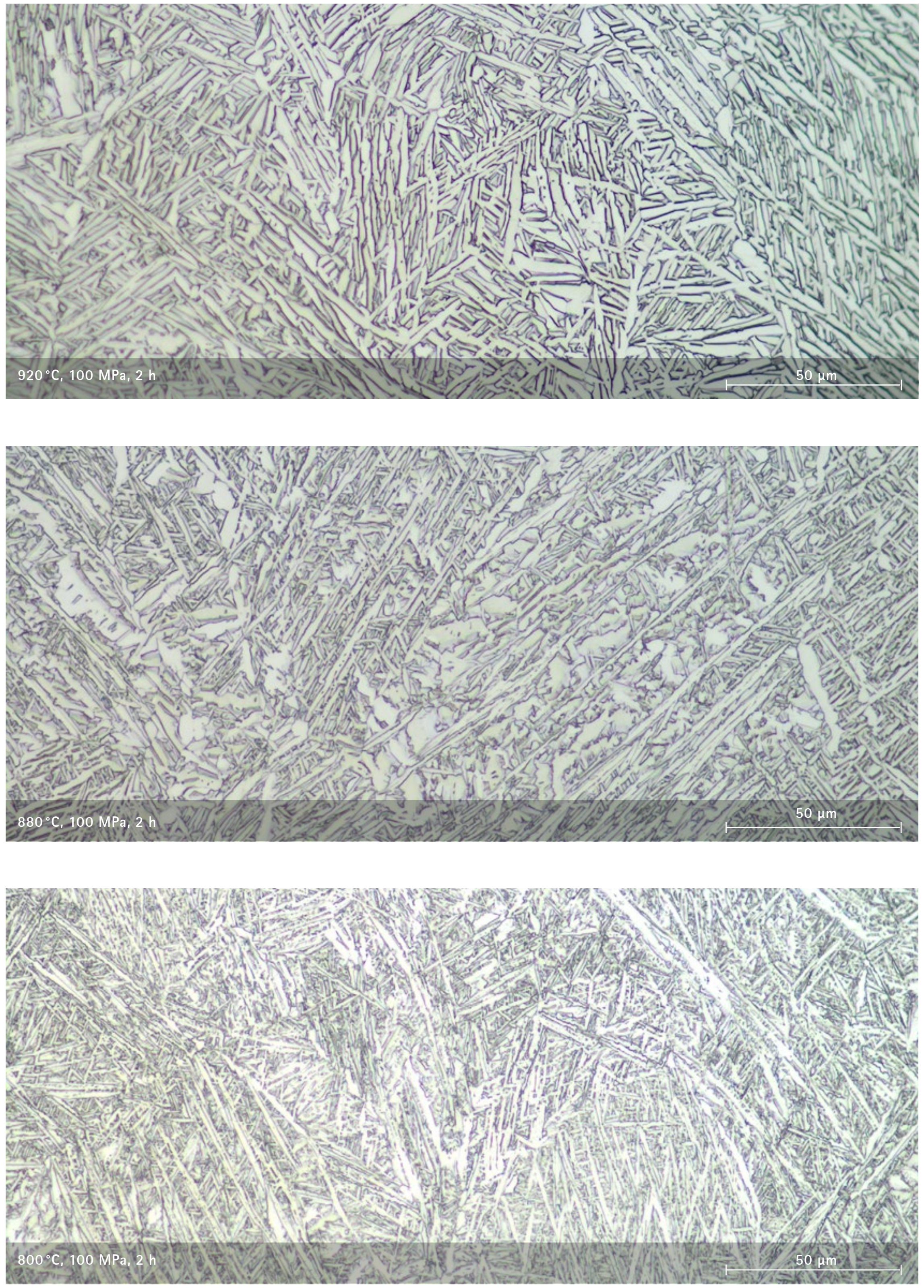


Figure 5. Effect of HIP temperature on the microstructure of LPBF Ti64 material. A smaller lath size is reached with lower HIP temperature

Effect of HIP time

HIP time optimization tests also show a microstructure coarsening with prolonged HIP time (Figure 6). This leads to some decrease in yield strength values (Table 4).

