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To the Graduate Council:

I am submitting herewith a dissertation written by Lauren Marie Van Beek Miller entitled “Impact of Manufacturing Pauses in Additive Friction Stir Deposition Aluminum 7075.” I have examined the final paper copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Energy Science and Engineering.

Tony L. Schmitz, Major Professor

We have read this dissertation
and recommend its acceptance:

Brett G. Compton

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Accepted for the Council:

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(Original signatures are on file with official student records.)

Impact of Manufacturing Pauses in Additive Friction Stir Deposition Aluminum 7075

A Dissertation Presented for the
Doctor of Philosophy
Degree

The University of Tennessee, Knoxville

Lauren Marie Van Beek Miller

May 2025

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For Scott and Lily

In loving memory of Don and Deb

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”Wonder is the desire of knowledge.”

- Saint Thomas Aquinas

Abstract

This study investigates the effects of manufacturing pauses on the mechanical properties and residual stress evolution in additive friction stir deposition (AFSD) of aluminum alloy 7075 (AA7075). Single-walled builds were produced using a closed-loop temperature control system, with intentional 15- and 100-hour pauses introduced midway. Some builds were reheated to 120 °C before resumption; others restarted without preheating. Structured light scanning evaluated base plate deflection, and neutron diffraction characterized residual stresses. Samples were sectioned via wire EDM for surface displacement measurements using a coordinate measurement machine (CMM), followed by tensile testing, hardness mapping, and metallographic analysis.

In continuously deposited material, microhardness in the central region increased over 15 weeks post-deposition due to natural aging, with hardness rising from 113 HV to 147 HV. The base plate beneath the deposit consistently showed lower hardness. In paused builds, the material beneath the pause interface aged prior to resumption, leading to elevated hardness in those layers. For example, the 100-hour pause without reheat yielded initial layer hardness of 100–110 HV, versus 90–100 HV in the no-pause sample.

Tensile testing showed that all samples exhibited yield and ultimate strengths between O-temper and T73 conditions, consistent with W-temper natural aging. Elongation at failure ranged from 1.2%–9.1%, with the 100-hour pause without reheat producing the lowest ductility, greatest residual stress, and largest EDM deflection. Neutron diffraction revealed tensile residual stress concentration near the base plate in all builds. Paused builds with warm restarts showed increased tensile stress in pre-pause layers, while cold restarts led to sharp stress variations near the pause. Variations in measured d_0 spacing reflected changes in thermal history and microstructural evolution.

This is the first comprehensive evaluation of AFSD AA7075 with manufacturing pauses. Motivated by delamination observed in large-scale builds, the study shows that each new AFSD layer contributes to in-situ heat treatment. A pause interrupts thermal continuity, allowing premature aging and introducing mechanical and microstructural heterogeneity. These insights are critical for ensuring consistent performance in aerospace-grade aluminum components and refining AFSD process control strategies.

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Nomenclature

$3D - DIC$	3D Digital Image Correlation
η	Equilibrium $MgZn_2$
η'	Metastable $MgZn_2$
ω	Tool rotational speed
AA7050	Aluminum alloy 7050
AA7075	Aluminum alloy 7075
AFSD	Additive friction stir deposition
AM	Additive manufacturing
CMM	Coordinate Measuring Machine
DIC	Digital Image Correlation
FEA	Finite Element Analysis
FSP	Friction stir processing
FSW	Friction stir welding

<i>GP</i>	Guinier-Preston
<i>HFIR</i>	High Flux Isotope Reactor
<i>HIDRA</i>	High Intensity Diffractometer for Residual Stress Analysis
<i>MFR</i>	Material feed rate
<i>ORNL</i>	Oak Ridge National Laboratory
<i>TFR</i>	Tool feed rate
<i>UTS</i>	Ultimate Tensile Strength
<i>XRD</i>	X-ray Diffraction
<i>YS</i>	Yield strength

Chapter 1

Introduction and Motivation

1.1 Introduction

Additive manufacturing (AM) of metals has garnered much interest for industrial applications because it enables the creation of complex metal parts through layer-by-layer deposition. With applications spanning many industries, including aerospace, automotive, medical devices, and tooling, metal AM is revolutionizing the way products are designed and produced, offering advantages such as faster production times, reduced material waste, and the ability to create parts with superior mechanical properties (3; 4). One such technology is additive friction stir deposition (AFSD), a solid-state metal AM process (5). The solid-state nature of AFSD distinguishes the technology from other metal AM processes, enabling faster deposition rates and the AM of different materials compared to many metal AM methods (6; 5; 7; 8). AFSD is based on previous solid-state processing technologies, friction stir welding (FSW) and friction stir processing (FSP) (7; 9; 10).

AFSD is enabled by forcing a rod of metal through a hole in a rotating tool 1-3mm above a base plate or the previous layer. The metal and base plate meet and frictionally heat. As the material heats, it plasticizes and begins to deform and fill in the gap between the base plate and the tool face. Once plastic flow has been achieved, the tool assembly will move in X, Y, and Z to deposit material in a near-net shape geometry. The solid-state nature of AFSD enables deposition of non-weldable alloys such as aluminum alloy 7075 (AA7075) (6; 10). This alloy is the material of choice for this research

1.2 Research Motivation

The practical application of AFSD to generate pre-form geometries for industrial applications involves creating components on the scale of 100+ kilograms over the course of days or weeks. In facilities without continuous operation, AFSD must be paused at the end of the workday and restarted on the next working day (11). Research on the material properties of components created over multiple days is currently limited. This research focuses on (AA7075) AFSD samples produced with intentional pauses in deposition before restarting.

This work was motivated by defects observed in a large AFSD sample produced by MELD Manufacturing over several days, which exhibited visible cracking and failure at the interface between layers where significant manufacturing pauses occurred (Fig. 1.1). After deposition, this sample was sent to the University of Tennessee, where it was machined to a final geometry. Seeing the cracking within the deposit in Figure 1.1 inspired me to study residual stress and mechanical strength in AFSD samples produced with pauses of

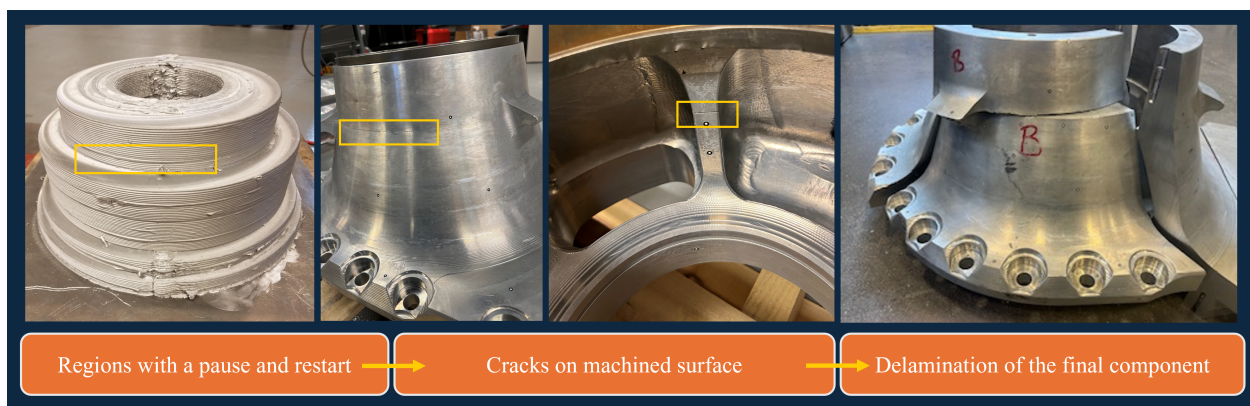


Figure 1.1: Delamination of a large component produced over multiple days which motivated my research. Total component was approximately 845 mm x 508 mm x 286 mm.

approximately 15 and 100 hours, mimicking an overnight or weekend long break in work for a machine operator.

1.3 Current State and Research Gaps

Previous research on 7XXX series aluminum AFSD have focused on mechanical and metallurgical properties, process parameter modeling, repair of grooves and holes, and generation of complex geometries. Early studies of AFSD 7075 exhibit fully dense material with refined grain structure. In 2020, Avery et al. demonstrated AFSD of AA7075 and reported fine grains within the microstructure. They noted heterogeneity in age hardening through the build height of the deposit (12). A study on Aluminum 7050, a comparable alloy, revealed differences in the thermal history regions of the deposit resulted in differences in microstructure and mechanical properties, noting that overgrown strengthening precipitates acted as nucleation sites for voids, causing fracture. Ding et al. demonstrated potential advantages in annealing feedstock material for AFSD prior to conducting deposition (13). Recently, Bagheri et al. studied the as-deposited and post heat treatment microstructure, electroconductivity, and microhardness of AFSD AA7075, finding a significant reduction in mechanical properties when compared to the feedstock material in the as-deposited state which was then improved to 90% of the feedstock material properties by conducting heat treatment on the deposited material (14). Liu et al. achieved extruded AA7075 properties in AFSD material by modifying the post processing procedure from a conventional T6 heat treatment (15).

Practical applications of AFSD include repair of older components and generation of complex geometries for replacing forging and casting components in industrial applications. In repair applications, Griffiths et al. has shown AFSD AA7075 to effectively fill groove and hole volumes in repair applications (16). Wang et al. successfully deposited AA7075 in plate repair in T6 and solution-treated heat treatment states, and found the solution-treated state to effectively fill the repair zone with small, equiaxed grains (17). Stubblefield et al. demonstrated repair of ballistic damage in an AA7075 plate (18). Avery et al. showed promising results in the recovery of wrought-light tensile properties of grooves repaired by AFSD (19). To demonstrate the practical applicability of creating AFSD 7075 geometries, Charles et al. demonstrated the ability to create an aerospace component for legacy aircraft industrial needs (20). Cahalan et al. studied a range of material overlaps which might occur during practical geometry deposition, and found that a larger overlap increases the mechanical strength of the deposited material (21). Shi et al. created two models, a physics-informed machine learning model and a human-AI model, to explore the governing equations and predict the temperature evolution for the tool and build during multi-layer AA7075 deposition (22; 23).

The observations of heterogeneity in mechanical properties of AFSD AA7075 within the current publications and industrial need for production of large, complex geometries present a gap in the available literature on understanding the impact of manufacturing pauses in AFSD AA7075. Expanding beyond AA7075 to other alloy systems, studies characterizing impacts of manufacturing interruptions are limited to a single study conducted by Yakubov et al. on 6061 aluminum (24). There are currently no studies known to this author on AFSD

AA7075 produced with manufacturing pauses, nor do there exist studies on the impact of pause durations on AFSD mechanical properties.

1.4 Research Objective

AFSD of large-scale AA7075 components is relevant to the aerospace and defense sectors. However, the production of these components may require pauses and restarts of deposition, a topic which is currently not explored in the published literature. This dissertation investigates the effects of AFSD pauses on the mechanical properties and residual stress of fabricated components. Specifically, it examines how pauses influence these properties, the impact of pause duration on material performance, and how preheating the material before resuming deposition affects the mechanical properties in interrupted samples. The publication of these results will provide insight for industrial application of AFSD.

Chapter 2

Literature Review

2.1 Additive Friction Stir Deposition

AFSD is a solid-state metal AM process derived from the principles of FSW. Distinguished from other AM processes by its solid-state nature, AFSD mitigates hot cracking, a common issue during the melting and solidification cycle of fusion based AM methods (25; 16; 26). With deposition rates of up to 9 kg/hr, AFSD is significantly faster than other metal AM processes, which range between 0.05-1.8 kg/hr (27; 3). However, AFSD generates excess material along the edges which must be machined away (28). The plastic deformation and heating during AFSD generate a refined material microstructure enabled by dynamic recrystallization (29). Findings of AFSD mechanical properties are mixed, though many studies have found near-wrought properties in the as-deposited material (30; 31). AFSD is of particular interest to the aerospace and defense sectors for the generation of high strength components, replacement components of legacy forged and cast parts, and repair of components (16; 32; 19).

2.1.1 Manufacturing Process

Several feedstock forms and tool configurations are used to enable AFSD, including round rods, square rods, wire, and powder. The most common feedstock and tool setup studied uses a rotating tool with a square hole through which a square rod is fed as shown in Fig. 2.1 (31). In this configuration, AFSD is enabled by the feedstock material heating as the rotating rod is forced down through the tool assembly onto the base plate. Heat is generated in two ways: frictional heat between the rotating rod and tool assembly, and adiabatic heat from plastic deformation within the feedstock. This heat softens the material and it yields, deforms, and begins to flow, filling in the interface between the machine tool head and the substrate. Once material has softened, the machine tool moves to commanded locations in X, Y, and Z, depositing material into a pre-form geometry (33; 5; 25). Key process parameters for AFSD are tool rotational speed (ω), material feed rate (MFR), and tool feed rate (TFR). Parameters vary for feedstock and tool shape and size, and are chosen by iterative testing and optimization (31).

2.1.2 Necessity of Pauses for Large Scale Additive Friction Stir Deposition

AFSD is considered a "lights-on" manufacturing process, meaning that deposition must be monitored by a machine operator and paused if the operator needs to walk away from the machine (11). In facilities without continuous operation, this practically means that manufacturing will be paused for breaks and over night or over a weekend outside of working hours. Additionally, pauses may occur during deposition if the feedstock bars need to be

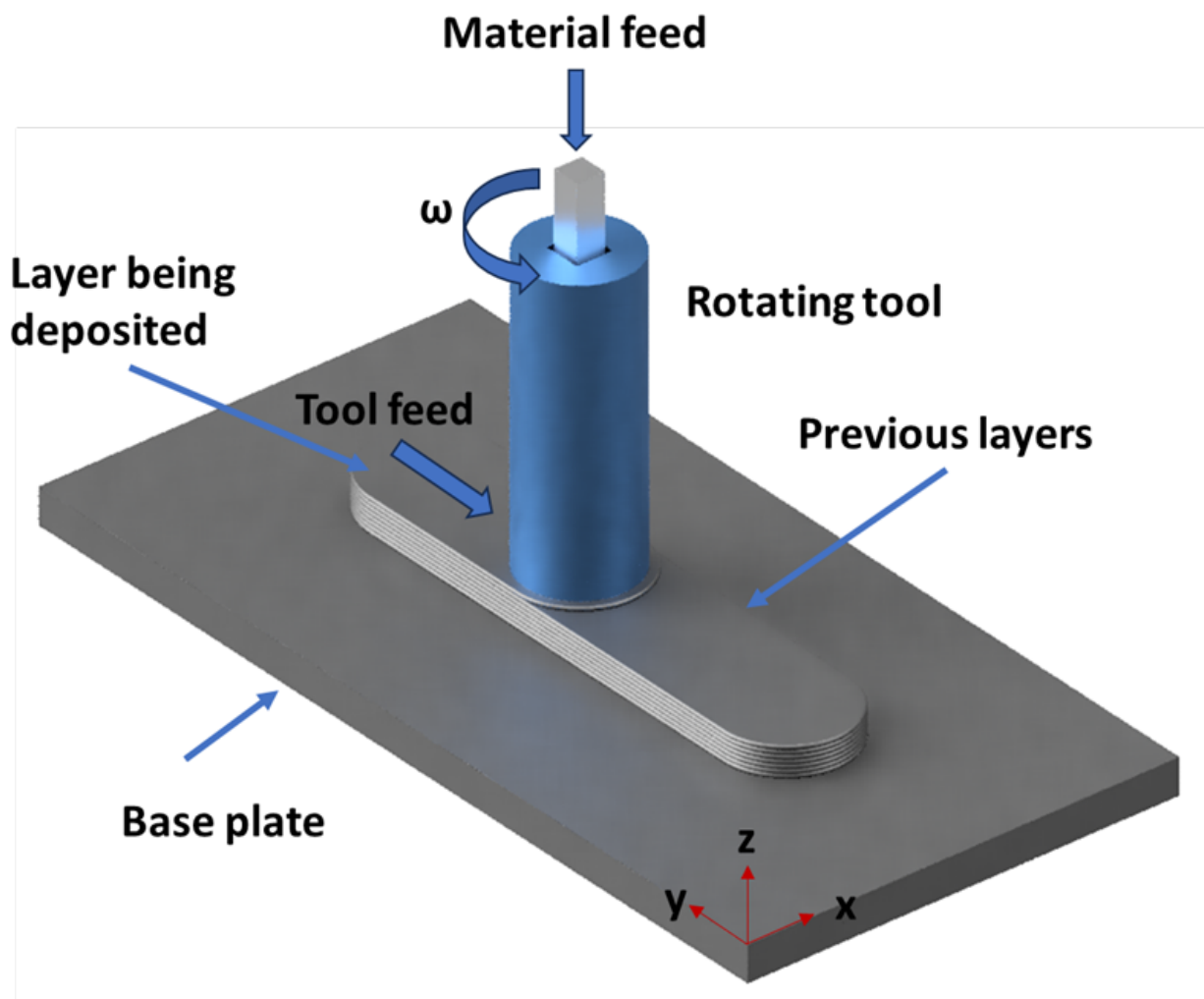


Figure 2.1: Schematic of AFSD, showing the rotating tool, feedstock, base plate, previous layers, and a layer being deposited.

reloaded, which may happen more or less frequently depending on the machine configuration. Certain AFSD equipment is designed to only deposit a single rod of material at a time, while other machines are equipped to load 400+ rods. A final reason why a pause might occur is if there is an issue with manufacturing that requires troubleshooting before continuing the manufacturing process (34).

2.1.3 Impact of Manufacturing Pauses on AFSD Material

The current literature on AFSD material produced with intentional pauses between layers is limited to a single study of AFSD 6061 material published in 2024 by Yakubov et al. In this study, AFSD was conducted to produce a single wall eleven layers high. After a 20 hour pause, deposition was resumed on half of the length of the wall, leaving the other half in the initial state. Microhardness evaluation of this sample indicated that the AFSD material properties stabilize in the deposit, with the highest hardness in the final deposited layers and lowest in the first layer deposited. Tensile properties of the paused sample were not evaluated (24).

2.1.4 Comparisons to Other Manufacturing Processes

AFSD Roots in Friction Stir Welding

The process origins of AFSD come from the solid-state joining process FSW. FSW, established at TWI (UK) in 1991, is a solid-state bonding process enabled by frictionally heating and plastically deforming the material to generate a weld. To achieve FSW, a non-consumable tool is inserted between two sheets or plates to be welded together. The tool

rotates and traverses along the line of the joint. As it traverses, the rotating tool generates frictional heat and encourages mixing between the two materials by plastically deforming and forcing the material to the other side of the weld. Like AFSD, FSW does not melt the material, but rather, it plasticizes and joins the two plates together below the solidus temperature of the material. The solid-state nature of FSW enables the bonding of alloys such as 2XXX and 7XXX series aluminum that are not suited to rapid solidification in fusion welding processes. The AFSD process builds upon the FSW literature, with the addition of a consumable feedstock for depositing layer-by-layer rather than solid-state joining of plates (35).

Comparisons to Other Metal Additive Processes

There are many ways to differentiate between metal AM processes - for the purposes of this discussion, a distinction is made between fusion (melting) based systems and solid state based systems. Fusion AM involves introducing new feedstock material to a base plate or previously melted layer and melting the new material along with some of the previous layer, enabling bonding between the two layers. The feedstock material options for these systems include powder and wire. Arc, laser, and electron energy sources are all used to heat up and melt the material (3). These processes have much interest in industrial and prototyping applications, but there are several limitations to fusion AM. Firstly, there are limitations of alloy composition. As previously mentioned, this work focuses on a structural aluminum alloy which is prone to cracking during rapid solidification and therefore is not suited to fusion AM. There are several metal alloys which are similarly not suited to rapid solidification. Second, many fusion AM processes require specialized feedstock material in powder form, which is

costly to produce and, in certain alloys, must be carefully handled to avoid explosions. These limitations have inspired scientists and engineers to pursue alternative metal AM processes in the solid-state temperature regime (36; 7).

Solid-state metal AM is conducted such that the material stays below the solidus temperature, and thus never experiences melting and rapid solidification. When compared to conventional fusion processes, solid-state material processing has advantages of low heat input and enables bonding between dissimilar materials. Beyond AFSD, key solid-state processes are ultrasonic AM and cold-spray AM (36; 37).

2.2 Aluminum 7075

AA7075 is a high-strength aluminum alloy most commonly used in aerospace and automotive applications. It is an Al-Zn-Mg-Cu alloy which achieves its strength due to precipitation hardening. The primary hardening phases of AA7075 are nano-scale precipitate particles η (equilibrium $MgZn_2$) and η' (metastable $MgZn_2$), and other intermetallic phases such as Al_2CuMg . In peak aged state, AA7075 can reach an ultimate tensile strength (UTS) of 580 MPa and a yield strength (YS) of 500 MPa (38). The high strength-to-weight ratio of AA7075 makes it desirable in structural aerospace applications and other high stress applications (39; 40).

2.2.1 Conventional Manufacturing

AA7075 is conventionally formed via hot-working from a cast ingot. First, the alloy is cast and direct cooled to form an ingot. Ingots are then heat treated to solutionize the

alloying elements and then typically hot-worked via forging, rolling, or extrusion to the desired geometry. After forming, a heat treatment will often be performed to achieve desired mechanical properties for industrial use (39).

2.2.2 Heat Treatments and Age Hardening

In metals, heat treatment refers to the process of heating and cooling material to precise temperatures, creating desired microstructural features and mechanical properties. Four heat treatment states are relevant to this study of AFSD AA7075: O temper (annealed), W temper (solution treated), T6 (solution treated and peak aged), and T73 (solution treated and overaged). The relevant heat treatments and associated properties are summarized in Table 2.1 Each of these heat treatment processes causes variations in metallurgical properties in the alloy, resulting in variation in mechanical strength of components. The heat treatment procedures for achieving these tempers vary for different shapes of base material. For this study, heat conditions are compared to those for heat treatment of forged components, which most closely resembles the shape of the AFSD material studied. (41; 42; 38; 43).

AA7075 is a naturally aging alloy, meaning that in the right condition, the material will continually increase in hardness over time at room temperature. These increases in hardness occur most quickly in the first two hours following quenching (41) and will continue to occur over years (43). Naturally aged material is referred to W temper (Tab. 2.1). For better control and faster achievement of desired properties, artificial aging is often performed. Artificial aging refers to using a multi step heat treatment process to achieve desired mechanical properties. To do this, material will first be revolutionized and quenched,

Table 2.1: AA7075 temper conditions detailing heat treatment procedures, primary hardening mechanisms, UTS and YS, and typical Vickers hardness values.