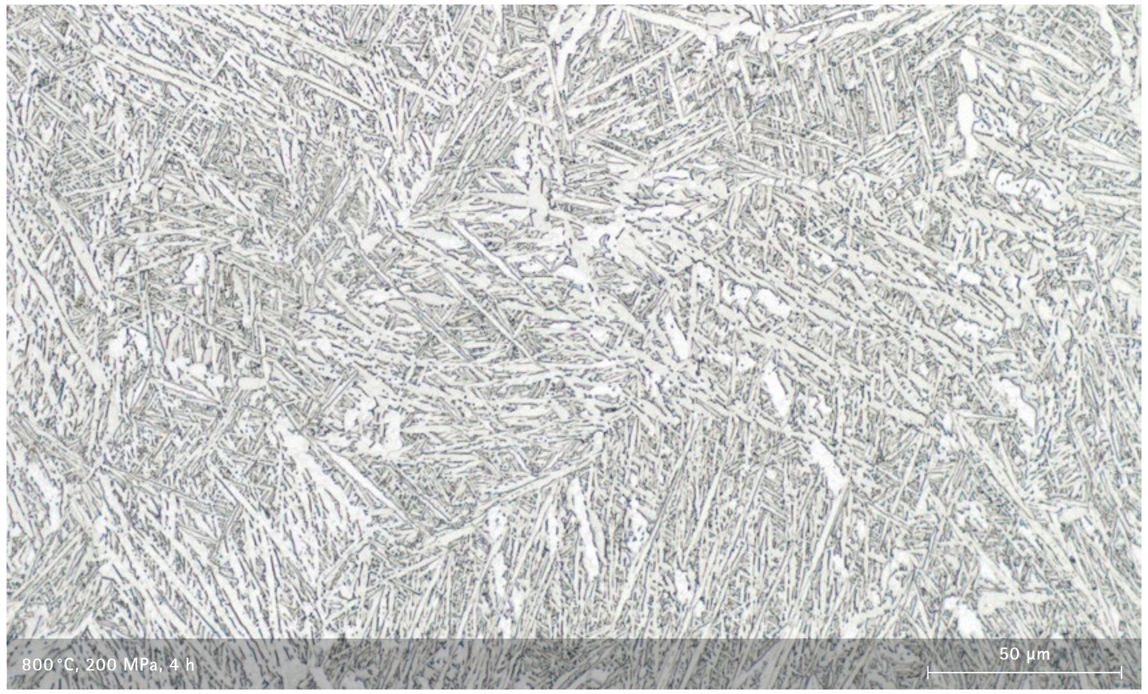


Figure 6. Effect of HIP time on the microstructure of LPBF Ti64 material. Slight lath growth observed with longer HIP time

Heat Treatment / HIP Treatment	Yield Strength $R_{p0,2}$ [MPa]	Elongation A [%]
Standard reference ASTM F1472 min.	860	10
Heat treatment 800 °C, 2 h, EOS Ti64 typical	936	14.8
HIP 920 °C, 100 MPa, 2 h	870	15.2
HIP 800 °C, 200 MPa, 2 h	958	13.4
HIP 800 °C, 200 MPa, 4 h	921	13.4

Table 4. Effect of HIP time on the mechanical properties of LPBF Ti64 material: Yield strength (Rp0.2) & Elongation (A).

Effect of HIP pressure

A HIP pressure increase from 100 MPa to 200 MPa had a minimal effect on static mechanical properties (Table 5). The slightly improved elongation could indicate more efficient closure of defects. There was no noticeable effect on microstructure.

Heat Treatment / HIP Treatment	Yield Strength $R_{p0,2}$ [MPa]	Elongation A [%]
Standard reference ASTM F1472 min.	860	10
Heat treatment 800 °C, 2 h, EOS Ti64 typical	936	14.8
HIP 920 °C, 100 MPa, 2 h	880	15.2
HIP 800 °C, 100 MPa, 2 h	949	13.3
HIP 800 °C, 200 MPa, 2 h	949	14.2

Table 5. Effect of HIP pressure on the mechanical properties of LPBF Ti64 material: Yield strength (Rp0.2) & Elongation (A).

Optimized HIP cycle

Based on individual tests of HIP temperature, time and pressure, an optimized HIP cycle was planned (Figure 7a and Figure 7b). With the parameters 820 °C, 140 MPa, 2 h, a fine microstructure was retained with full densification of the material (Figure 8). This HIP cycle shows higher strength but lower elongation compared to HIPping with standard parameters (Figure 9). The samples have a higher anisotropy in elongation after the optimized HIP cycle, presumably resulting from the lower hold temperature.