Temper Condition	Heat Treatment Procedure	Hardening Mechanism	UTS (MPa)	Vickers Hardness (HV)	Ductility
O (Annealed)	Heat to 415 – 440°C, slow cool	Minimal/no precipitates	276	60–80	10–18%
W (Solutionized)	Solution heat treat at 465–480°C, water quench	Supersaturated solid solution, formation of nano-scale η (MgZn ₂) precipitates over time	280–300+	80–100+	$\tilde{8}\%$
T6 (Peak aged)	Solutionize (465 – 480°C), water quench, age at 120°C for 22 hrs	Fine η' (MgZn ₂) precipitates	570–590	160–180	7–11%
T73 (Over-aged)	Solutionize (465 – 480°C), water quench, temper at 105°C for 6–8 hrs, age at 160 – 180°C for 8–24 hrs, air cool	Coarse η (MgZn ₂) precipitates	505–530	140–160	9–13%

and subsequent heating will be conducted to achieve desired hardness. The T6 condition is the peak hardness state, but the overaged T73 is often chosen for its good combination of strength and increase of ductility compared to T6 (38).

The primary mechanism for the increase in strength in AA7075 is through precipitate formation occurring during age hardening. In the annealed condition, η and Al_2CuMg particles are approximately 1-10 μm in diameter and do not provide additional strength. The solutionized and quenched condition of AA7075 saturates the alloying constituents within the bulk α -Al microstructure. Upon quenching, the alloying constituents diffuse to form tiny, coherent clusters, known as Guinier–Preston (GP) zones. GP zones enhance alloy strength. During aging, GP zones grow and from those zones stable nano-scale η precipitates develop, which is why naturally aged material continues to harden over time. In the peak artificially aged T6 condition, nano-scale η' precipitates form coherently with the bulk α -Al microstructure, while the T73 overaged condition promotes the formation of incoherent nano-scale η precipitates (43).

2.2.3 Additive Manufacturing of Aluminum 7075

Additive manufacturing of AA7075 has historically proven to be difficult due to the previously mentioned tendency for solidification cracking (6). However, Martin et al. showed promise for powder bed fusion based metal AM of AA7075 with an introduction of an additional constituent such as Zr, mitigating hot cracking and promoting an equiaxed microstructure (44). Despite the promise demonstrated in this study and the potential to develop AA7075 variants more suited to fusion processing, the demand for AA7075

components without modification of the alloy persists (45). This requirement and the need for repair and replacement manufacturing of AA7075 motivate research in solid-state processes such as AFSD (16; 20; 45).

2.2.4 Additive Friction Stir Deposition of AA7075

Since the inception of AFSD, several studies of AFSD of 7075 and related alloys have been conducted. A 2023 review discussed the potential of AFSD for large scale, near-net-shape production of 7XXX components. The authors highlight previous studies of AFSD material with achievable properties near those of the wrought feedstock (45). Mechanical property results of 7XXX series AFSD studies are summarized in Table 2.2. The first study of AFSD AA7075 was conducted by Griffiths et al. on groove and hole repair applications. This study demonstrated the ability to fill an entire volume using AFSD, with sufficient mixing up to 1.68 mm below the substrate surface. A key finding is a reduction in Vickers microhardness in the deposited material of 15% from 164 HV in the feedstock material to 140 HV in the repaired material (16).

In 2020, Avery et al. published the first comprehensive study of microstructure and mechanical properties in AFSD AA7075. This study showed that AFSD refined the grain structure from the elongated grains in the rolled feedstock material to a refined, equiaxed grain structure with grains on the scale of $\sim 5\mu\text{m}$. This study characterized the age hardening behavior of AFSD AA7075 as region-dependent, where the first deposited layers were overaged and the microhardness remained around 60 HV during the first 35 days post-deposition, the intermediate layers had a moderate increase in hardness from 65 to 85 HV,

Table 2.2: Comparison of mechanical properties (Tensile and Vickers Hardness) of AFSD-produced aluminum 7XXX alloys.

Study (Year) (Ref.)	Alloy	Heat Treatment Condition	Tensile Strength (MPa)	Vickers Hard- ness (HV)
Griffiths et al. (2019) (16)	AA7075	As-deposited (no heat treatment)	–	~140
Avery et al. (2020) (12)	AA7075	As-deposited (no heat treatment)	150 (YS) 317 (UTS)	60-105 after depo- sition, 61-135 af- ter ~35 days
Mason et al. (2021)(46)	AA7050	As-deposited (no heat treatment)	~100-240 (YS) ~240-490 (UTS)	110–140
Yoder et al. (2021) (47)	AA7075	Solution + T6 aged	477 (YS) 541 (UTS)	~180
Avery et al. (2022) (19)	AA7075	As-deposited vs. T6 aged	285/490 (YS) 421/555 (UTS)	210 (heat treated)
Stubblefield et al. (2023) (18)	AA7075	As-deposited re- pair	–	160
Wang et al. (2024) (17)	AA7075	As-deposited T6 feedstock / as-deposited solutionized feedstock / post-heat treated from T6 feedstock deposit	312 / 252 / 491	116 / 135 / 221
Bagheri et al. (2024) (14)	AA7075	As-deposited / T6 heat treatment	297-311 / 464	76-84 / 160-166
Hahn et al. (2024) (48)	AA7050	Solution treated + T74 aged	430 (YS) 491 (UTS)	~170
Cahalan et al. (2024) (21)	AA7075	As-deposited (varied overlap)	229–299 (YS) 258–434 (UTS)	121–135
Rojas et al. (2025) (49)	AA7075	As-deposited re- pair with tool bias tested	Max YS: 304 (advancing bias) Max UTS: 424 (advancing bias)	105–165 (no tool bias)

and the uppermost layers hardness increased from 105 HV to 135 HV. These changes in hardness within the deposited material are attributed to the repeated thermal cycling of AFSD affecting the microstructure and aging properties of the layers. Tensile properties were studied in the build (Z) direction, with the gauge of testing in the intermediate hardness region. When compared to the T651 feedstock, the AFSD material exhibited reduced YS from 525 MPa to 150 MPa and UTS from 583 MPa to 317 MPa after 35 days of natural aging (12).

A 2021 study conducted by Yoder et al. demonstrated a YS of 477 MPa and UTS of 541 MPa in heat treated AFSD AA7075. Heat treatment also resulted in recovery of T6 Vickers hardness to 180 HV. Mechanical loading was conducted in the longitudinal direction of deposition. These values present significant improvement over the as deposited properties previously characterized (47).

A second study on AFSD AA7075 conducted by Avery et al. demonstrated repair of a groove with heat treatment to achieve T6 properties. The YS and UTS improved from 285 MPa (YS) and 421 MPa (UTS) to 490 MPa (YS) and 530-555 MPa (UTS) following heat treatment. Vickers hardness of the heat treated of 210 HV material exceeds the expected peak wrought property 175 HV. This study demonstrated that AFSD with heat treatment could be a successful strategy for repairing commercial AA7075 components (19). A study conducted by Stubblefield et al. also investigated repair applications AA7075 with ballistic damage. This study showed that repaired regions exhibit a decrease in hardness when compared to the base material from 175 HV to 160 HV, and recommends investigations on heat treatment of AFSD material after repair for hardness recovery (18). Another repair study conducted by Wang et al. evaluated the influence heat treatment of the feedstock

before deposition on the subsequent material properties. They found that solution heat treated feedstock results in lower UTS (212.1 MPa) and higher microhardness (135.3 HV), compared to the T6 feedstock UTS (312.6 MPa) and microhardness (116.4 HV). After heat treating the repaired specimen, the UTS improved to 491.2 MPa and the microhardness to 221.2 HV (17).

Recently, Bagheri et al. demonstrated effective T6 heat treatment of AFSD AA7075, recovering 90% of feedstock tensile properties in the longitudinal direction of the build, from as-deposited UTS ranging between 297-311 MPa to T6 heat treated samples achieving 464 MPa UTS.

A 2024 study conducted by Cahalan et al. studied a range of -6.4 mm to 19.1 mm overlaps between parallel passes in AFSD AA7075 and studied the transverse tensile strength and microhardness across the overlapping region of the deposits. This is a very practical consideration, as generation of practical geometries will often involve overlaps, and material conservation is promoted by optimizing overlap width to minimize waste in post-processing. In this study, YS varied from 229-299 MPa (feedstock: 525 MPa), UTS varied from 258-434 MPa (feedstock: 583 MPa), and hardness varied from 121.1-135.0 HV. This study showed that overlap material properties are comparable to those of AFSD AA7075 single wall properties (21).

Recently, Rojas et al. conducted a study on repair of AFSD AA7075 where the tool path location relative to the groove being repaired was varied. Tensile testing was conducted on the samples across the filled grooves. Findings show that the YS and UTS are both reduced compared to the wrought values of 469 MPa (YS) and 524 MPa (UTS). A tool bias toward the advancing side was favorable in terms of mechanical strength, with a YS of 304 MPa and

a UTS of 424 MPa. Hardness was measured across the centered (unbiased) groove fill, with values ranging from 105-165 HV. The softest material is in the heat affected zone below the groove fill (49).

2.2.5 Studies on other 7XXX Alloys

Other 7XXX alloys are concurrently being studied for AFSD. By weight, both alloys constitute a majority Al-Zn-Mg, and therefore have similar microstructural and mechanical properties. Mason et al. conducted AFSD of aluminum alloy 7050 (AA7050), a comparable alloy in composition to AA7075, showed similar grain sizes and grain refinement to those from the Avery et al. study. This study additionally looked at overlap regions of AFSD material, which demonstrated the anisotropic material properties in regions with different thermal and mechanical processing conditions. Tensile samples were extracted in the transverse and normal directions in different regions of the deposit, and a range of values are present. The yield stress ranged from ~ 100 -240 MPa, and the UTS ranged from ~ 240 -490 MPa. Both of these ranges are below those of the feedstock material (46). In 2024, Hahn et al. conducted a study of AFSD AA7050 material. Tensile samples were extracted in the longitudinal direction of the build and mechanical properties were compared to those of the feedstock material. Following an overaging (T74) heat treatment, forging-like tensile properties of 430 MPa YS, 491 MPa UTS, and ~ 170 HV were achieved (48).

2.3 Residual Stress

Residual stresses are stresses that persist in a material structure in the absence of applied loads. They are self-equilibrating stresses within a body which result from thermal, chemical, or mechanical changes to the material structure. Residual stress may be designed into a material structure to achieve a benefit, such as increased fracture toughness, or may be an undesirable result of a forming process that must be mitigated. There are generally three categories of residual stress based on the length scale at which they occur: Type-I stresses occur over multiple grains and up to the entire part scale, Type-II stresses occur within individual grains, and Type-III stresses occur on an atomic scale. This study focuses on Type-I stresses, which are of particular interest in engineering applications as they may cause warping and a decrease in mechanical strength within components (50).

2.3.1 Nature and Origins of Residual Stress

Residual stresses originate in materials as a result of thermal, mechanical, or chemical differences within the material. When two materials with differences in temperature, variation of thermal expansion within the different regions of the material can result in residual stress (50). Practically, this occurs during AM of each layer in most metal AM processes, as hot material is being deposited onto cold material (51). As temperature equilibrates within the material, the hotter regions equilibrate with tensile residual stresses and the cooler regions to compressive residual stresses. The second cause of residual stress in materials is plastic deformation induced stresses. As plastic deformation occurs, the optimal atomic stacking of atoms is compromised, creating mismatches in the crystal and

grain structures of the material. These mismatches create residual stresses within the bulk material. The final cause of residual stresses in materials is chemical changes through the structure. In the case of an aluminum alloy, the alloying constituents in solution within the aluminum lattice will cause a slight expansion (in the case of X or Y) or contraction (in the case of Z). When ageing occurs and precipitates and intermetallic particles form, particles also cause changes in lattice spacing, creating residual stresses (50).

2.3.2 Residual Stress Characterization Methods

Residual stress characterization fall into two categories: destructive (relaxation), and non-destructive testing (52; 53; 54). While often referred to as residual stress measurements, these methods measure changes in strain, lattice spacing, and material deformation values, from which residual stress values may be calculated (52). Destructive methods of residual stress analysis require material removal. These methods include: hole drilling, slotting, contour, curvature, compliance, and digital image correlation (DIC) (52; 55; 50; 56; 57). In hole drilling, slotting, contour, curvature, and compliance, a cut is introduced which relieves the stress in the area local to the cut. As the local stress is relieved, the material distorts. From the observed distortion, the stress field may be reconstructed. Care must be taken to ensure the cutting process does not introduce new stresses to the measured area . Strain gages attached to the surface measure the surface deformations upon relaxing the residual stress. From the measured strain values, residual stress may calculated (56). In contrast to methods measuring distortion caused by a cut, DIC measurements are performed by exposing the surface normal to the plane in which stress measurements are performed (57).

Non-destructive residual stress characterization methods include neutron, x-ray, and electron diffraction methods, as well as ultrasonics (55).

Neutron Diffraction

The most common non-destructive methods of studying residual stress are diffraction methods (50; 56) which rely on Bragg's law:

$$\lambda = 2d_{hkl}\sin\theta \quad (2.1)$$

Bragg's law defines the relationship between the wavelength of the incident beam, λ , diffraction angle, θ , and the lattice plane spacing, d_{hkl} (55; 58; 59). In unstressed samples, d_{hkl} is equivalent to d_0 , the strain-free lattice spacing. Where residual stress is present, the lattice spacing will be slightly larger than d_0 (tensile stress) or smaller than d_0 (compressive stress). From d_{hkl} and d_0 , lattice strain may be calculated (55):

$$\epsilon_{hkl} = \frac{(d_{hkl} - d_0)}{d_0} \quad (2.2)$$

The d_0 value is important to obtain, as localized changes in alloy chemistry, precipitates, and solute concentration may cause changes the lattice parameter and, if unaccounted for, may therefore cause uncertainty in the residual stress calculation. The d_0 value may be measured by stress-relieving a sample and measuring the lattice spacing in the absence of residual stress (56).

When strain values are calculated in the three principle directions, residual stress may be calculated using Hooke's Law (55):

$$\sigma_i = \frac{E}{1 + \nu} \epsilon_i + \frac{\nu}{1 - 2\nu} (\epsilon_x + \epsilon_y + \epsilon_z) \quad (2.3)$$

Where i is the principal direction, E is the elastic modulus, and ν is Poisson's ratio of the material being studied.

Three main types of incident radiation are available to study lattice spacing: electron, x-ray photon, and neutron beams (60). Diffraction data is often refined for study using a least-squares method to fit the measured data points to a curve (56; 58).

Contour Method

In contrast to neutron diffraction, the contour method is a destructive residual stress measurement method, where a component is carefully cut into two pieces. The cut surface is measured to capture the surface deformation that is a result of stress relieved during the cutting process. This measured deformation information is imposed as boundary conditions in a finite element model. The result of this method is a two dimensional residual stress map of the stress normal to the cut face (61).

The material removal process for performing the contour method is typically done on a wire EDM. This process is chosen because EDM does not use force to achieve cutting and therefore minimizes potential errors within the surface as a result of the cutting process. The component should be fixtured such that the part cannot move as a result of relaxed stresses during cutting. After cutting, the surface of both cut faces is measured and averaged, which cancels out any deviations present due to shear stresses and leaves only the stress normal to the cut face to be analyzed. Surface measurement is commonly performed on a coordinate

measuring machine (CMM), which provides the necessary accuracy to capture the surface contours (61).

The contour method is commonly employed in welding literature and is particularly useful when studying highly textured materials which are prone to uncertainty in diffraction measurements (61).

2.3.3 Residual Stresses in Additive Manufacturing

In AM, residual stresses are generally attributed to the steep thermal gradients during deposition. In the case of melt based metal AM, molten metal is deposited onto solid metal. During rapid solidification of the molten metal, the solidifying material contracts, resulting in a tensile stress state in the bulk deposit. Balancing compressive stresses will form in the base plate material to counter balance with the tensile stresses induced during metal AM. The layer-by-layer nature of AM results in an accumulation of residual stresses, which can ultimately cause failure in deposition if the residual stresses exceed the forces required to keep the deposit constrained (62).

Studies have shown that residual stresses in metal AM can adversely affect component performance, as tensile residual stress is associated with a reduction in fatigue performance. Common mitigation strategies for residual stress in metal AM is to heat treat the material post-deposition. The base plate and deposit interface is often a stress concentration area. Efforts to mitigate residual stress in this region include preheating the base plate to reduce the thermal gradient within the bulk material. Additional stress mitigation efforts for metal additive processes include designing the manufacturing process to account for inevitable

distortion and performing intermediate heat treatment during deposition to relieve stresses (62).

Overall, residual stress is a major issue within metal AM, with many studies focusing on characterizing its nature and testing potential solutions for mitigating the stress. Part size and cooling rate both contribute to the overall residual stress development within the material - larger parts and faster cooling rates both result in a higher magnitude of residual stress if not properly managed during deposition. In extreme cases, residual stresses result in part failure (ie. delamination between layers) during deposition (62).

Current State of Residual Stress in Additive Friction Stir Deposition

As additive friction stir deposition is performed below the solidus temperature, many reports state that AFSD material is not plagued with the residual stress difficulties present in melt-based additive processes (31). However, studies performed by us and others have found significant residual stresses within small AFSD components, particularly in the base plate and deposit interface. In 2022, Zhu et al. published the first study characterizing residual stress in AFSD AA6061 with neutron diffraction methods, finding maximum tensile stresses of 91 MPa in the central region of the deposit, and maximum compressive stresses of -127 MPa in the finishing end of the deposit. These stresses are attributed to the part size of AFSD compared to the relatively smaller scale of powder based additive processes, and the significant plastic deformation that is induced during friction based processing (63). Zhu et al. recently expanded upon this research, studying the stress distribution in AA7075 plate repaired with AFSD. In this study, maximum tensile stresses in the center of the weld had a magnitude of 171 MPa, with balancing compressive stresses outside of the weld ranging in

value from -113 to -12 MPa (64). These studies suggest that residual stress is a significant concern in AFSD, despite the lack of fusion-based processing.

These reports are corroborated by experimental data from the Ground Vehicle Systems Center, where residual stresses in large components caused extensive warping in the final material. These data, as well as anecdotal experiences within our lab where deposition has not been completed because the base plate warping caused premature deposition failure, inform us that mitigating residual stress within AFSD is necessary to produce parts on the meter-scale.

Chapter 3

Materials and Methods

3.1 Additive Friction Stir Deposition

AA7075 (Al: bal., Zn 5.1-6.1%, Mg 2.1-2.9%, Cu 1.2-2%, Cr 0.18-.28%, Fe<0.5%, Si<0.4%, Mn<0.3%, Ti<0.2%, wt%) in a T6 heat treatment condition was deposited on a rolled base plate of the same composition using a MELD L3 discrete feed AFSD machine. This study uses both 9.525 mm square and 12.7 mm square bars approximately 508 mm in length, with changes to deposition parameters for each feedstock geometry (Table 3.1). Prior to deposition, a sparse graphite coating was applied to the feedstock material. The experimental parameters were chosen to achieve steady-state deposition while not exceeding the force limits on the machine. In each sample set, AFSD was performed using a tool with an embedded K-type thermocouple. Closed-loop PID control at a user-selected temperature enabled deposition at the selected temperature by modulating the spindle rotational speed, ω . The tool was retracted between each layer and moved back to the starting side, so that the traverse direction was consistent for each layer deposited.

Table 3.1: Processing parameters for different feedstock geometries.

Parameter	Feedstock Cross Section	
	9.525 mm square	12.7 mm square
Rotational speed (ω)	85 rpm – 350 rpm	105 rpm – 350 rpm
Material feed rate	1.8 mm/sec	2.8 mm/sec
Tool feed rate	2.12 mm/sec	3.8 mm/sec
Layer height	1.5 mm	2.0 mm
Number of layers	18	15
Build height	30.5 mm	30.5 mm
Build width	40 mm	40 mm
Build length	190 mm	190 mm
Base plate thickness	12.7 mm	12.7 mm
Feedstock lubricant	Graphite	Graphite
Temperature setpoint	380°C	440°C
Tool composition	H13 tool steel	H13 tool steel
Tool source	MELD Manufacturing	University of Tennessee

3.1.1 12.7 mm Feedstock Parameters

For this experiment, 15 layers of AA7075 were deposited onto a base plate close to the edge of the base plate to allow for minimal post-processing sample extraction time. An image of the deposit is shown in Fig. 3.1. The height of the first layer was approximately 1 mm, and the subsequent layers were deposited at a prescribed height of 2 mm. The tool used for the deposition was manufactured at the University of Tennessee, Knoxville Machine Tool Research Center and is composed of H13 tool steel with 2.29 mm nubs on the face of the tool to promote mixing between layers.

3.1.2 9.525 mm Feedstock Parameters

A series of samples were produced using the 9.525 mm square feedstock to study the impact of manufacturing pauses, which were introduced during deposition between the 9th and 10th layers of select deposits. Each sample was eighteen 1.5 mm layers, with the first layer being 0.7 mm in height. These samples were produced using a commercially available tool for AFSD purchased from MELD Manufacturing composed of H13 tool steel with 2.28 mm nubs on the face to encourage mixing. An image of an example deposit is shown in Fig. 3.2. Select paused samples were reheated prior to restarting deposition using a commercially available welding blanket. These samples were reheated until a thermocouple attached to the base plate of the deposit near the edge of the plate read 120 deg C. The test matrix for these samples is shown in Table 3.2. Duplicate samples for each test condition in the matrix were created for extraction of material to use for various characterization methods.

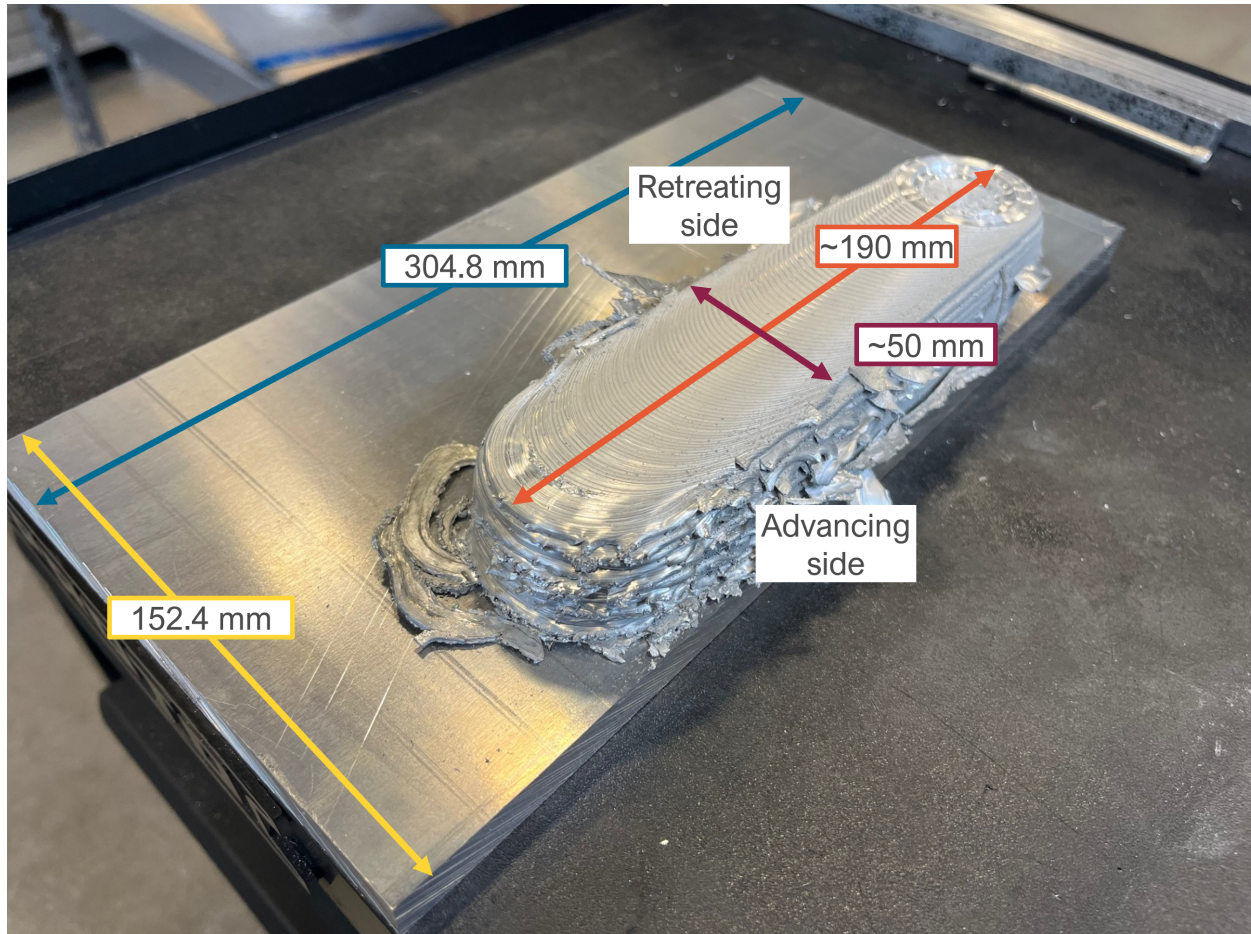


Figure 3.1: Image of the 12.7 mm feedstock deposit before post-processing.