Defect analysis (Figure 10) shows a difference in the defect content between as-manufactured and heat treated samples. Given the random variation in crosscuts and the resolution of optical microscopy, this difference can be considered minor.

Both of the HIP cycles effectively reduced the number of both small (< 15 µm) and medium-size defects (> 15 µm), with the result being very similar for both cycles. This indicates that with the optimized HIP parameters a similar degree of densification can be reached as with the standard HIP parameters.

High-strength and defect-free material also results in improved fatigue properties. Significantly higher fatigue strength was reached with optimized HIP parameters compared to standard HIP parameters (Table 6). The fatigue strength of the samples HIPped with the optimized parameters is similar to that of forged material.

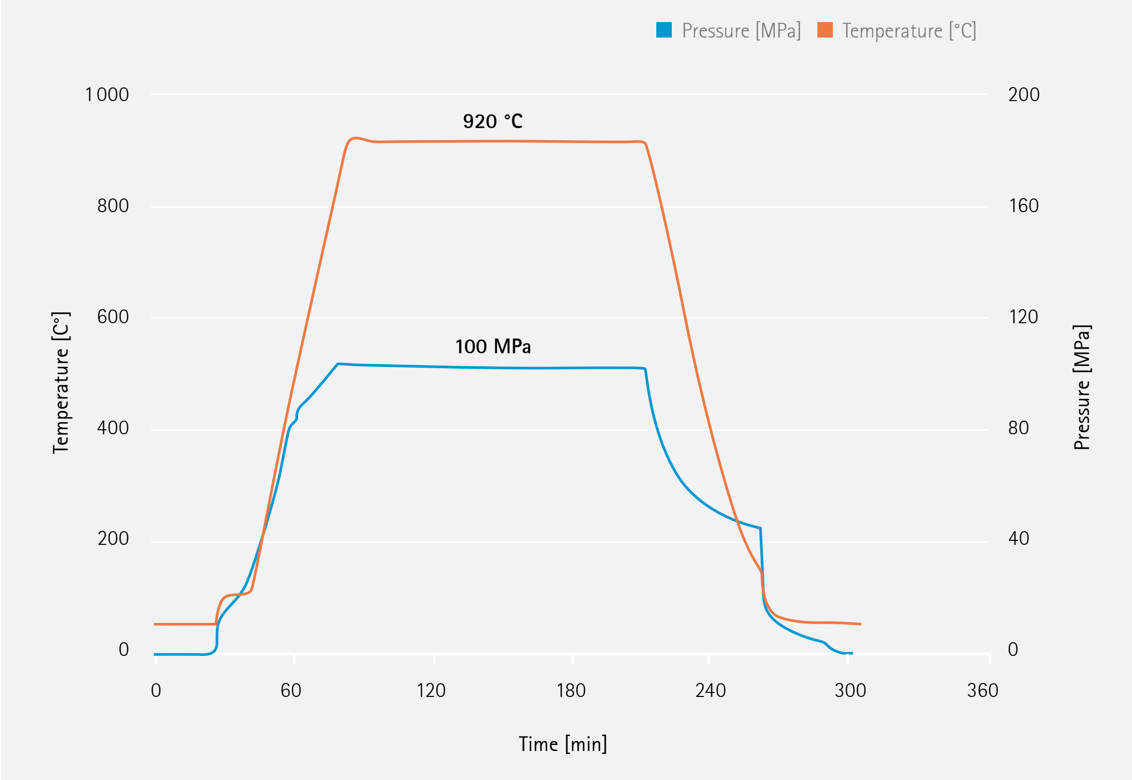


Figure 7a: Standard HIP cycle for titanium alloys (920 °C, 100 MPa, 2 h)