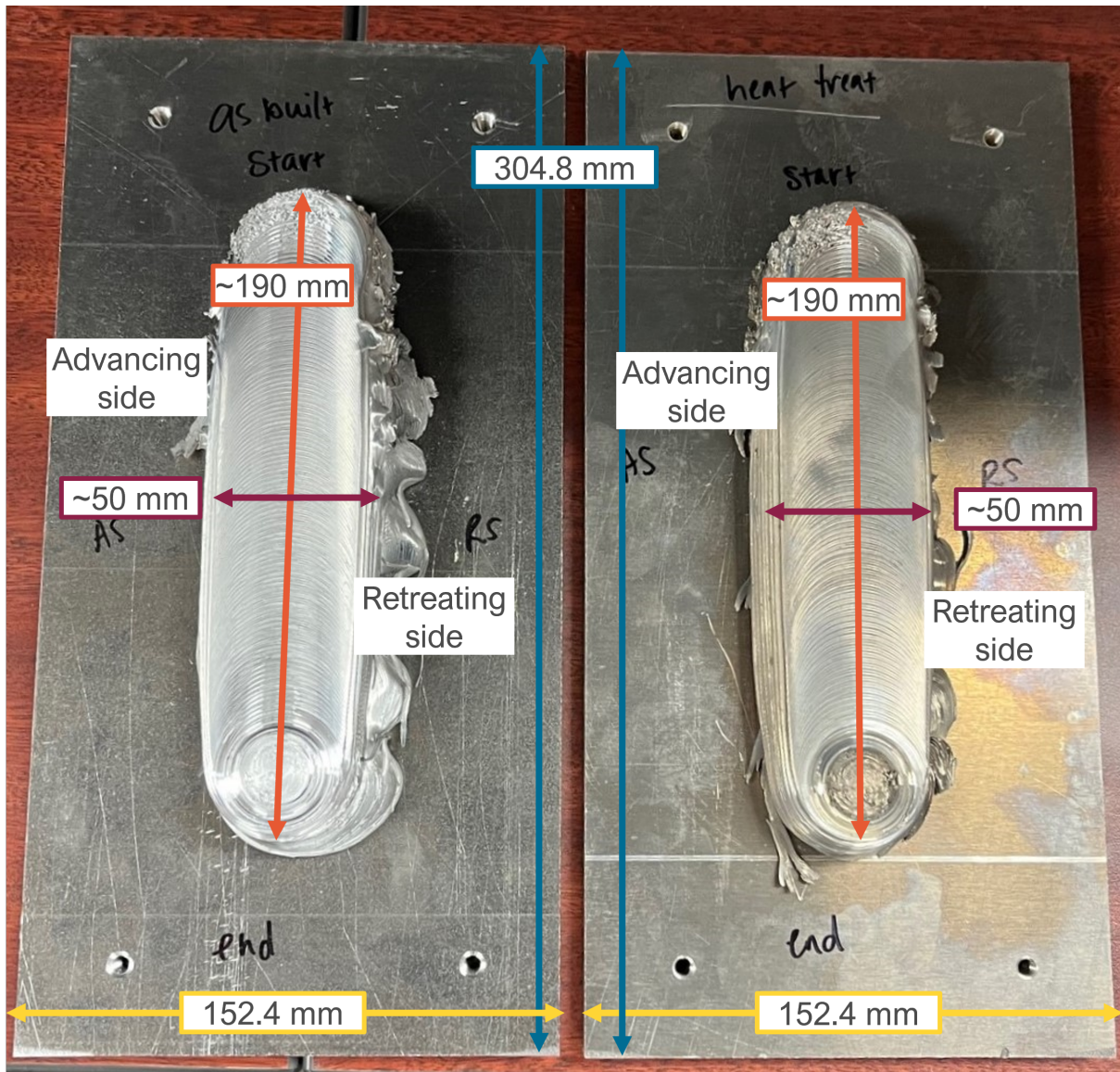


Figure 3.2: Image of two 9.525 mm feedstock deposits before post-processing.

Pause Sample Test Matrix

Table 3.2: Paused sample test matrix with Sample ID noting the duration of the manufacturing pause and the presence of a reheat.

Sample ID	Pause Location	Reheat temperature
No pause	N/a	N/a
15hr, no reheat	Between 9 th & 10 th layer	N/a
15hr, reheat	Between 9 th & 10 th layer	120 deg C
100hr, no reheat	Between 9 th & 10 th layer	N/a
100hr, reheat	Between 9 th & 10 th layer	120 deg C

3.1.3 In-Situ Data Collection

The MELD L3 machine is equipped with sensors and internal data logging at a rate of 1 Hz. The data collected include date, time, spindle velocity (TFR), spindle torque, spindle power, spindle speed (ω), material feed velocity (MFR), feed position, feed torque, path velocity, XYZ position, XYZ velocity, XYZ torque, actuator force, and tool temperature. Optionally, additional temperature data may be collected by installing thermocouples into the machine. On certain samples discussed in this study, thermocouples were attached to the edge of the base plate or embedded into the base plate below the deposit. Machine signal data were collected and plotted using Python and Excel.

3.2 Mechanical Testing

3.2.1 Microhardness

A LECO microindentation system was used to generate 100 and 200 gf Vickers indentations across the polished cross-section of the base plate and deposit material. Samples were extracted normal to the AFSD tool travel direction on a Mitsubishi MV2400 wire EDM. The cut surface of the sample was ground to a final grind of 1200 grit SiC paper and polished using a diamond suspension to a 1 μm finish. The embedded optical microscope with a 50x objective in the LECO microindentation system was used to measure the diagonal lengths of the indents and calculate microhardness of the material according to ASTM E384-22 (65). Indentation size measurements were manually verified for accuracy in the detection software.

Age Hardening Testing

The 12.7 mm sample was indented with 200 gf Vickers indentations at a series of time intervals to characterize the evolution of hardness over time. At each time interval tested, indentations were taken at 400 μm intervals through the base plate and deposited material as shown in Fig. 3.3. The first indentations were taken approximately 10 hours after the deposit was completed and the final set of indentation data was captured 15 weeks after the deposit was completed. The material was kept in a temperature-controlled environment at approximately 21 deg C between hardness tests.

As Built Cross Sectional Microhardness Mapping

Vickers microhardness mapping was performed in the build cross-section. Indentations were applied in a grid of 41 columns by 138 rows at a spacing of 1000 μm across the width of the build and 250 μm through the height of the build (Fig. 3.4) with settings of 100 gf Vickers and 13 seconds dwell time. This testing was performed approximately 6 months after the deposition was conducted. Contours of microhardness values were plotted using Python.

Paused Samples Hardness vs. Build Height

Each test condition described in Table 3.2 was tested for microhardness through the base plate and build height in the central region of the deposit (Fig. 3.5). These data were captured approximately 22 months following deposition for the no pause sample and 15 months following deposition for the paused samples. A 400 μm (Z) by 200 μm (X) grid 4 indents wide and 103-105 indents tall in the center of the deposits was captured. Hardness values were plotted using Excel and Python.

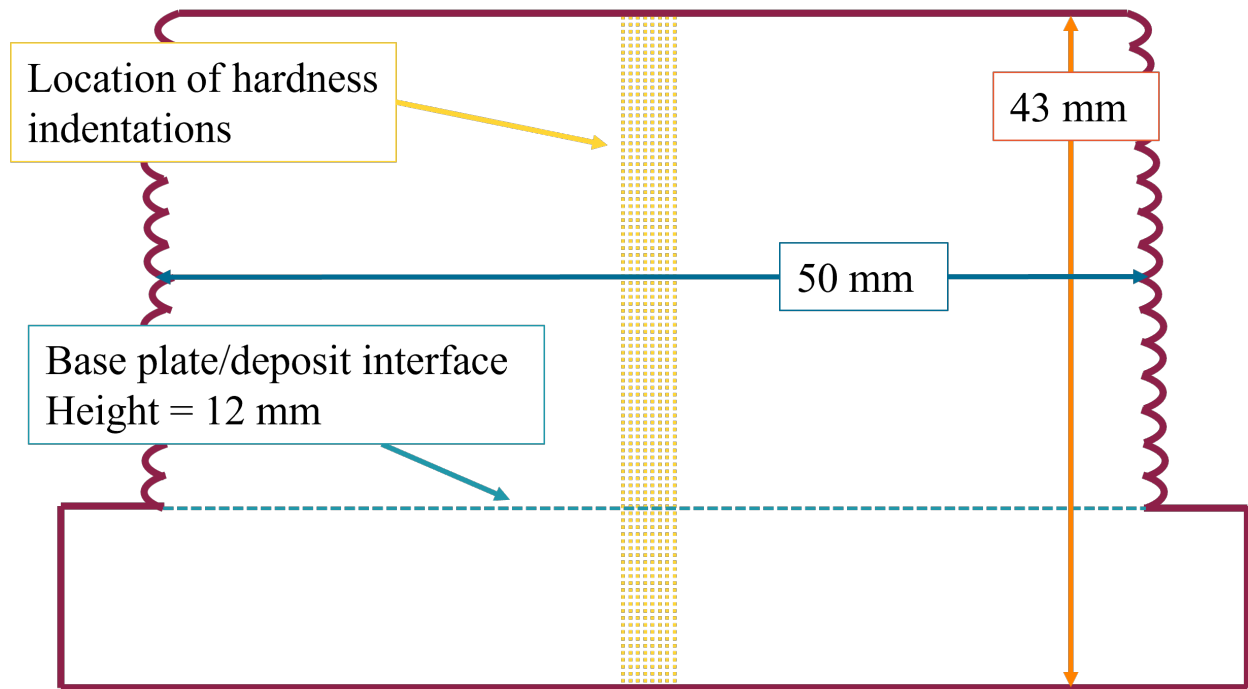


Figure 3.3: Schematic of microhardness indentation locations in the central 3.6 mm of the deposit. At each time of testing, a series of indentations spaced $400\text{ }\mu\text{m}$ apart were taken through the base plate and deposited material. Subsequent tests were spaced $250\text{ }\mu\text{m}$ horizontally from previous tests.

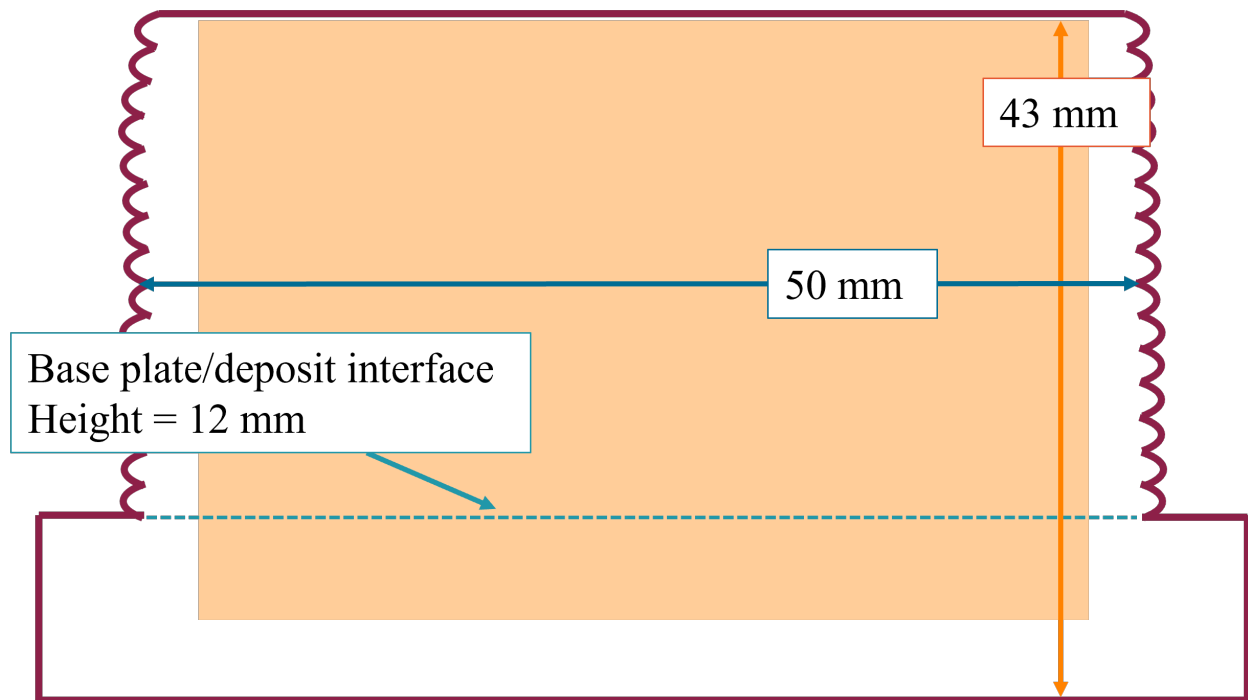


Figure 3.4: Schematic of microhardness indentation locations through the width of the no pause AFSD build with the 9.525 mm deposition parameters. Indentations were spaced 1 mm apart in the width the build and .25 mm apart in the height of the build.

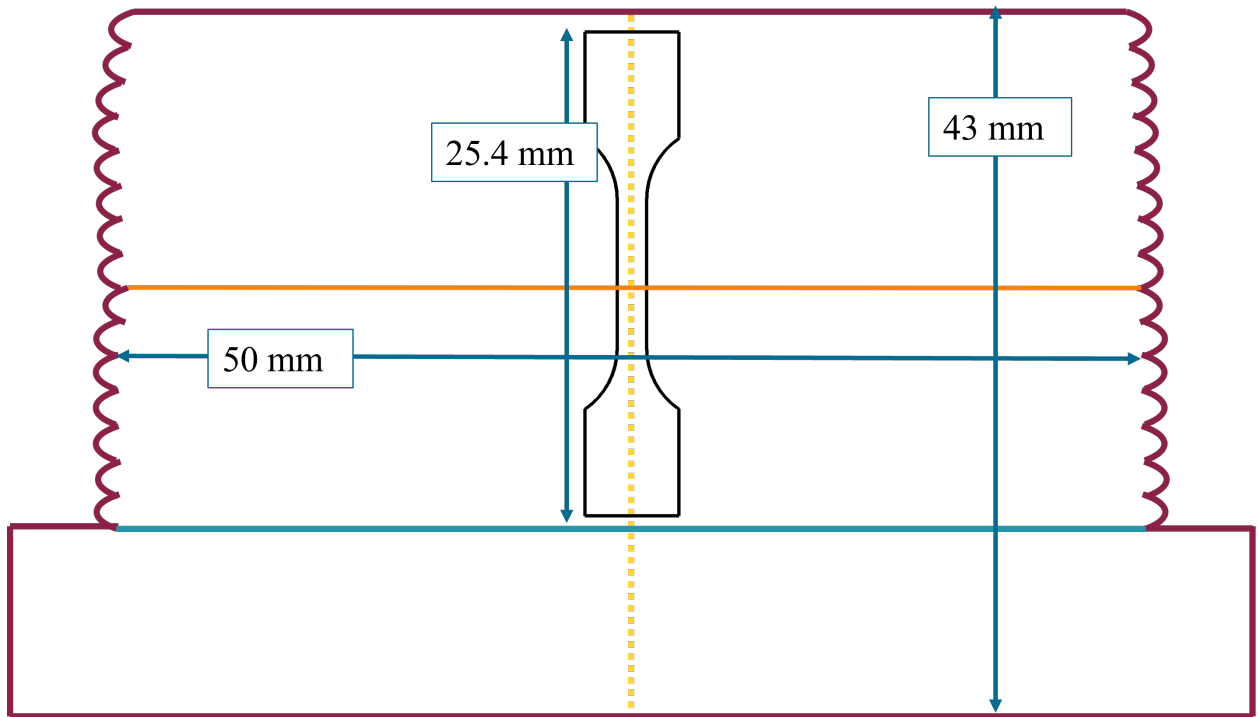


Figure 3.5: Schematic of microhardness indentation locations (yellow) and tensile sample extraction location and orientation (gray) through the height of the AFSD builds with the 9.525 mm deposition parameters. The orange line denotes the location of the manufacturing pause and the blue line denotes the base plate/deposit interface.

3.2.2 Tensile Testing

Tensile testing was performed on each of the 9.525 mm sample conditions (Table 3.2). Tensile bars were extracted using a Mitsubishi MV2400 wire EDM with the aluminum machining setting as recommended by the user manual. A sub-size tensile bar (Fig. 3.6) was extracted from the cross-section of the deposited material such that the location of the manufacturing pause was within the gage volume of the tensile sample (Fig. 3.5). Testing was conducted on a servo-hydraulic MTS 858 Table Top System with a calibrated 25 kN load cell using displacement control to approximate a strain rate as recommended by ASTM E8 7.6.3.2 (66). Samples were placed into a grip fixture with hydraulic wedge grips. Each test condition was tested five times for repeatability, with all tensile samples extracted from the central steady-state deposition region.

Digital Image Correlation

Additional 3D digital image correlation (3D-DIC) data were collected on select samples from the no pause and 100hr pause subsets to further probe the stress-strain behavior during tensile loading. This was enabled by an airbrushed black speckle pattern applied over a white base coat on the specimens and a two camera setup. Images of the sample under loading were acquired at a frequency of 5 Hz and processed using *VIC-3D v9* (Correlated Solutions USA). The DIC software was calibrated using a calibration target prior to testing.

Fractography

Following tensile testing, fracture surfaces were imaged and the depth range of the fracture surface was measured using a Keyence VHX-5000 digital microscope. The cross-sectional

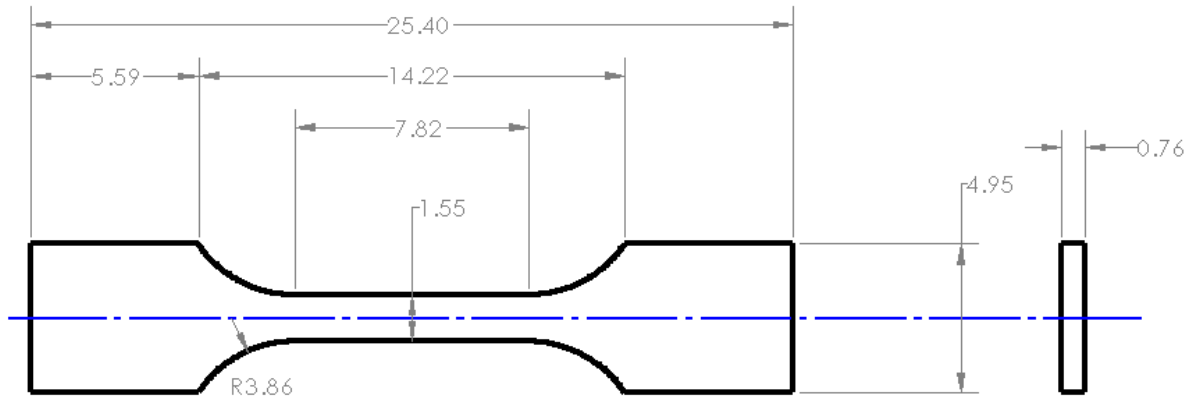


Figure 3.6: Subscale tensile bar geometry. Dimensions are in mm.

area of the tensile specimen after fracture was measured from the images using *ImageJ* software (67).

3.2.3 Base plate Deflection

Prior to any sample extraction, select samples were scanned with a GOM ATOS Q (ZEISS Industrial Quality Solutions) structured light scanner. ZIESS Inspect software was used to analyze deviation from flat in the base plate following deposition. Traces through the center of the deposit in measuring the deflection of the base plate in the longitudinal and transverse direction were measured and the deflection magnitude is compared.

3.3 Metallography

Following deposition, samples were prepared for metallographic analysis and hardness testing. First, a 12.7 mm thick section was extracted normal to the AFSD tool travel direction using wire EDM on a Mitsubishi MV2400 EDM (Fig. 3.7). Samples were then ground using SiC grinding paper to a 1200 grit final grinding finish and were then polished to a 1 μm diamond suspension final polish. After the final polishing step, samples were prepared for bulk optical imaging using a NaOH solution and Weck's reagent. Unetched samples were instead prepared for electron backscatter diffraction (EBSD) by vibratory polishing to a 0.05 μm colloidal silica suspension for a minimum of 1 hour.