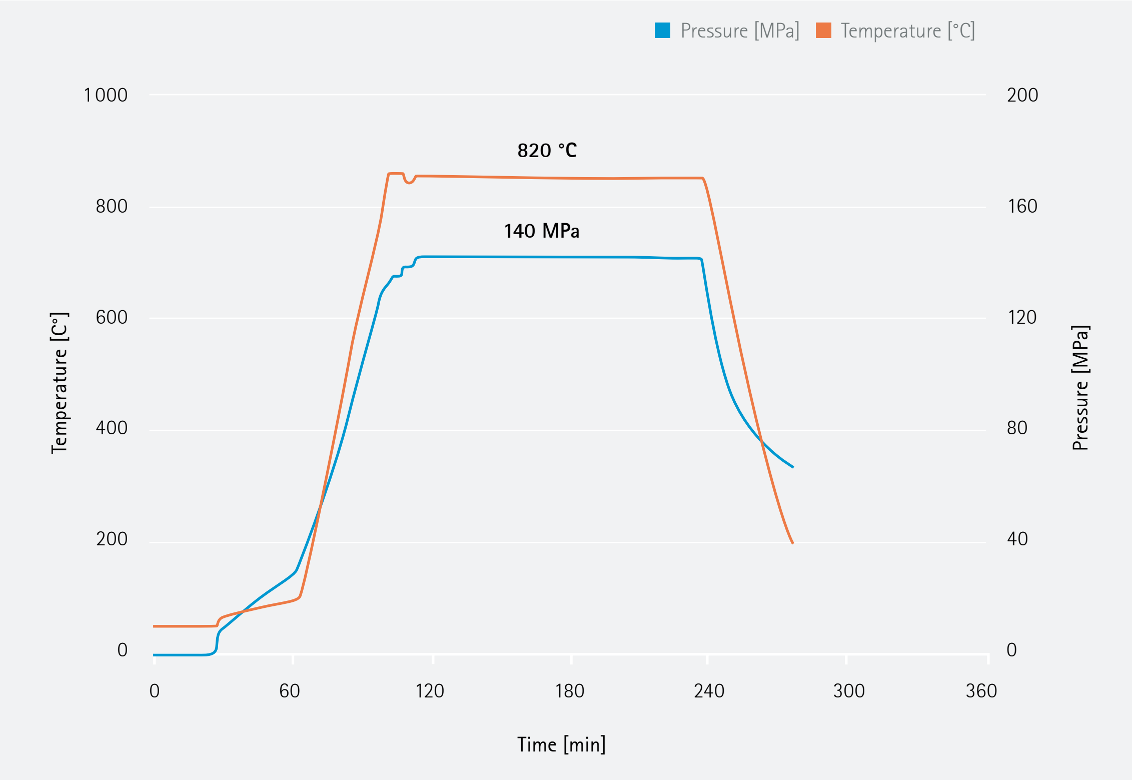


Figure 7b: Optimized HIP cycle for LPBF Ti64 (820 °C, 140 MPa, 2 h)

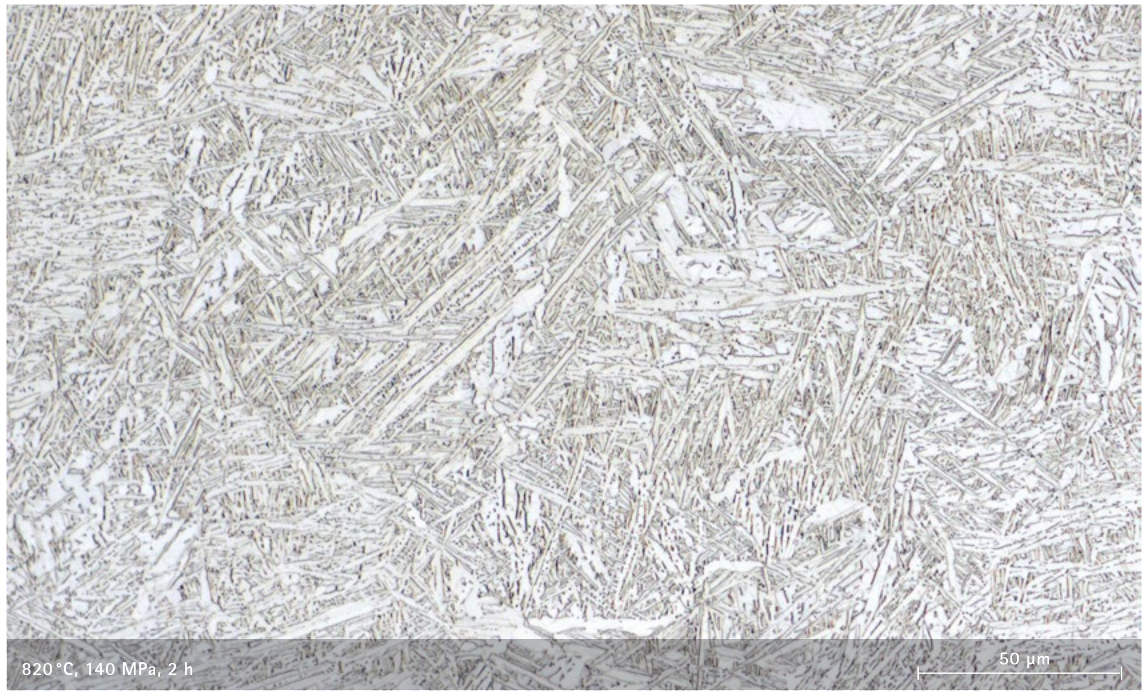
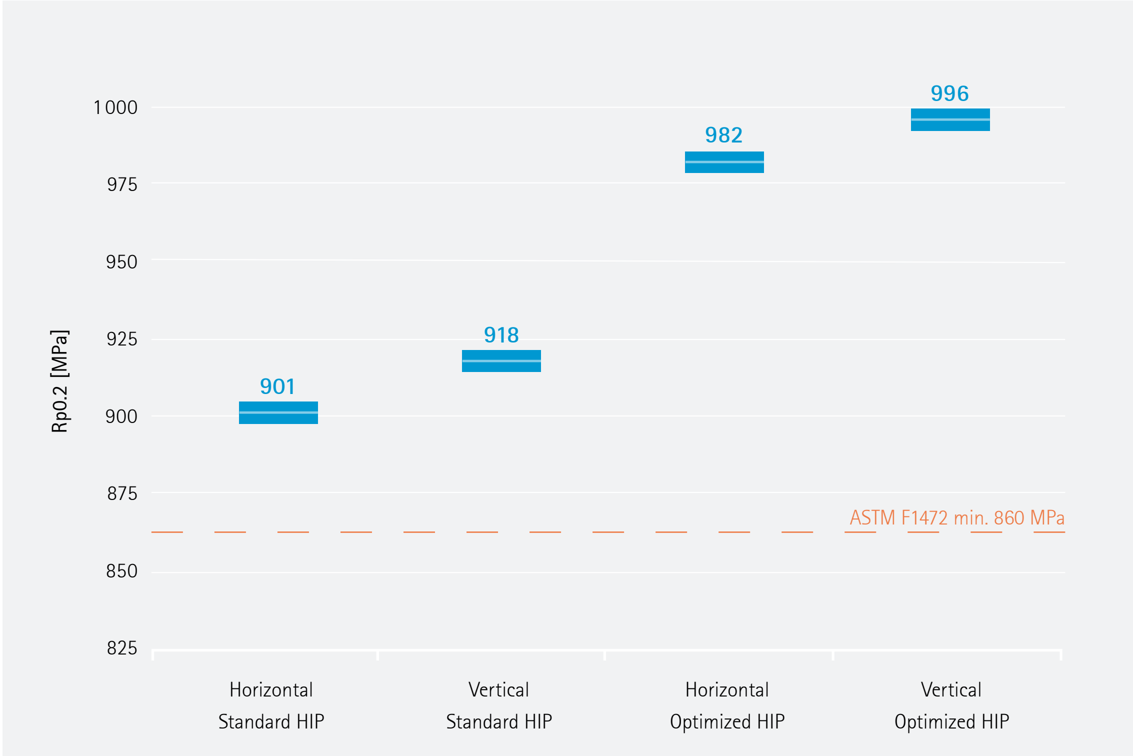


Figure 8. Microstructure of LPBF Ti64 material HIPped with standard HIP parameters (920 °C, 100 MPa, 2 h) and with optimized HIP parameters (820 °C, 140 MPa, 2 h).