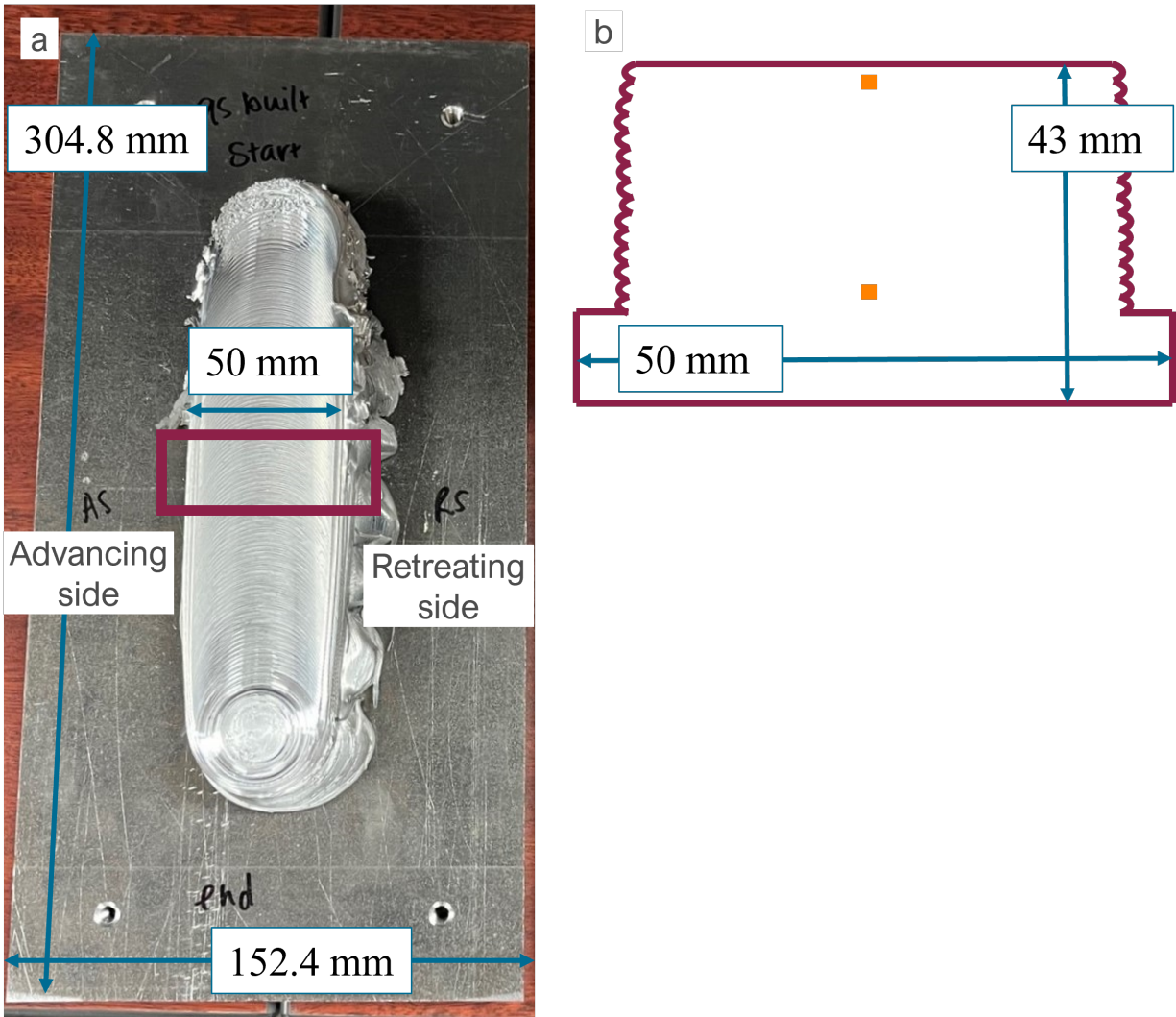


Figure 3.7: Image showing the extracted region of interest for metallographic analysis (a) and highlighted locations for EBSD analysis (b).

3.3.1 Electron Backscatter Diffraction

EBSD was performed using a Helios 5 Hydra (Thermo Fisher Scientific) equipped with a Velocity Pro EBSD camera (EDAX) and a field-emission electron gun operated at 25 kV accelerating voltage and 6.4 nA beam current. Samples were angled to 70 deg. EBSD data were collected in the central region of the deposit spanning 40 μm x 51 μm in the lower and upper regions of the deposit (Fig. 3.7). Patterns were collected at 0.1 μm x 0.1 μm spatial resolution. EBSD patterns were indexed using the EMSphInx software package to perform dictionary indexing (68; 69). Grain characteristics were analyzed using the *MTEX* algorithm available within MATLAB (70; 71). Grain size statistics, grain aspect ratio, grain boundary misorientation distribution were calculate and plotted, and inverse pole figure (IPF) plots, kernel average misorientation (KAM) plots, and grain reference orientation deviation (GROD) plots were generated.

3.3.2 X-Ray Diffraction

X-ray diffraction (XRD) was conducted on the polished surfaces of the no pause and 100 hr pause, no reheat samples. A beam footprint of 12 mm across the width of the build by 4 mm in the height of the build was chosen, centered within the deposited material. Data were taken through the build height at $Z = 12, 16, 20, 24, 28, 32, 36,$ and 40 mm, where $Z = 12.7$ is the approximate baseplate and deposit interface height. Data fitting was performed using a Rietveld refinement method in *Topas* software.

3.4 Residual Stress Analysis

3.4.1 Neutron Diffraction

Non-destructive residual stress analysis was conducted on the no pause and 100 hr pause samples via neutron diffraction. Neutron diffraction was performed at the Oak Ridge National Laboratory (ORNL) High Flux Isotope Reactor (HFIR) High Intensity Diffractometer for Residual Stress Analysis (HIDRA) beamline HB-2B using a standard instrument configuration (72). In studying the no pause sample, a 331 monochromator was chosen for an incident neutron beam wavelength of $\lambda = 1.721 \text{ \AA}$. Subsequent studies on the 100 hr pause samples and d_0 comb measurements were performed using a 422 monochromator, resulting in an incident neutron beam wavelength of $\lambda = 1.523 \text{ \AA}$. A 2D detector was positioned approximately 90 deg 2θ to the neutron beam for the 331 monochromated beam and 78 deg 2θ for the 422 monochromated beam, to observe both the 311 and 222 aluminum diffraction peaks. Beam slits were used to define a gauge volume of 3 mm x 3 mm in the transverse/normal direction of the sample (figure). Within the longitudinal direction of the deposit where AFSD was assumed to be steady-state, and the beam was opened to a 6+ mm gauge volume to allow for faster data acquisition (Fig. 3.8). Mapping was accomplished using a motorized stage and the CUBOID planning software at HIDRA.

Unstressed Comb Preparation and Measurement

Measurement of the unstressed d_0 spacing was conducted on a 3 mm thick slice extracted from builds created with replicated processing parameters and conditions to the stressed

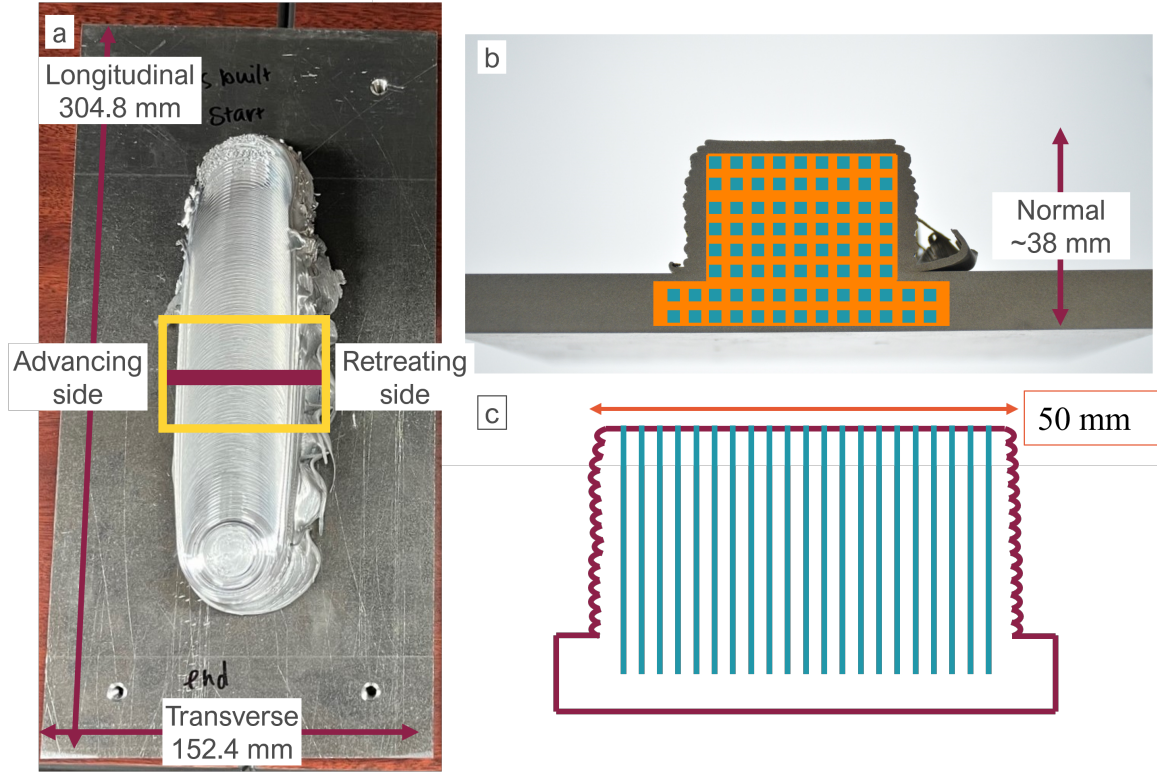


Figure 3.8: (a) Image of the as built deposit with the purple region highlighting the location of the unstressed d_0 spacing comb and the yellow region outlining the larger steady-state deposition region studied, (b) the cross-section of the build with the orange and blue regions indicating the region that 3 mm x 3 mm voxels were characterized via neutron diffraction, and (c) unstressed comb for d_0 spacing measurement, with blue lines indicating locations of wire EDM cuts to create teeth. Transverse, normal, and longitudinal directions of the build are labeled on images (a) and (b).

samples. Slices were extracted from the central portion of the deposit using wire EDM. Slits were then cut into the sliced samples to further relieve stresses within the samples (Fig. 3.8). Unstressed d_0 spacing was measured using the standard instrument configuration at HIDRA in the longitudinal direction of the build in 3 x 3 mm voxels through the thickness of the comb, as stresses were assumed to be relieved and therefore additional measurements in the transverse and normal directions of the build were not necessary.

Data Treatment and Analysis

Following neutron diffraction, peak fitting for d-spacing extraction was performed using the software *Topas v7* (73). From the 2θ values d_{hkl} values were calculated using Bragg's Law (Eq. 2.1). Partially buried volumes and volumes where the signal-to-noise ratio exceeded 2 were filtered from the data. Following filtration, a Gaussian process regression was performed to impute and estimate d-spacing at each point in the cross-sectional map. After data treatment, residual stress was calculated using the previously discussed calculations for lattice strain (Eq. 2.2) and Hooke's Law (Eq. 2.3).

3.4.2 Contour Method

Sample Preparation

The contour method was used to evaluate the residual stress in each test condition listed in Table 3.2. To prepare samples for contour measurement, samples were cut normal to the face upon which the contour analysis was performed on a Mitsubishi MV2400 EDM. Cutting parameters were chosen based on the thickest region of the sample according to the EDM

manual. Samples were fixtured to the corner of the EDM to restrict movement of the sample as stress is relieved during cutting, therefore minimizing bulge error on the cut surface (Fig. 3.9).

Data Collection

Following cutting of a sample, both halves of the sample were measured using a 0.5 mm spaced grid measurement method on a ZEISS DuraMax coordinate measuring machine (CMM) with 2.4 μm measurement error specification (Fig. 3.10). Data were collected using a raster pattern across the build and around the edge of the sample with a 0.5 mm offset distance from the edge of the sample. Samples were aligned on the CMM such that $X = 0$ on each measurement was on the same side, so when the data were imported for analysis they could be mirrored across the X axis and averaged to a grid.

Data Treatment

Measured data from the CMM were exported as .txt files and plotted using Python. Data were then interpolated to a 1.27 mm grid using `scipy.interpolate SmoothBivariateSpline` fitting in Python. The data were filtered to eliminate outlier values. Smoothing parameters were iterated upon to retain the minimum and maximum deflection while smoothing noise within the measurement. A smoothing parameter of $s=0.5$ was chosen to meet this criteria. Where the interpolation could not compute on the target points, extrapolation was performed to the median value within 2 mm of the target point. Data were plotted and exported as .csv files.

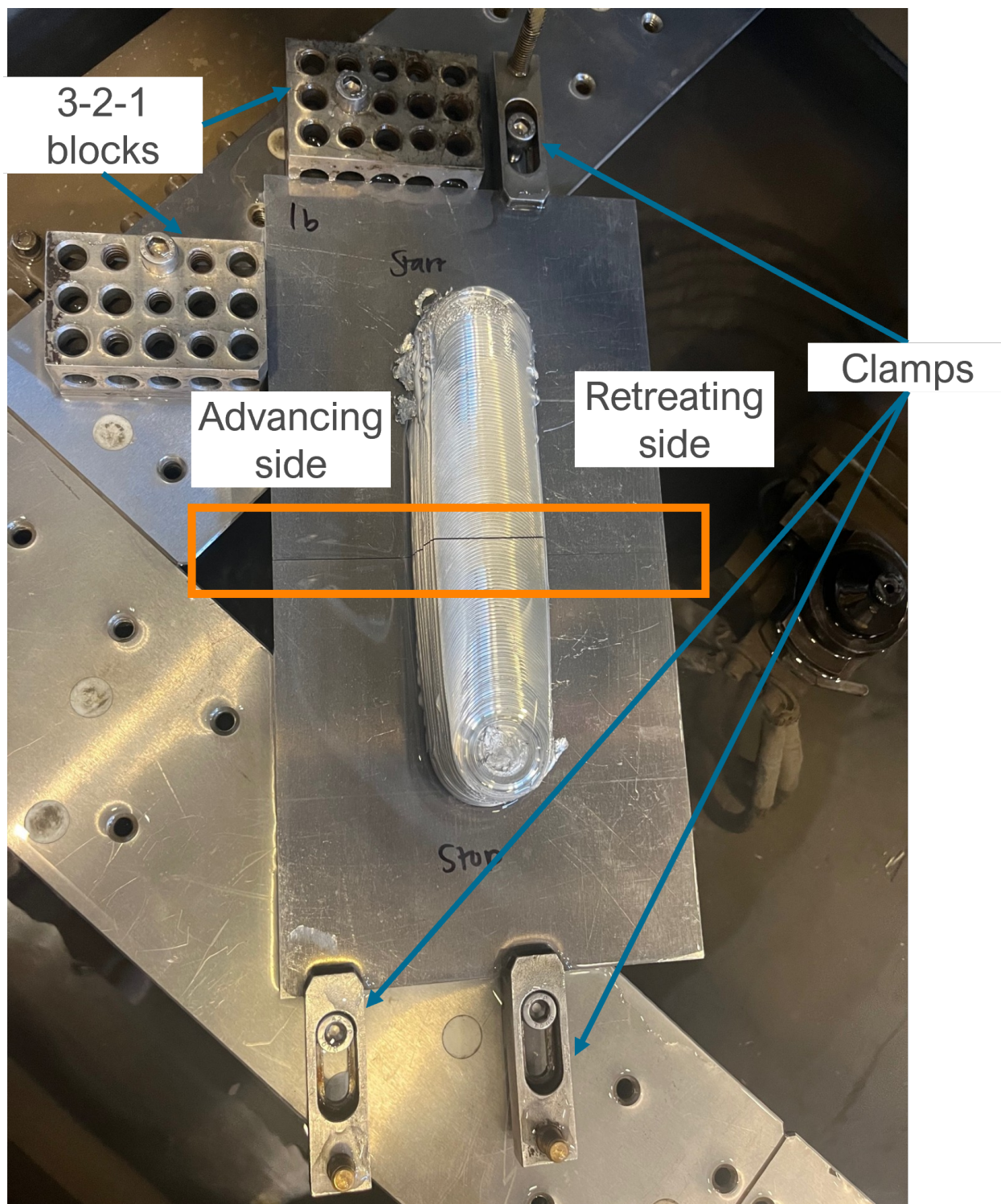


Figure 3.9: (a) Image of fixturing setup for cutting across the center of the deposit with the cut line outlined in orange.



Figure 3.10: (a) Image of the ZEISS DuraMax CMM measuring a cut face of a sample for contour analysis.

Data Analysis

Finite element analysis (FEA) of the contour data was performed to calculate the stress relieved normal to the cut and measured face. To achieve this, a mesh of the same geometry as the cut half of the sample was generated with 1.27 mm nodal spacing to match the spacing of the interpolated contour data. The open source Python package for residual stress analysis, pyCM, was implemented to generate the mesh and interpret the data (74). Additionally analysis was conducted using Abaqus commercial FEA software. Then, a boundary condition was imposed on the surface to force the model back to flat, and the stress state caused by this force was calculated. Poisson's ratio was 0.33 and the elastic modulus was 71700 MPa based upon the Aerospace Specification Metals standard for average AA7075 values (75).

Chapter 4

Experimental Results

4.1 Mechanical Testing

4.1.1 Microhardness

Age Hardening

The resultant microhardness within the deposited material and base plate is plotted in Fig. [4.1](#) with data collected after 10 hours, 1 week, 4 weeks, and 15 weeks after the deposit was completed. The bottom of the base plate is set at 0 mm, the interface of the base plate and deposited material is approximately 12 mm, and the topmost layer is 41.6 mm. The hardness profile taken approximately 10 hours after deposition shows a gradient of microhardness that is softest at the base plate and deposit interface and hardest in the top layer of the deposit. The minimum hardness was 70 HV and the maximum hardness was 113 HV, significantly lower than the base plate stock material measured hardness of 173 HV. Approximately 15 weeks after deposition occurred, the shape of the hardness profile

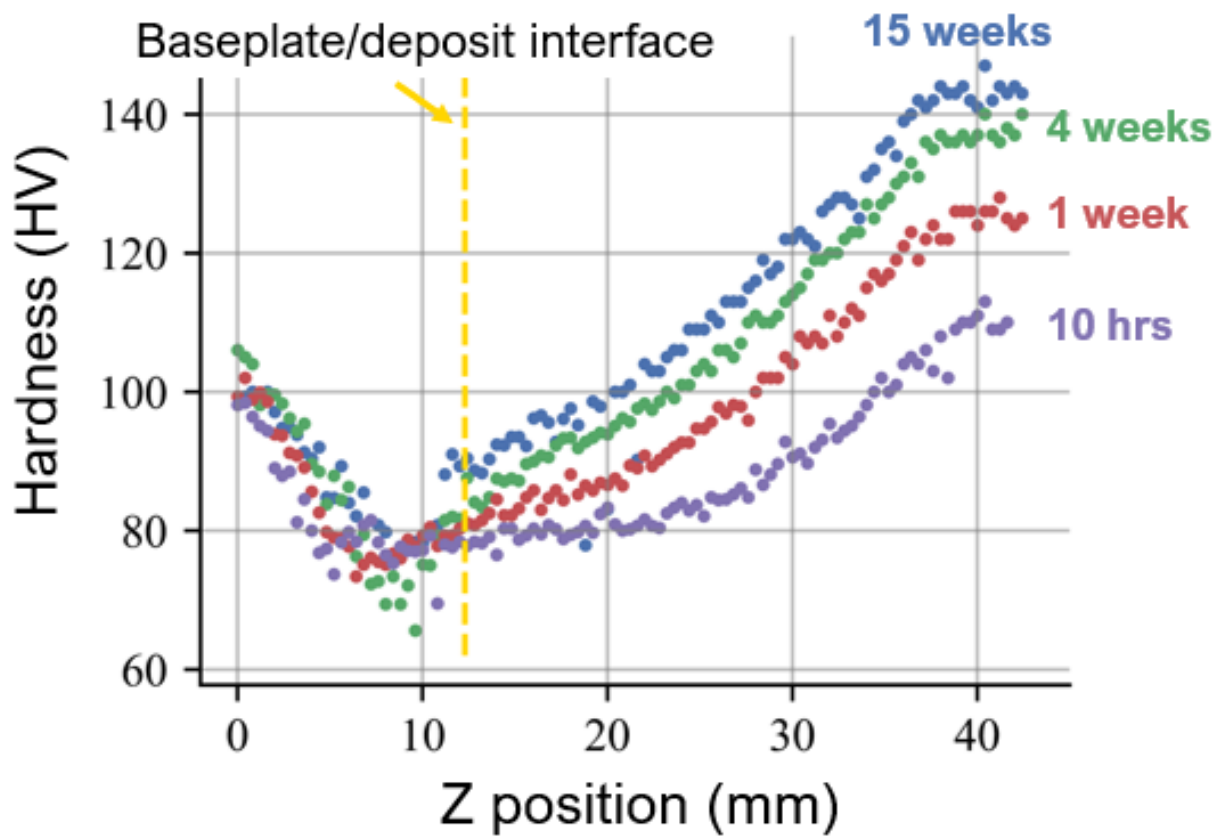


Figure 4.1: Vickers microhardness vs. build height (Z direction). The base plate/deposit interface is located near the 12 mm location on the horizontal axis.

remained the same, but the material hardened to a minimum hardness of 77 HV and the maximum hardness of 147 HV. The resultant hardness at each timestep shows the base plate hardness reduces through the height as it approaches the deposit interface. The hardness within the deposited material is consistently lowest at the base plate/deposit interface and steadily increases through the build height. The hardness profile of deposited material shows natural age hardening occurred in all deposited material and that the hardness of the base plate material changed minimally for the duration of the experiment. The material hardened rapidly in the first week following deposition, but the hardening rate slowed considerably over longer aging times, as expected. Specifically, the maximum hardness increased from 113 to 128 HV (13.3%) over the first week of aging, but only from 140 to 147 HV (5%) between the 4th and 15th weeks of aging. The peak hardness of 147 HV after 15 weeks of aging is lower than the AA7075-T6 stock material hardness of 179 HV (76). However, the deposited material may continue to age, as studies have shown that natural aging in AA7075 increases tensile strength over many years (43).

Further understanding of the age hardening observed was enabled by studying the temperature in AFSD AA7075 with a 125 RPM constant spindle speed conducted at the University of Tennessee, as reported by Schmitz et al. (77) and using the experimental setup in Fig. 4.2. The temperature within the base plate immediately below the deposition during AFSD as measured by the four thermocouples shown in Fig. 4.2 is plotted in Fig. 4.3.

Hardness Map in Cross-Section of No Pause Build

The Vickers microhardness in the cross-section of the no pause sample is plotted in Fig. 4.4. As deposition occurred, thermal input from active deposition increases the time at an

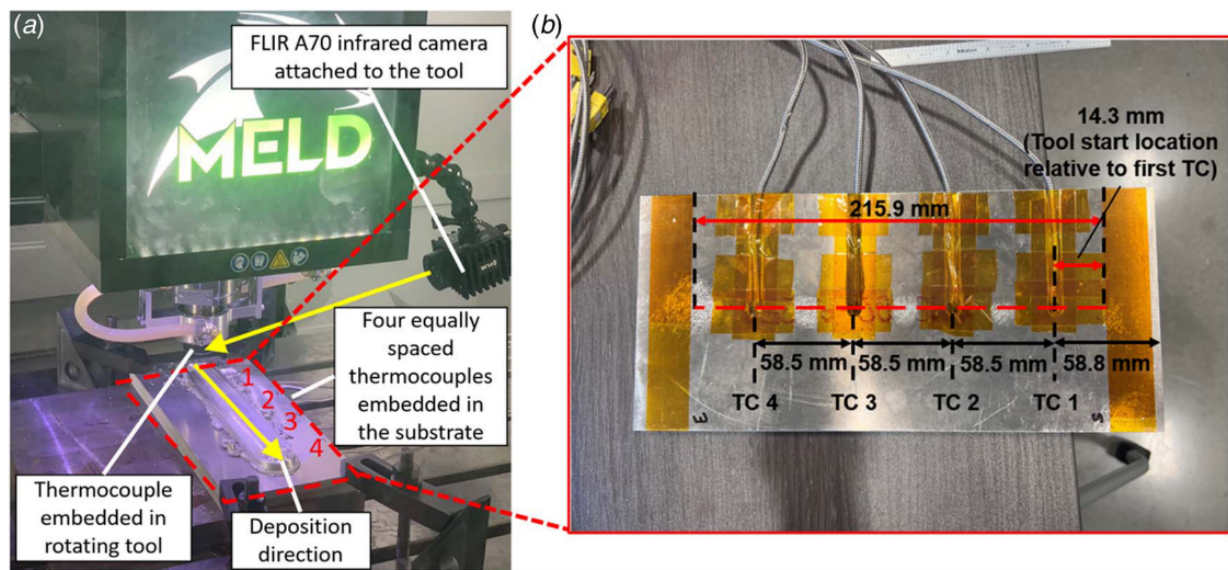


Figure 4.2: Location of thermocouples embedded into the build plate below a deposit conducted with comparable processing conditions to the 12.7 mm sample generated in this study (1).

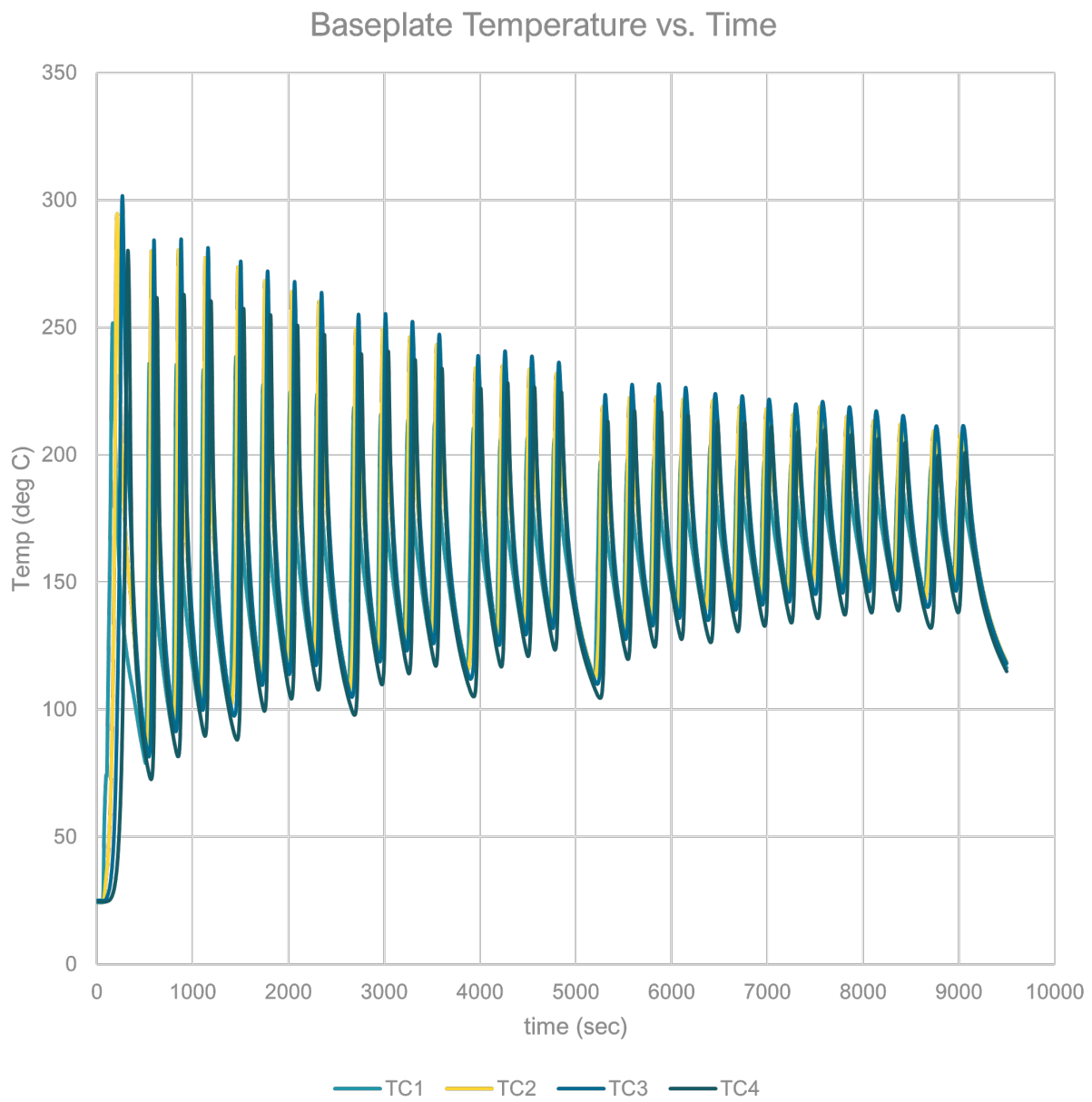


Figure 4.3: Temperature of the base plate region below the deposit in a AFSD AA7075 study captured by K-type thermocouples embedded within the base plate.

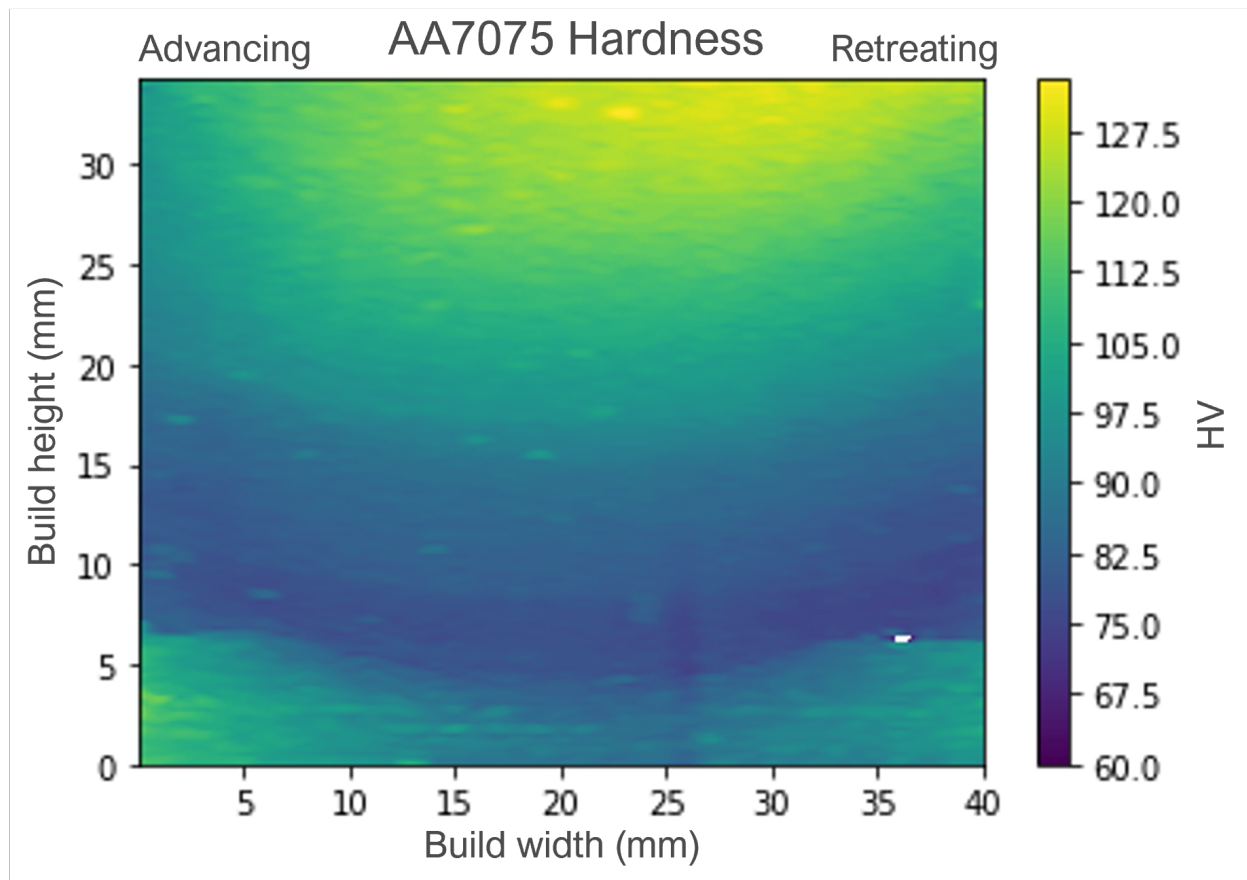


Figure 4.4: Hardness map in the transverse/normal cross-section of the no pause 9.525 mm build.

elevated temperature. Therefore, the top layers experience the least time at an elevated temperature and retain nearly 75% of T6 hardness (~ 177 HV), while the lowest regions are near O-temper hardness of 68 HV. The hardest regions skew toward the retreating side of the deposition with a peak value of 132 HV (2).

Hardness in Paused Samples

Each sample in the test matrix described in Table 3.2 was tested for Vickers microhardness through the build height. The results show that hardness in each sample is at the lowest near the base plate/deposit interface at an approximate build height of ~ 12 mm (Fig 4.5). However, there are differences between the sample produced with no pause and the samples produced with pauses introduced between the 9th and 10th layers. In the no pause sample, the hardness rises slowly through the first 9 layers. In the samples produced with pauses, the hardness rises sharply near $h=12$ mm where the first layer was deposited, and levels off around 105 HV. The 100 hr, no reheat sample hardness slightly exceeded the hardness of the other three samples in the layers deposited before the restart. The final 9 layers all have comparable hardness profiles through the build height, with lowest hardness values in the 10th layer and highest hardness values somewhere within the top three layers.

4.1.2 Tensile Testing

Tensile Results

Tensile testing was conducted on five samples for each of the sample sets, except for the 100 hr, no pause set, which had two of the five samples fail during loading in the tensile testing

Vickers Microhardness vs. Height - All Samples

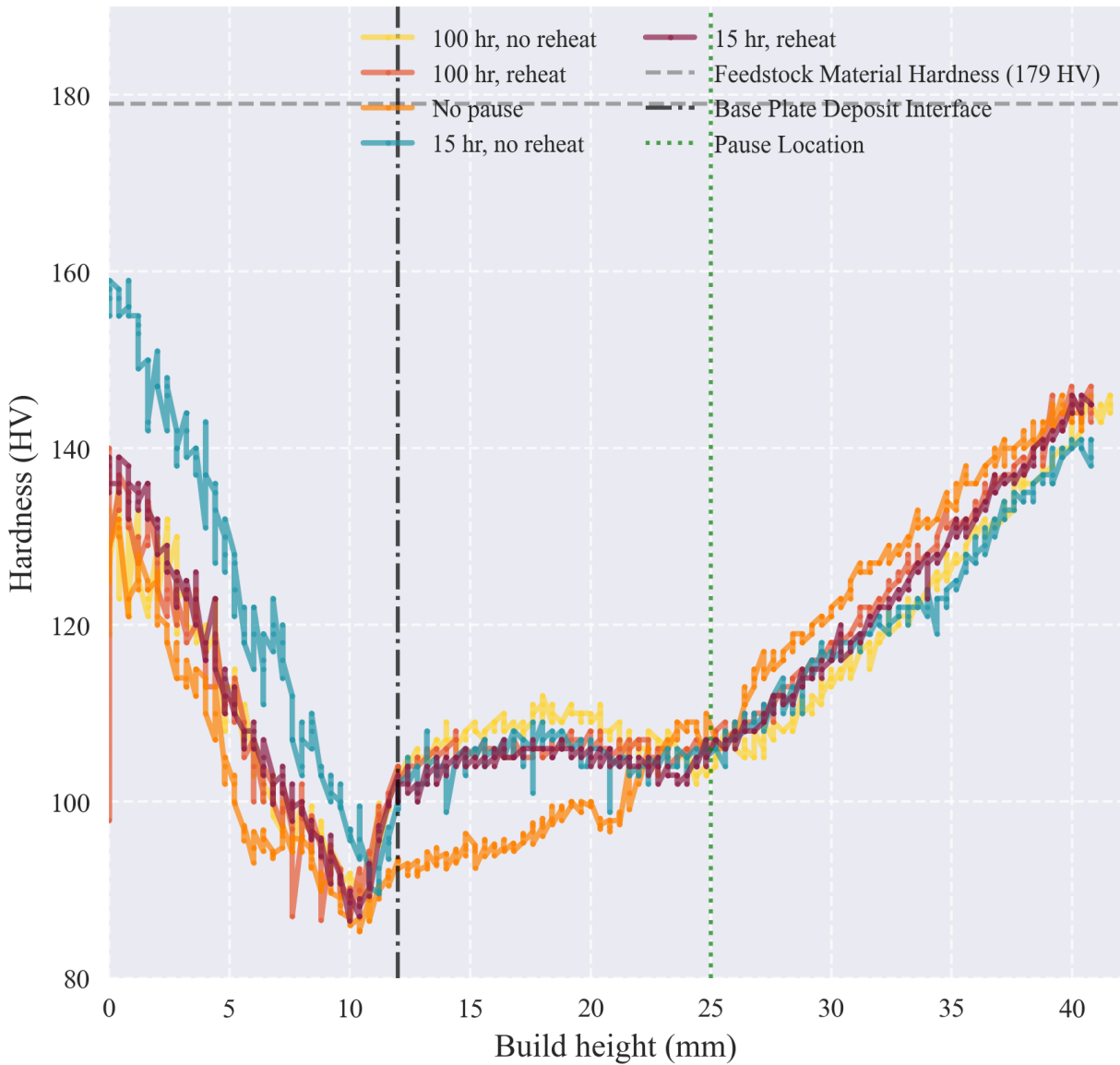


Figure 4.5: Hardness vs. build height in each of the test conditions. From left to right, the hardness is first measured at the bottom of the base plate, through the base plate to the base plate/deposit interface at approximately 11.2 mm (denoted by the dashed gray line), and through the build height. The pause location is near 25.5 mm, denoted by a dashed gray line.

configuration. The ultimate tensile strength values range from 259 - 340 MPa, yield strength values range from 224 - 264 MPa, and elastic modulus ranges from 72,395 - 76,187 MPa. The results of tensile testing are shown in Fig. 4.6 with additional details in Table 4.1. The location of failure relative to the layer number is shown in Table 4.2.

Digital Image Correlation

DIC analysis was conducted on two no pause samples, two 100 hr pause, reheat samples, and one 100 hr pause, no reheat sample. A second 100 hr pause, no reheat sample was attempted, but failed during loading. From the series of DIC images captured, the Von Mises strain immediately before failure is colorized on the samples (Fig. 4.7).

Fractography

Fractography was conducted on two samples from each of the pause conditions in the sample test matrix and images of the fracture surfaces and topographical measurements were plotted (Figs. 4.8 - 4.12). The cross-sectional area of the fracture surfaces was measured and the overall reduction in area is reported alongside the % ductility in Table 4.3. The reduction in area within the tensile samples ranged from 4.1% to 14.9%, and the elongation at failure ranged from 3.8% to 9.6%. The overall topographical characteristics of the failure surfaces are slanted, indicating that typical ductile failure and shear failure were both present.

4.1.3 Base Plate Deflection

Base plate deflection results were measured using structured light scanning on the bottom of the base plate through the centerline in the transverse (X) and longitudinal (Y) directions

Tensile Properties of AFSD AA7075 with Pauses

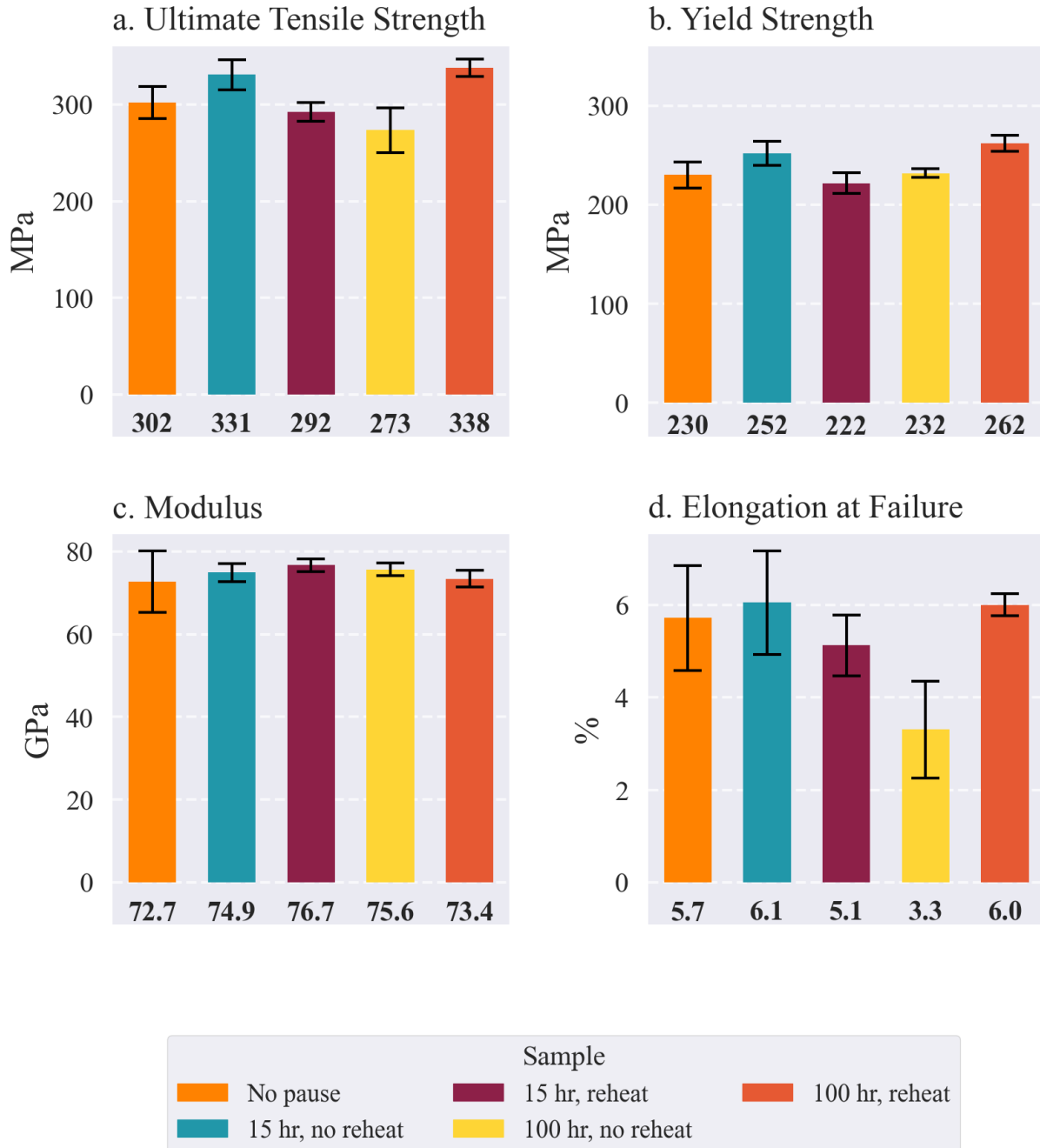


Figure 4.6: Ultimate tensile strength (UTS), yield strength, and elastic moduli of the paused and no pause samples.

Table 4.1: Summary of Tensile Properties**No pause**

Property	Avg	SD	SE	Min	Max
Elastic Modulus (GPa)	72.7	16.6	7.4	58.8	92.1
Ultimate Tensile Strength (MPa)	302.0	36.7	16.4	263.7	344.7
Yield Strength (MPa)	230.3	29.9	13.4	186.2	269.0
Elongation at Failure (%)	5.7	2.0	0.9	2.4	9.1

15 hr, no reheat

Property	Avg	SD	SE	Min	Max
Elastic Modulus (GPa)	74.9	4.9	2.2	70.7	80.3
Ultimate Tensile Strength (MPa)	330.9	34.4	15.4	292.0	365.9
Yield Strength (MPa)	252.3	27.0	12.1	220.6	282.7
Elongation at Failure (%)	6.1	2.5	1.1	3.6	9.6

15 hr, reheat

Property	Avg	SD	SE	Min	Max
Elastic Modulus (GPa)	76.7	3.4	1.5	73.1	80.9
Ultimate Tensile Strength (MPa)	292.3	21.0	9.4	269.6	320.8
Yield Strength (MPa)	222.0	23.5	10.5	199.9	262.0
Elongation at Failure (%)	5.1	1.3	0.6	4.0	7.3

100 hr, no reheat

Property	Avg	SD	SE	Min	Max
Elastic Modulus (GPa)	75.6	2.3	1.5	72.5	77.7
Ultimate Tensile Strength (MPa)	273.5	35.0	23.3	227.5	306.8
Yield Strength (MPa)	232.1	7.0	4.6	227.5	241.3
Elongation at Failure (%)	3.3	1.5	1.0	1.2	4.6

100 hr, reheat

Property	Avg	SD	SE	Min	Max
Elastic Modulus (GPa)	73.4	4.2	2.1	69.8	78.9
Ultimate Tensile Strength (MPa)	337.8	18.0	9.0	312.7	358.5
Yield Strength (MPa)	262.0	16.4	8.2	234.4	282.7
Elongation at Failure (%)	6.0	0.4	0.2	5.4	6.7

Table 4.2: Failure Layer Number by Sample for Each Test Condition

	No pause	15 hr, no reheat	15 hr, reheat	100 hr, no reheat	100 hr, reheat
1	6.49	6.85	7.26	8.43	10.31
2	6.25	7.06	6.98	8.05	10.06
3	6.48	7.11	6.79	8.05	10.15
4	6.32	6.85	7.06	8.23	9.85
5	6.46	7.17	6.84	7.87	6.98
Avg.	6.40	7.01	6.99	8.13	9.47

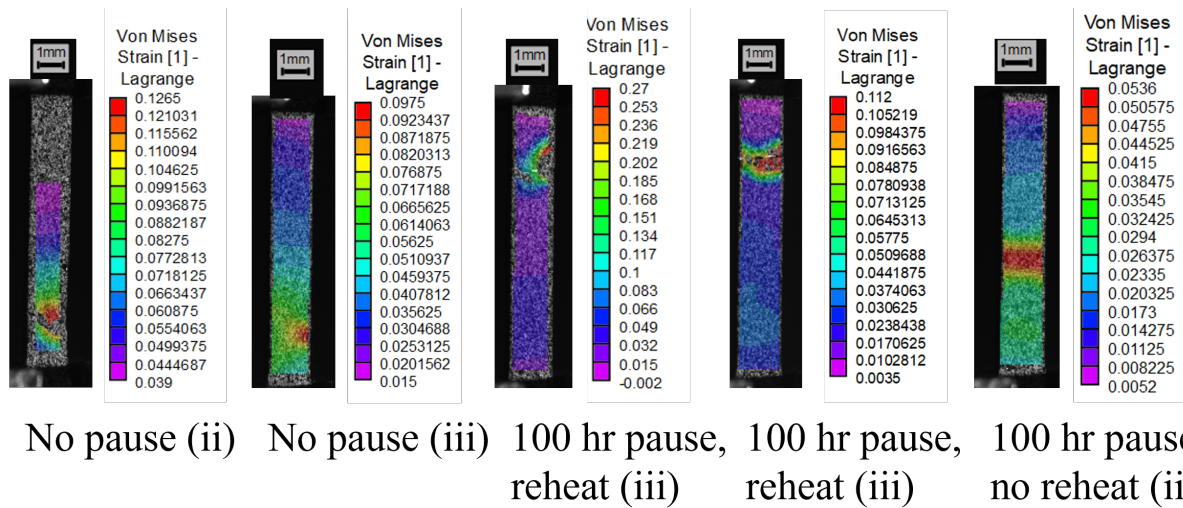


Figure 4.7: Von Mises strain maps of tensile samples analyzed in Correlated Solutions software using DIC.