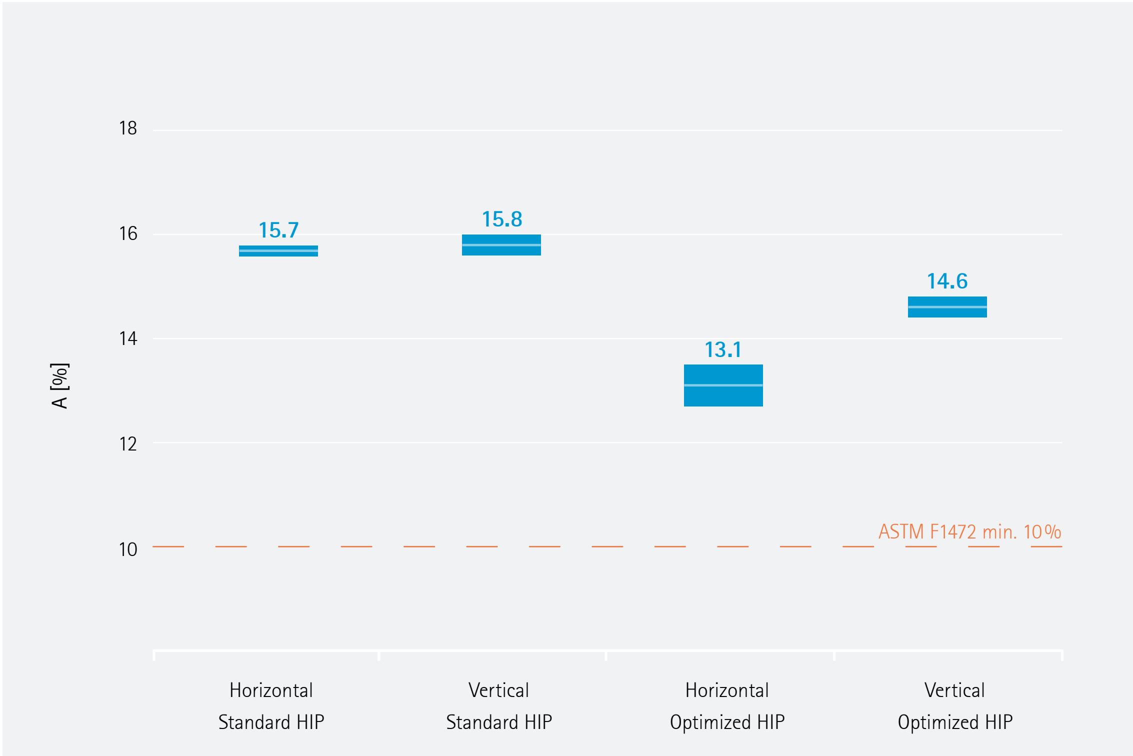


Figure 9. Mechanical properties of Ti64 material post-treated with optimized HIP cycle (820 °C, 140 MPa, 2 h) compared to properties reached with current industry standard HIP cycle (920 °C, 100 MPa, 2 h). Yield strength is higher with the optimized HIP cycle compared to standard HIPping. Elongation is slightly lower but well over the limit of the material standard (ASTM F1472, N=9 or N=11). Average results with 95 % confidence limits.