Table 4.3: Fracture Surface Characteristics and Elongation at Failure by Sample and Test Condition

Sample	Height Diff. (μm)	Change in Area	Elongation at Failure
No pause 2	766	12.4%	9.1%
No pause 5	511	6.7%	5.1%
15 hr pause, no reheat 2	522	7.7%	3.8%
15 hr pause, no reheat 5	699	12.7%	9.6%
15 hr pause, reheat 2	398	10.1%	7.3%
15 hr pause, reheat 5	275	9.6%	4.1%
100 hr pause, no reheat 2	578	4.1%	n/a
100 hr pause, no reheat 5	760	14.9%	4.6%
100 hr pause, reheat 2	380	6.2%	5.4%
100 hr pause, reheat 5	608	12.1%	6.7%

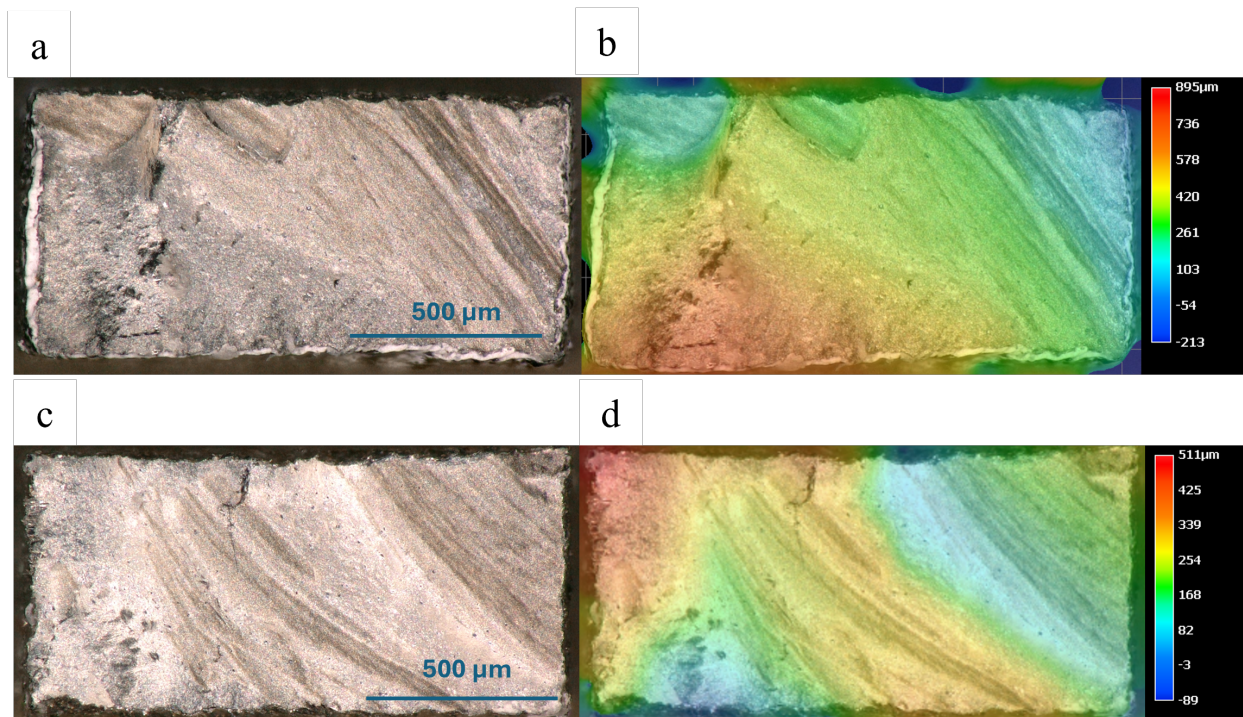


Figure 4.8: Images of fracture surfaces from the no pause sample. (a) and (b) are from the second tensile sample, and (c) and (d) are from the fifth tensile sample. Figures (b) and (d) display the measured surface displacement, with 0 being the lowest value on the fracture surface.

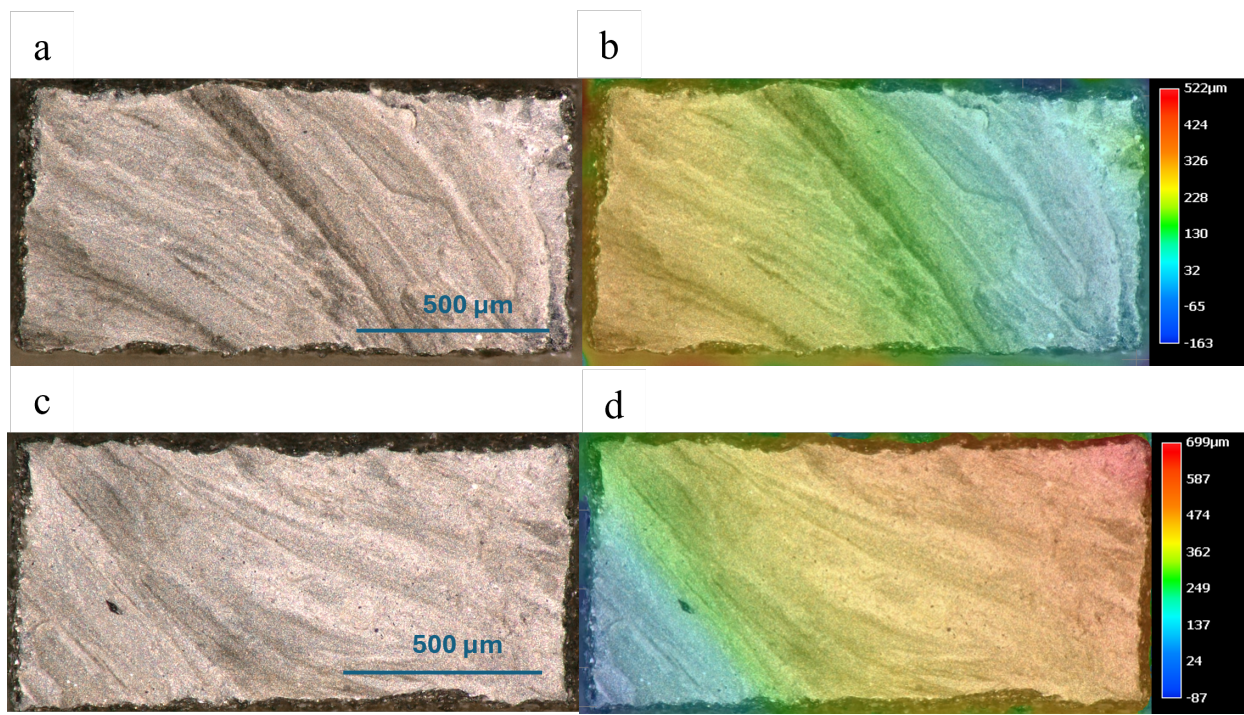


Figure 4.9: Images of fracture surfaces from the 15 hr, no reheat sample. (a) and (b) are from the second tensile sample, and (c) and (d) are from the fifth tensile sample. Figures (b) and (d) display the measured surface displacement, with 0 being the lowest value on the fracture surface.

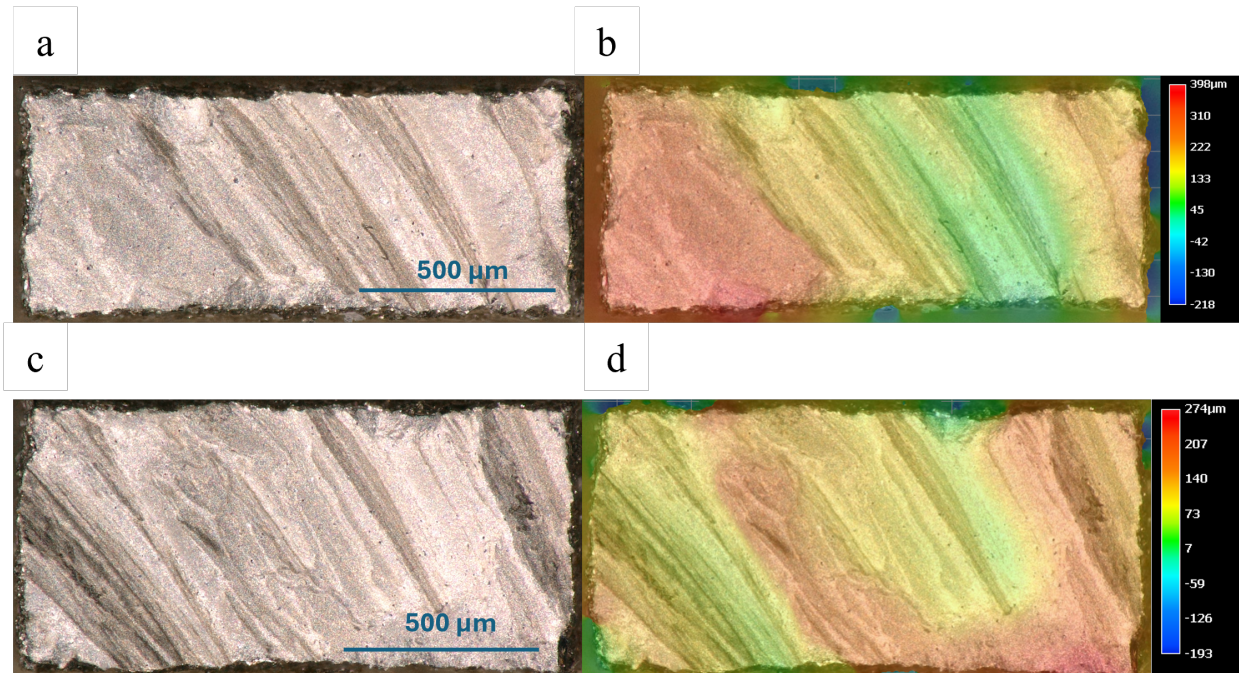


Figure 4.10: Images of fracture surfaces from the 15 hr, reheat sample. (a) and (b) are from the second tensile sample, and (c) and (d) are from the fifth tensile sample. Figures (b) and (d) display the measured surface displacement, with 0 being the lowest value on the fracture surface.

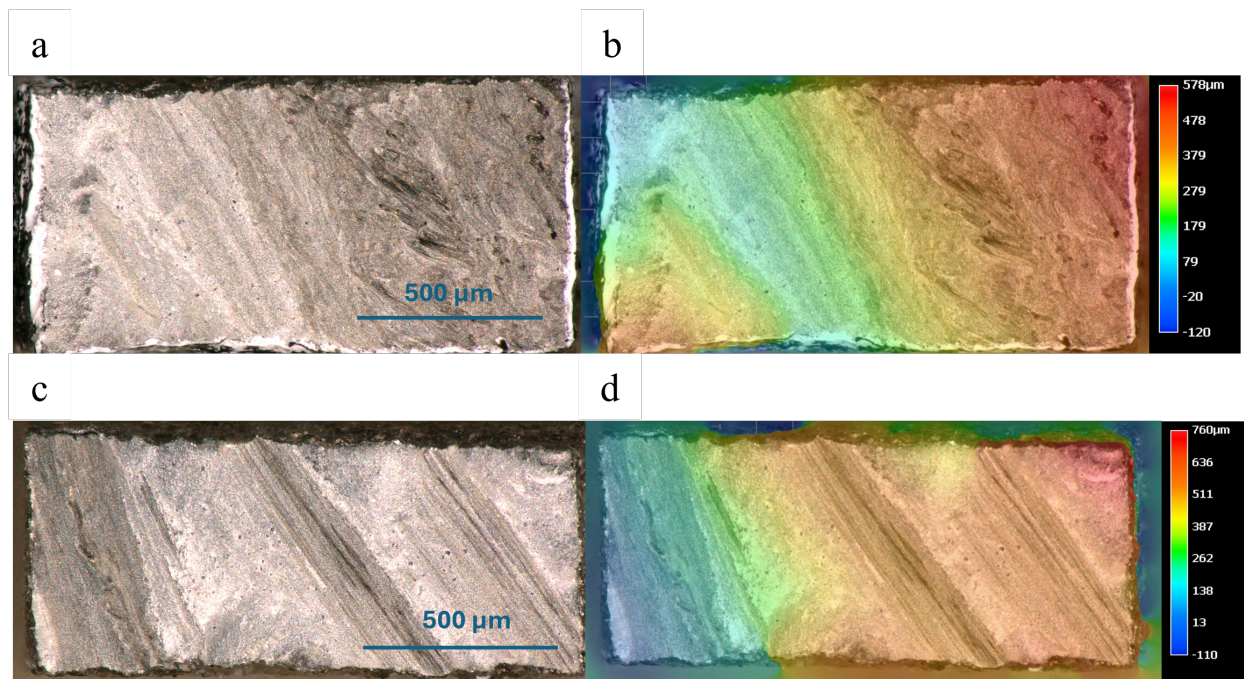


Figure 4.11: Images of fracture surfaces from the 100 hr, no reheat sample. (a) and (b) are from the third tensile sample, and (c) and (d) are from the fifth tensile sample. Figures (b) and (d) display the measured surface displacement, with 0 being the lowest value on the fracture surface.

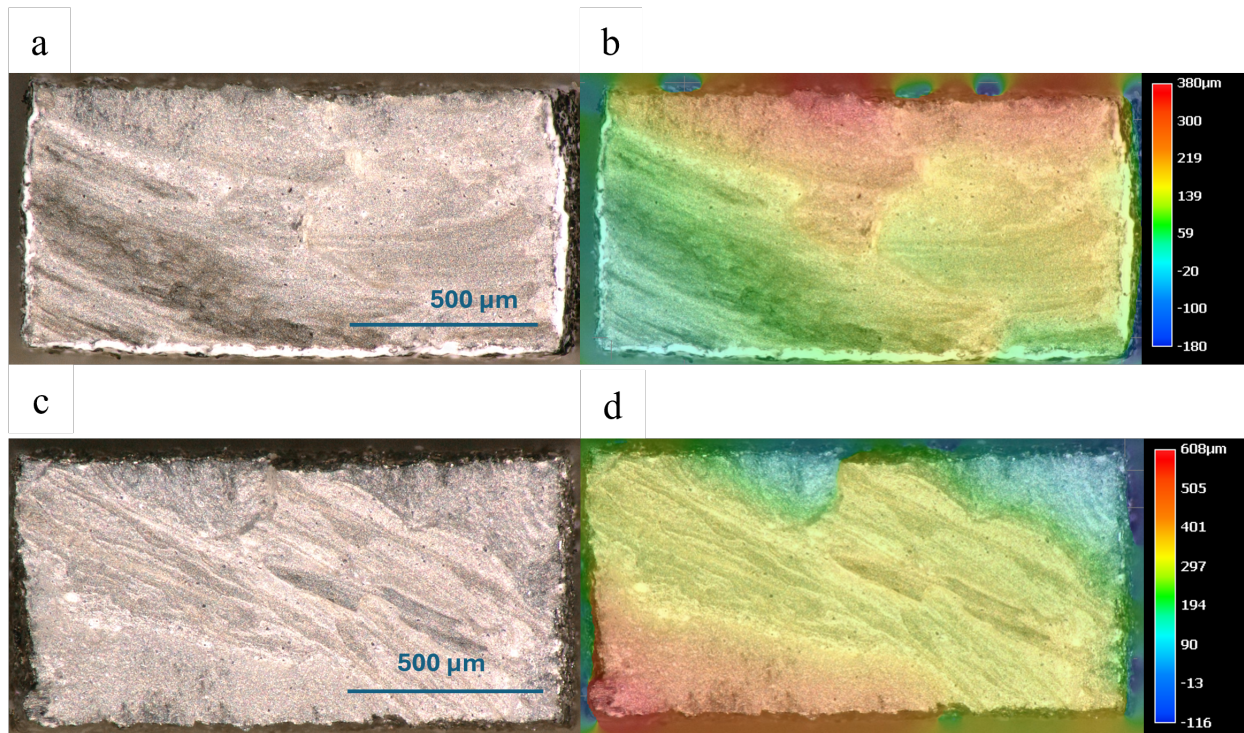


Figure 4.12: Images of fracture surfaces from the 100 hr, reheat sample. (a) and (b) are from the second tensile sample, and (c) and (d) are from the fifth tensile sample. Figures (b) and (d) display the measured surface displacement, with 0 being the lowest value on the fracture surface.

of the components, locations of which are shown in Fig. 4.13. Statistics for the deflection data are shown next to the plots. The range of deflection data across the width of the build (transverse direction) is plotted in Fig. 4.14 and the range and standard deviation of the deflection data for each of the pause conditions are plotted in Fig. 4.15. Similarly, the range of deflection data along the length of the build (transverse direction) is plotted in Fig. 4.16 and the range and standard deviation of the deflection data for each of the pause conditions are plotted in Fig. 4.17. In addition to these plotted lines, color plots to visualize the deflection within the base plate may be found in Appendix ??.

4.2 Metallography

To understand the grain characteristics of the deposited material, optical and scanning electron microscopy were conducted. Bulk imaging on the polished and etched cross-section of the AFSD material shows downward force from the AFSD process causes deformation into the base plate/deposit interface and the rolled microstructure of the base plate from the forming process (Fig. 4.18). High resolution optical analysis of the AFSD builds was difficult to achieve due to the variation in microstructure through the build height and small grain sized observed in as-built AFSD material, so scanning electron microscopy EBSD was conducted to characterize the grains (2).

4.2.1 Electron Backscatter Diffraction Results

Where optical imaging was unable to adequately image individual grains for quantification, EBSD was conducted on the no pause sample. Grain size statistics, a schematic indicating

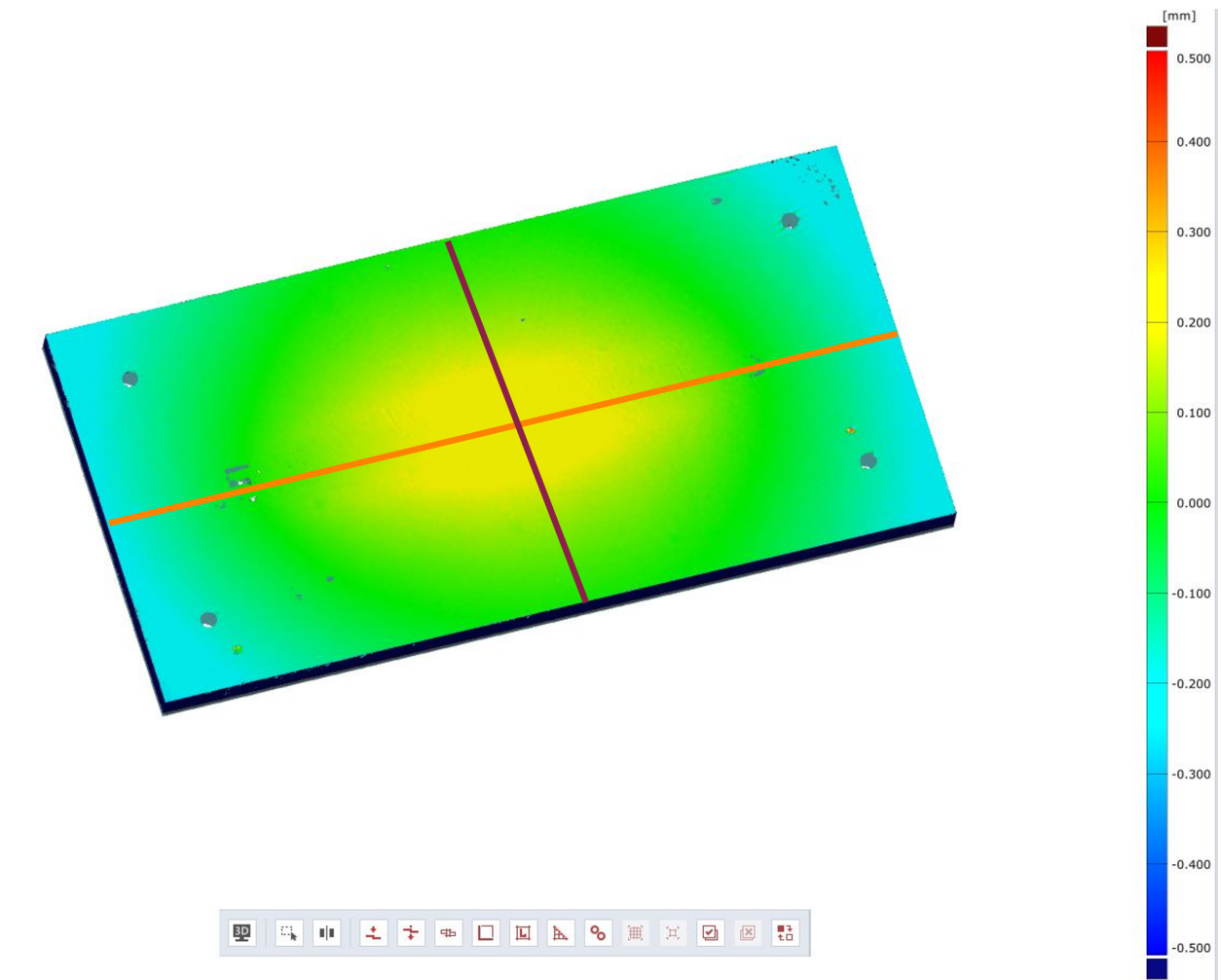


Figure 4.13: Location of deflection measurements on the bottom of the base plate (opposite to the side with the AFSD material) in longitudinal (purple) and transverse (orange).

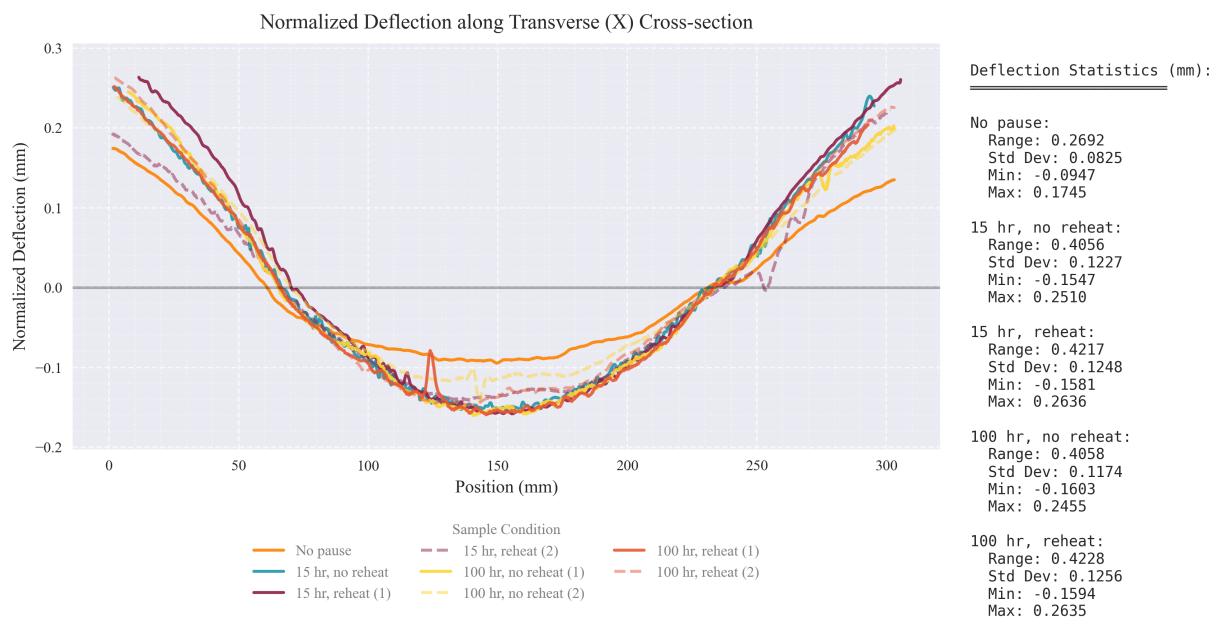


Figure 4.14: Deflection across the transverse direction of the paused sample test matrix deposits.

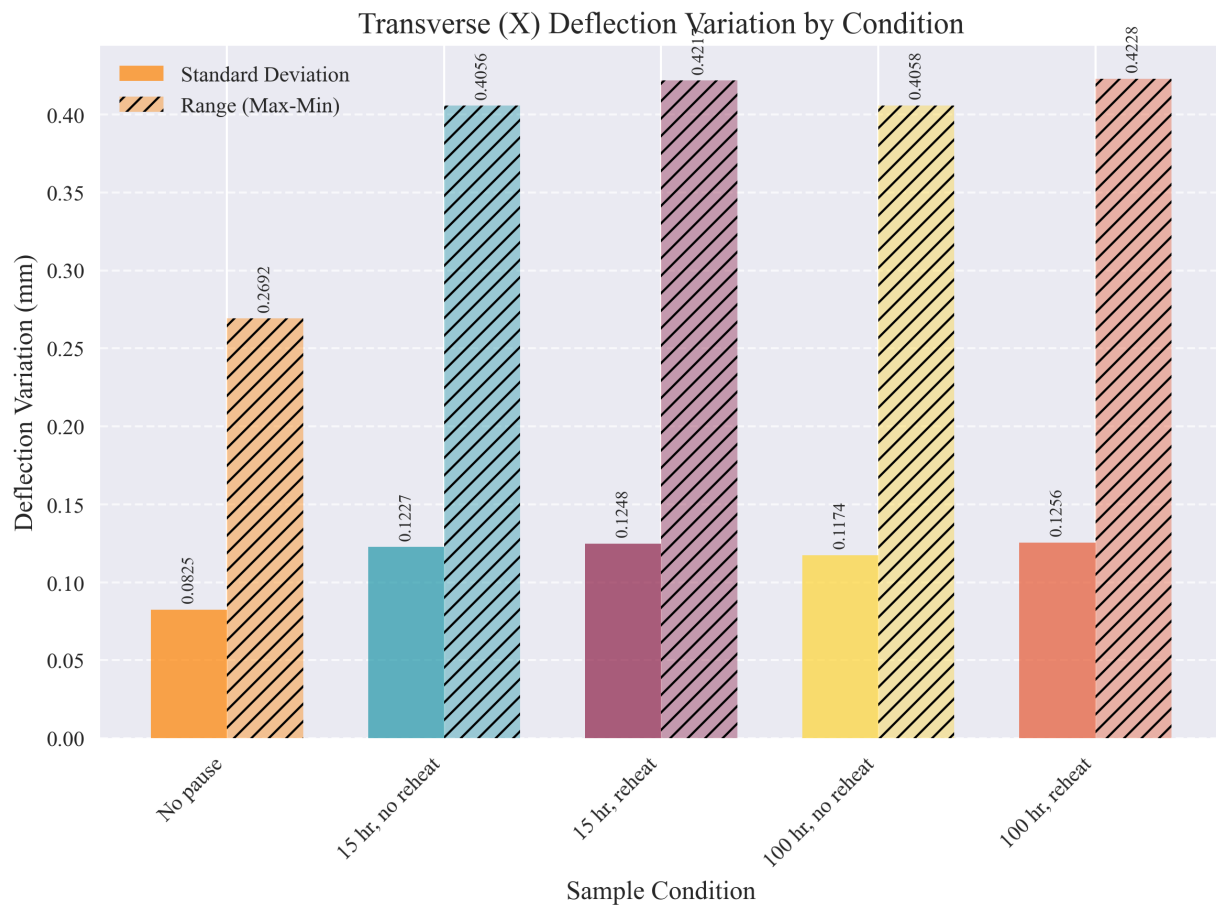


Figure 4.15: Statistical information about deflection across the transverse direction of the paused sample test matrix deposits.

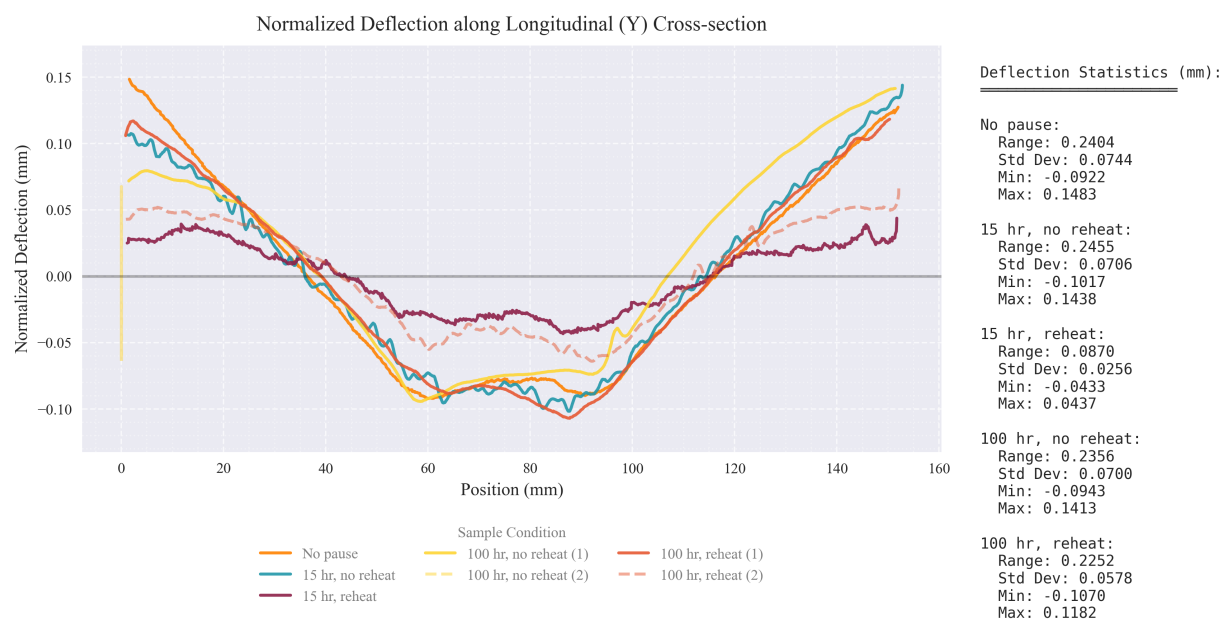


Figure 4.16: Deflection across the longitudinal direction of the paused sample test matrix deposits.

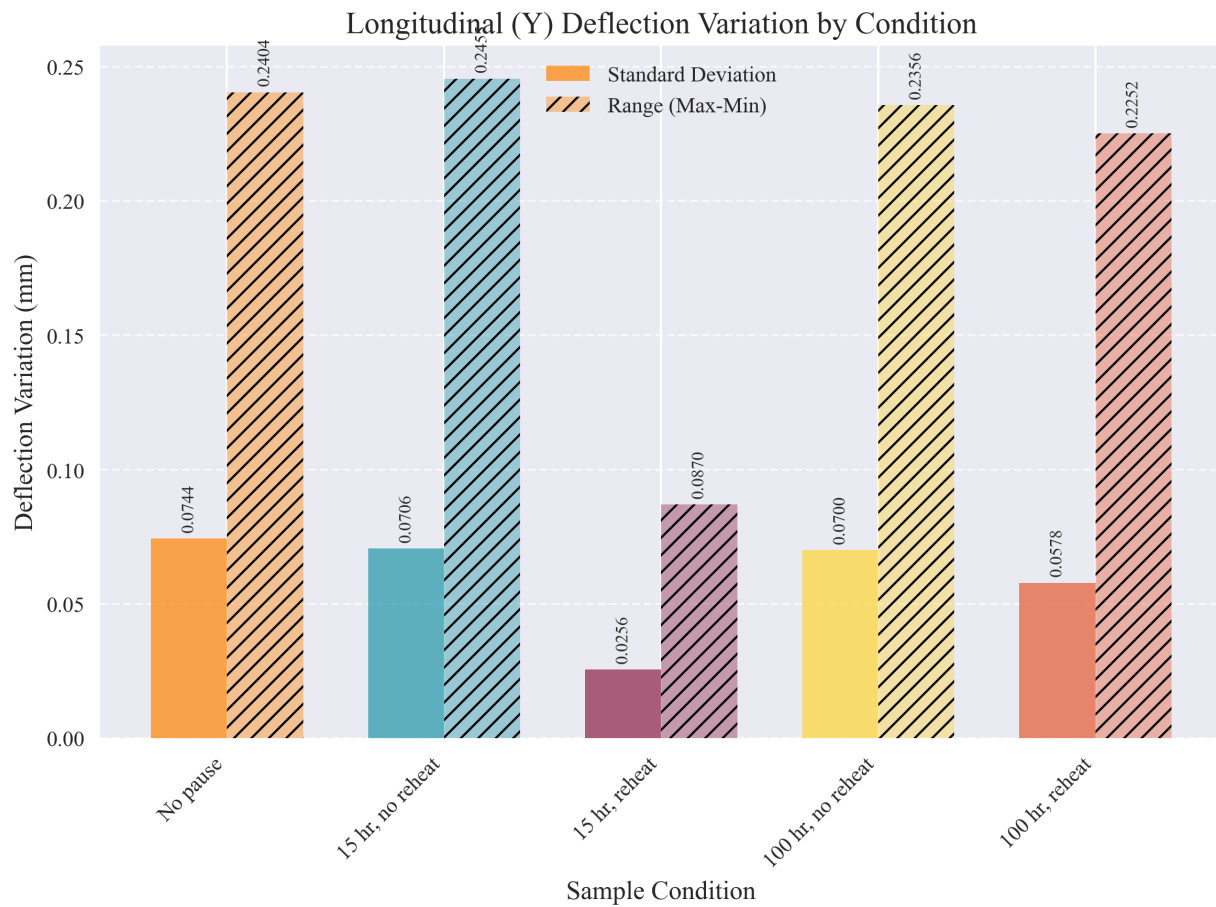


Figure 4.17: Statistical information about deflection across the longitudinal direction of the paused sample test matrix deposits.



Figure 4.18: Optical micrograph taken of a no pause cross section on a Keyence VHX 7000x Optical Microscope.

the location of EBSD data collection, and grain orientation maps analyzed using MTEX are shown in Fig. 4.19. The average grain size was 1.20 μm in the lower region of the sample and 1.61 μm in the upper region. In both regions, the grain sizes skew toward larger values, as shown in the histogram of grain sizes. In each region, grains are non-equiaxed, with the average grain aspect ratio of 2.01 in the lower region and 2.50 in the upper region (2).

The kernel average misorientation (KAM) and grain reference orientation deviation (GROD) are shown in Fig. 4.20. KAM maps show little localized misorientation within grains, indicating that significant recovery has occurred within the material. However, GROD maps show regions with clusters of grains with high GROD. This may have occurred because of the high strain rates during friction stir deposition generating grains with geometrically necessary dislocations from the plastic deformation induced during deposition (2).

4.2.2 X-Ray Diffraction Results

The phase volume fraction of $MgZn_2$, phase volume size of $MgZn_2$ and Al, and hardness vs. build height in the No pause and 100 hr pause, no reheat samples are plotted in Fig. 4.21. In the no pause sample, the volume fraction of $MgZn_2$ is higher than in the 100 hr pause, no reheat sample and highest in the lower build layers. In the 100 hr pause, no reheat sample, the phase volume fraction of $MgZn_2$ is highest in the uppermost layers. The lattice volume of Al in the lowest layers of the no pause sample is smaller than in the lowest layers of the 100 hr pause, no reheat sample. Both samples have similar phase volumes in the uppermost layers after the pause.

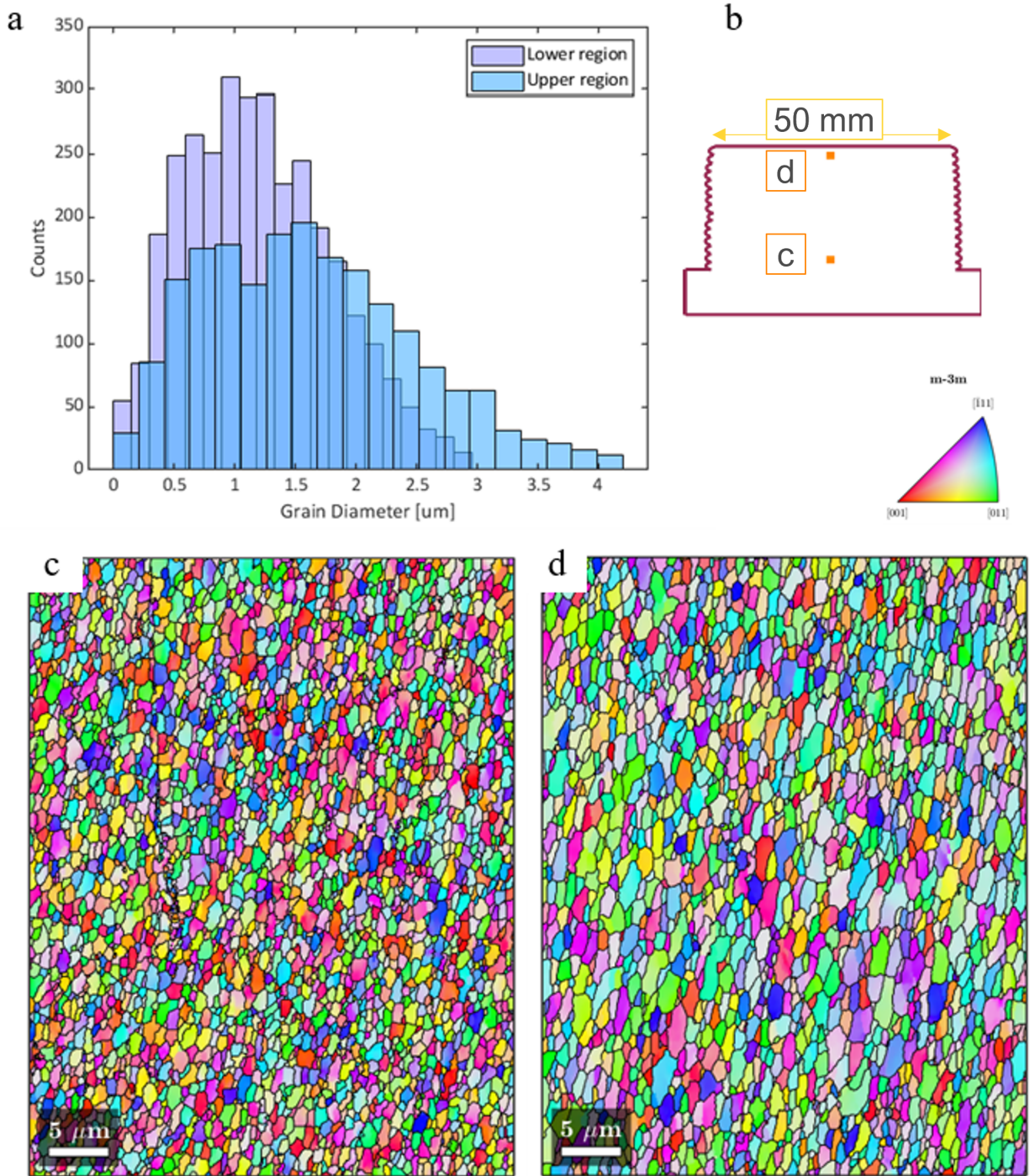


Figure 4.19: EBSD results of the no pause sample. (a) A histogram of grain size statistics, (b) a schematic indicating the upper and lower regions EBSD were performed on, (c) orientation map of the lower region, and (d) an orientation map of the upper region.

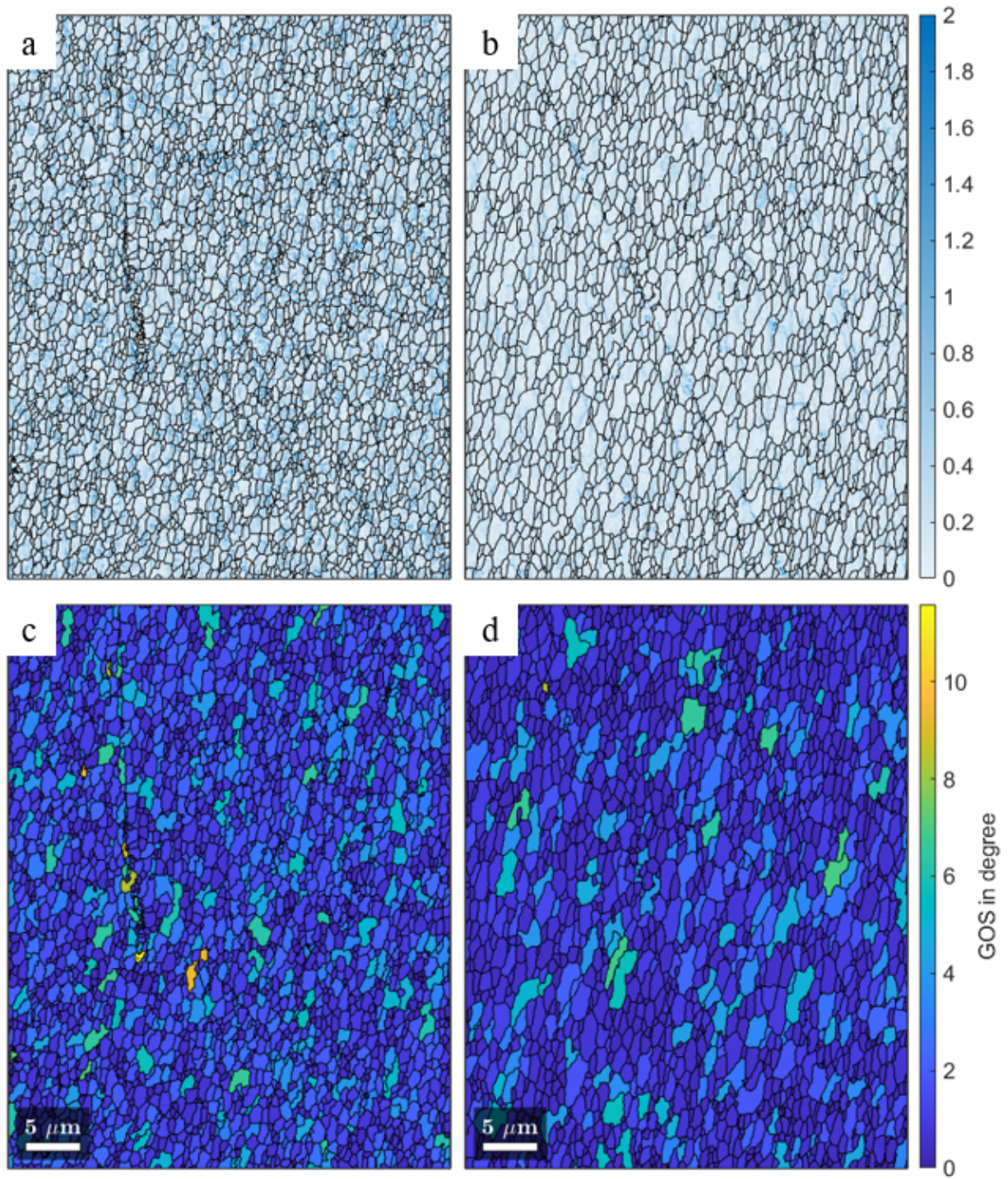


Figure 4.20: Kernel average misorientation (KAM) in degree in the lower (a) and upper (b) region, and grain reference orientation deviation (GROD) maps in the lower (c) and upper (d) regions. All data are from the no pause sample with the same locations as those displayed in Fig. 4.19.