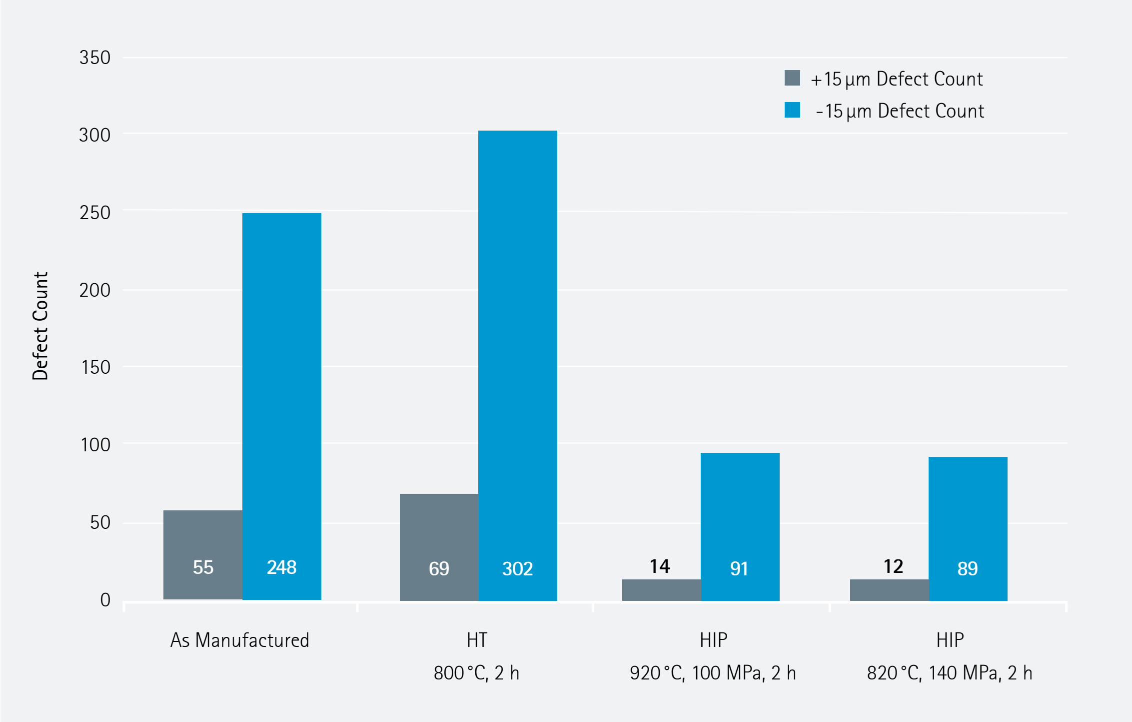


Figure 10. Defect analysis of as-manufactured, heat treated and HIPped LPBF Ti64

HIP cycle	Fatigue strength (10^7 cycles)
Standard (920 °C, 100 MPa, 2 h)	675 MPa, N = 9
Optimized (820 °C, 140 MPa, 2 h)	795 MPa, N = 9
Forged Reference	795 MPa, N = 14

Table 6. Fatigue strength of LPBF Ti64 material with standard and optimized HIP cycle in comparison to forged material.

Typical Ti64 Applications
in Medical and Aerospace

01. Skull Implant. Project Partner: Ceit Biomedical Engineering
02. Prosthetic Hip Socket with a Lattice Structure
03. Latch Shaft. Project Partner: Airbus Helicopters
04. Hinge Bracket. Project Partner: EADS

Titel page: hip stem prosthesis

Conclusions

Recent developments in materials, processes and hardware have shown that the difference in material properties between the heat treated and HIPped LPBF Ti64 material is not as significant as in the past. This indicates a demand to adapt current standard HIP treatment to further improve material properties.

This study of the effect of individual HIP parameters on LPBF Ti64 showed that:

- Higher HIP temperature and longer HIP time lead to coarsening of the microstructure, resulting in lower material strength.
- Increased HIP pressure improves defect closure and reduces deviation in the mechanical properties.

As a result, a new optimized HIP cycle for LPBF Ti64 was introduced: 820 °C, 140 MPa, 2 h. This cycle leads to a finer microstructure and better static and dynamic mechanical properties with full densification of the material. These optimized HIP parameters are also applicable at most commercial HIP service providers.

The EOS Titanium Ti64 Grade 5 80 µm material HIPped with the optimized parameters:

- Fulfilled static mechanical properties for forged material (ASTM F1472).
- Had a low defect content similar to that of material HIPped with standard HIP parameters.
- Showed significant improvement in fatigue strength that was similar to a forged reference material.

The results of the study indicate that the HIP process can be further optimized to gain better results in terms of reducing porosity but also creating the required strength and fatigue properties for LPBF Ti64. The results indicate a need for AM-specific post-treatments.

Acknowledgement: The authors are grateful to Quintus Technologies AB (Västerås, Sweden) for performing HIP test cycles.
Detailed results of this study were presented in AeroMat19, 30th Conference and Exposition:
Kakko, K., Kosonen, T. & Kupi, K. Evaluation of HIP-parameter effects on AM Titanium Ti-6Al-4V. In: AeroMat 19, 30th Conference and Exposition. May 6-8, 2019. Reno, Nevada, USA.

References:
[1] F. H. Kim, S. P. Moylan, “Literature Review of Metal Additive Manufacturing Defects”, Advanced Manufacturing Series (NIST AMS) - 100-16, 2018.

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Topi (M.Sc. in Technology) graduated from Helsinki University of Technology with a major in Applied Materials Science.

His Master's Thesis dealt with Hot Isostatic Pressing (HIP) and principles of metal powder densification during HIPping.

He started his career at the Technical Research Center of Finland (VTT) and worked extensively with new materials and advanced manufacturing technologies.

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