Comparison of Phase Fraction, Phase Volume, and Hardness
Between No Pause and 100 hr Pause (No Reheat) Samples

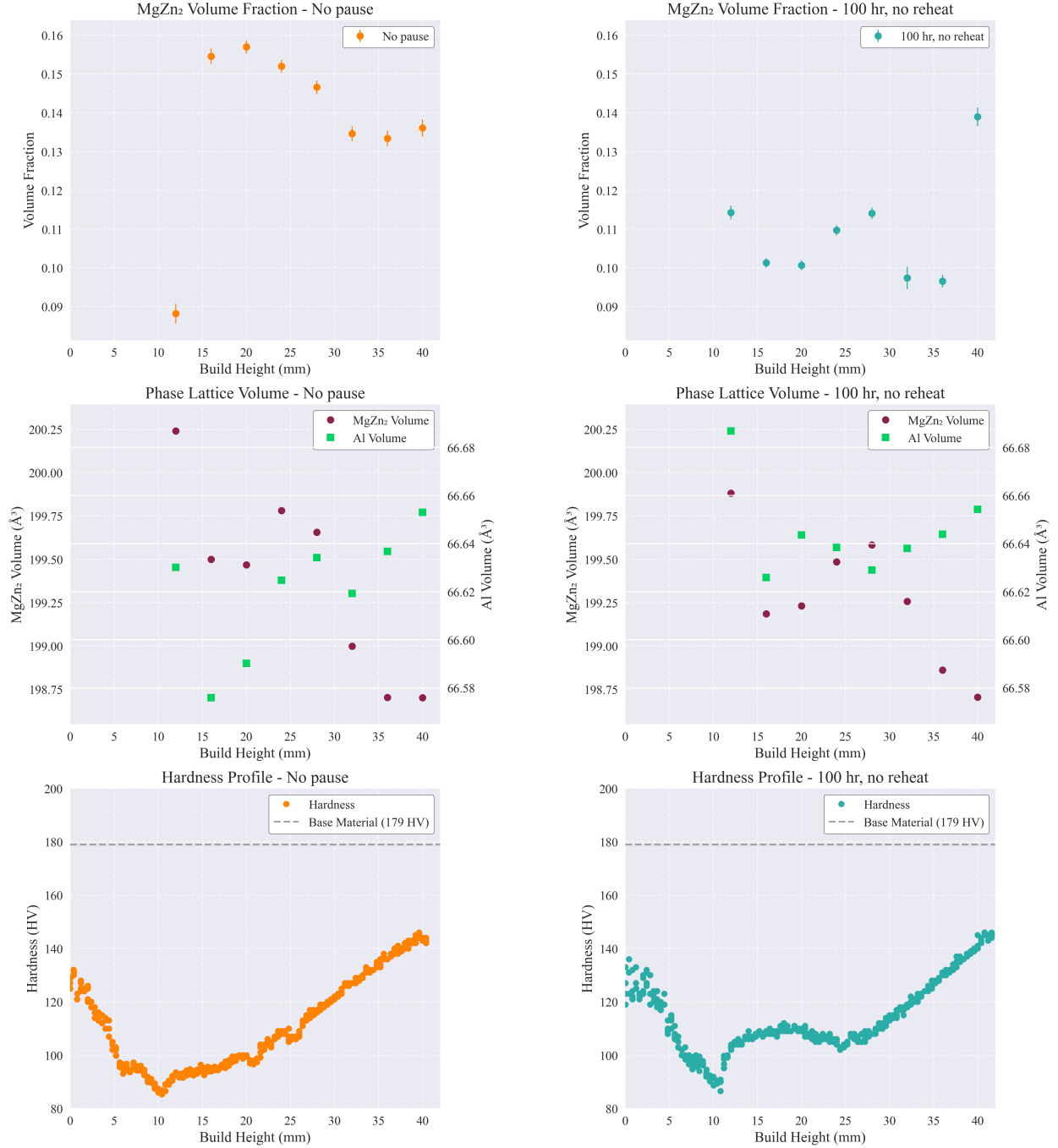


Figure 4.21: Phase volume fraction of $MgZn_2$, lattice volume of Al and $MgZn_2$, and microhardness through the build height in the no pause and 100 hr pause, no reheat samples.

4.3 Residual Stress Analysis

4.3.1 Neutron Diffraction

Unstressed d_0 Spacing Results

The measured d_0 spacing was interpolated to a grid and plotted using a gaussian process regression. The measured d_0 values and associated error values are plotted for the no pause (Fig. 4.22) (2), 100 hr pause, no reheat (Fig. 4.23), and 100 hr pause, reheat (Fig. 4.24) conditions. Estimated error values reported are calculated from a Gaussian process regression implement to impute data. This methodology calculates both the imputed value in regions where directly measured data are sparse and the error associated with that data, weighting the error associated with the imputed data by calculating the covariance. Data nearest to directly measured data have lower uncertainty than the calculated data. These methods have been previously used in neutron residual stress measurements (78). The periodicity in the estimated error of the paused samples is due to a difference in the distance from the directly measured points at different Z heights within the samples.

Residual Stress Results

The residual stress within three different 9.525 mm feedstock samples were calculated from atomic spacing values measured using neutron diffraction at the ORNL HFIR HIDRA beamline. In the no pause sample, calculated microstrain ($\mu\varepsilon$), and calculated residual stress (σ , MPa) are plotted in the transverse (TD) longitudinal (LD) and normal (ND) directions within the build (Fig. 4.25). Calculations were conducted assuming the TD, LD, and ND were the principal directions of stress within the build. In the no pause sample, the largest

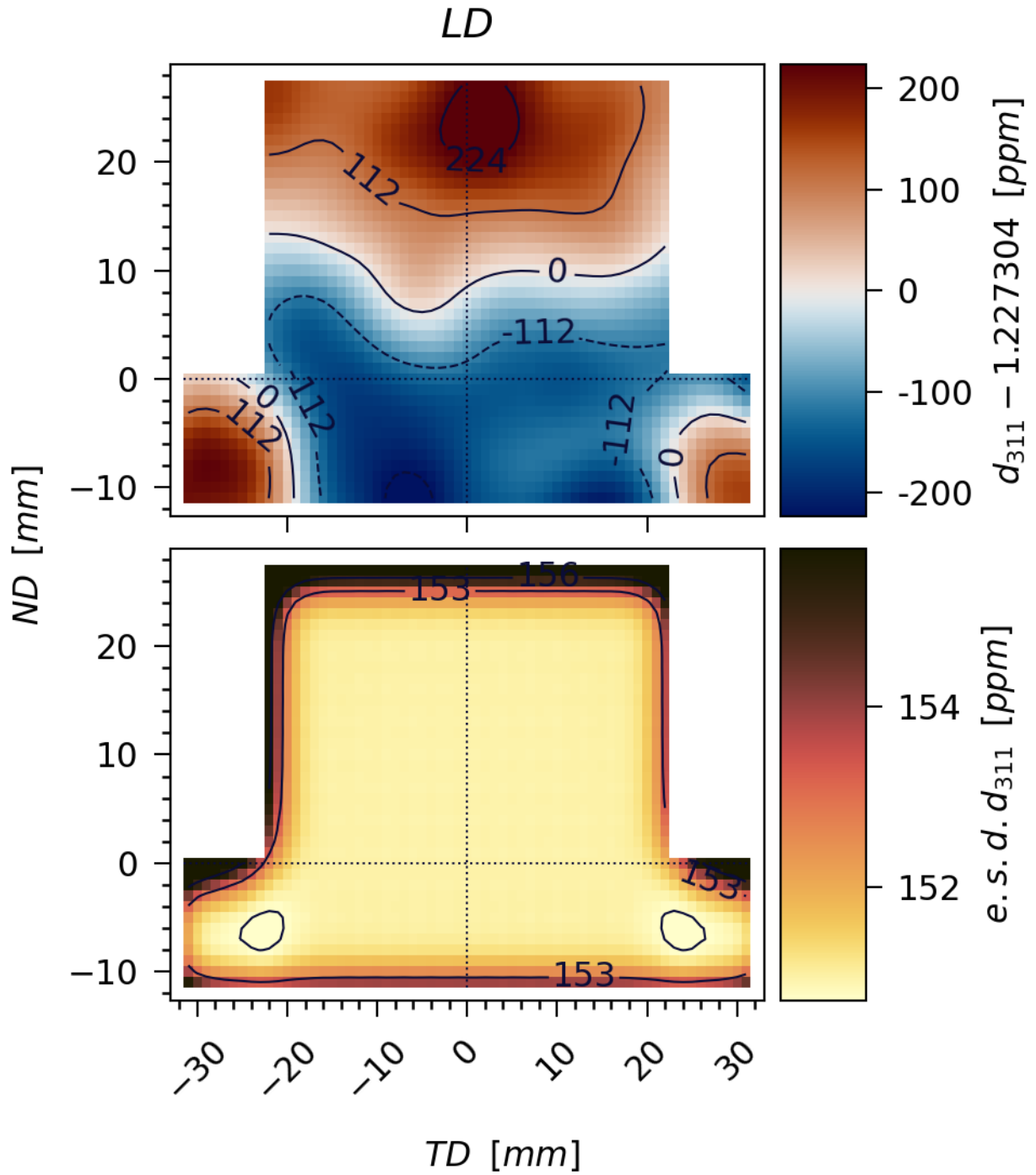


Figure 4.22: Measured d_0 values and their associated error for the no pause sample (2).

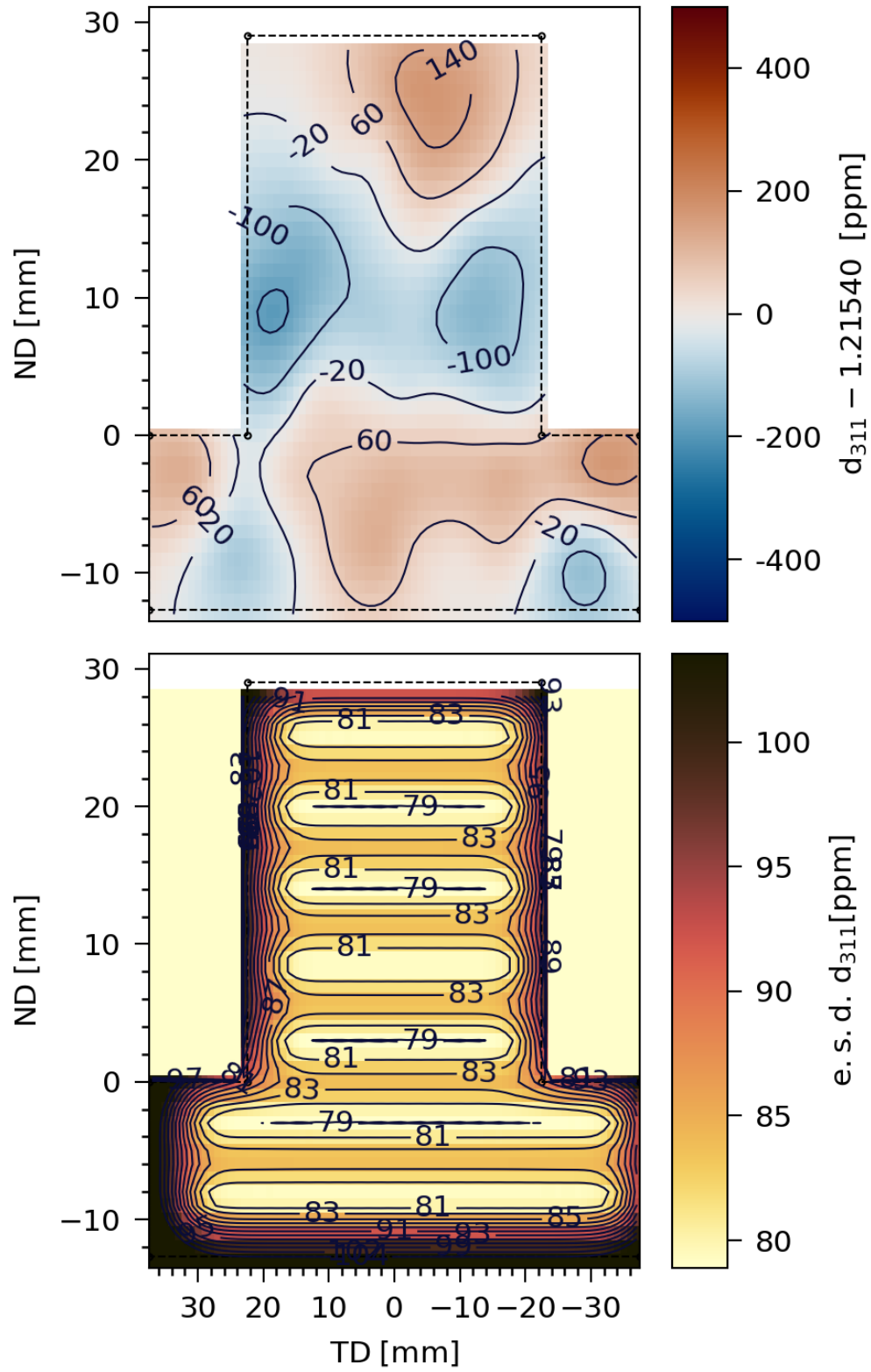


Figure 4.23: Measured d_0 values and their associated error for the 100 hr pause, no reheat.

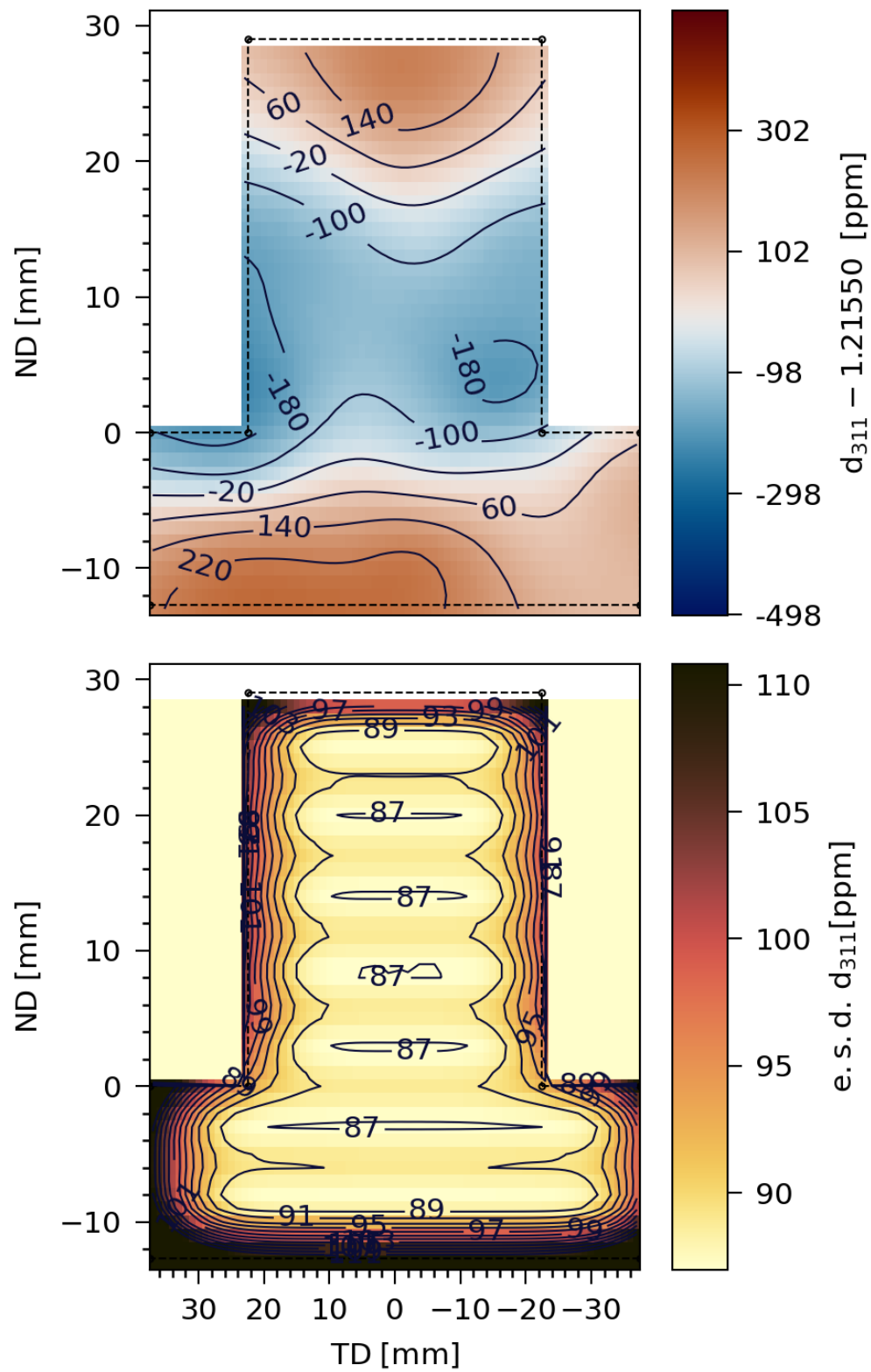


Figure 4.24: Measured d_0 values and their associated error for the 100 hr pause, rehear.

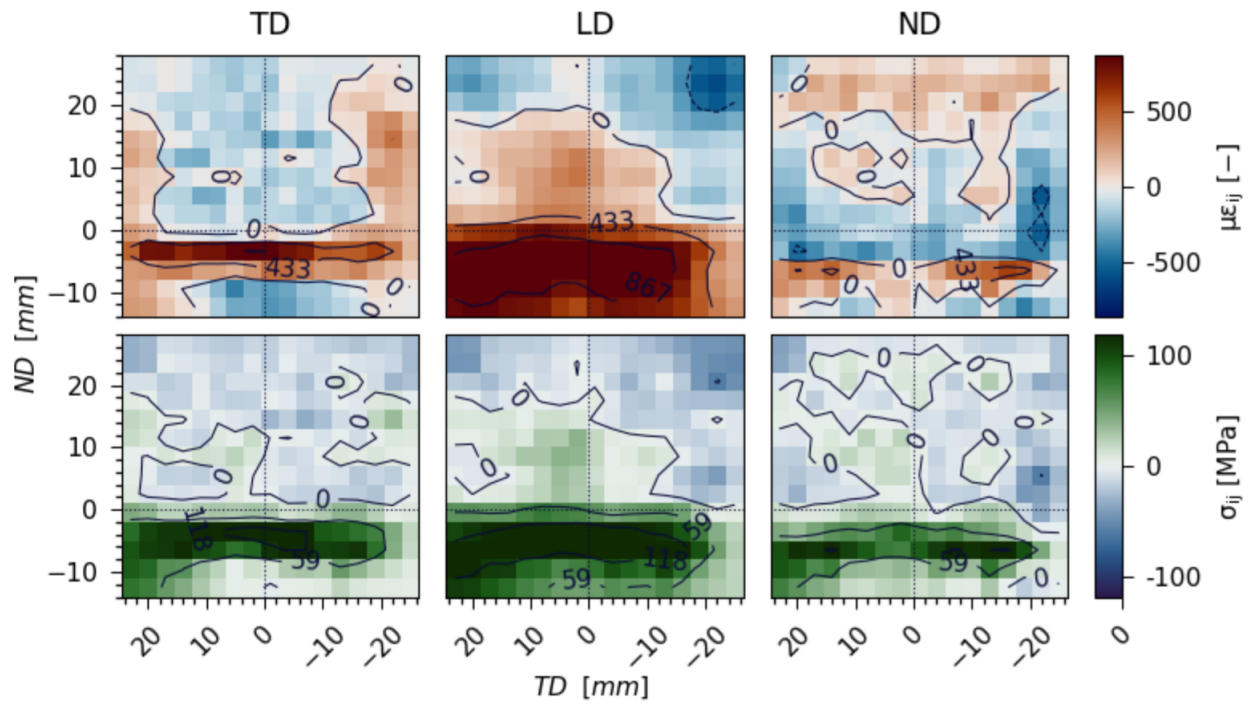


Figure 4.25: Calculated microstrain and residual stress in the three principal directions of the no pause deposit. The advancing side is on the left of the figures.

residual stresses are tensile in nature and occur approximately 5 mm below the base plate and deposit interface in the approximate center of the deposit for the LD (168 MPa) and TD (152 MPa), and on the advancing side of the deposit for the ND (129 MPa). Compressive stresses occur within the deposited material and are of smaller magnitude, with the largest values on the advancing side for the LD (-63 MPa) and ND (-67 MPa) and on the retreating side in the TD (-39 MPa) (2).

In the samples produced with pauses, similar methods were employed with the d_0 spacing, strain, and stress calculations. In the 100 hr pause, no reheat sample, the largest tensile stresses are located in the base plate on the advancing side of the deposit in the TD (112 MPa), approximately 4 mm below the base plate in the center of the deposit in the LD (113 MPa), and near the top on the retreating side in the TD (94 MPa). Maximum balancing compressive stresses are found on the retreating side of the sample in all three directions, with maximum values in the TD (-41 MPa), LD (-66 MPa), and ND (-44 MPa). In the 100 hr pause, reheat sample, the maximum tensile stresses are located immediately below the base plate and deposit interface, slightly biased toward the retreating side, in the TD (104 MPa) and LD (134 MPa). The maximum tensile stress in the ND of this sample is in the baseplate below the edge of the advancing side (78 MPa). The maximum compressive stress values in the 100 hr pause, reheat sample are located on the advancing side just above the base plate in the TD (-17 MPa) and ND (-47 MPa), and near the upper edge on the retreating side in the LD (-45 MPa). The 100 hr pause, no reheat sample strain and stress maps are shown in Fig. 4.26, and the 100 hr pause, reheat sample strain and stress maps are shown in Fig 4.27.

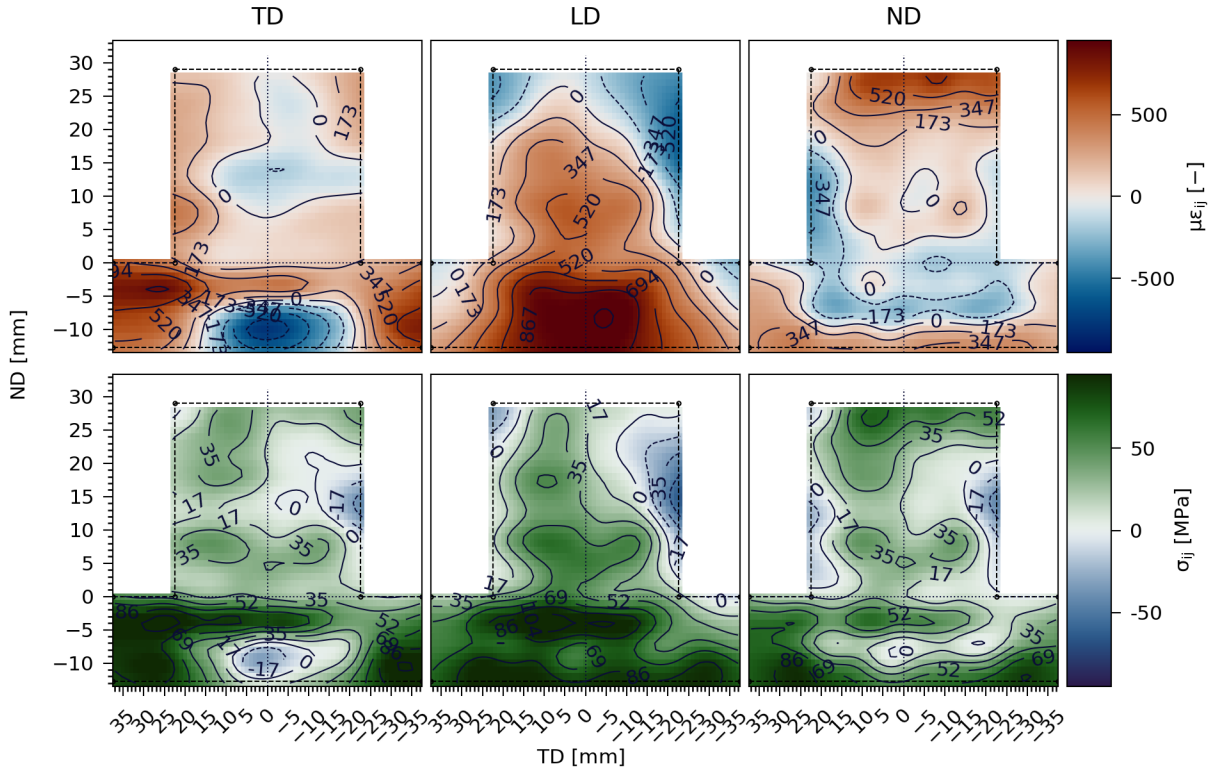


Figure 4.26: Calculated microstrain and residual stress in the three principal directions of the 100 hr pause, no reheat. The advancing side is on the left of the figures.

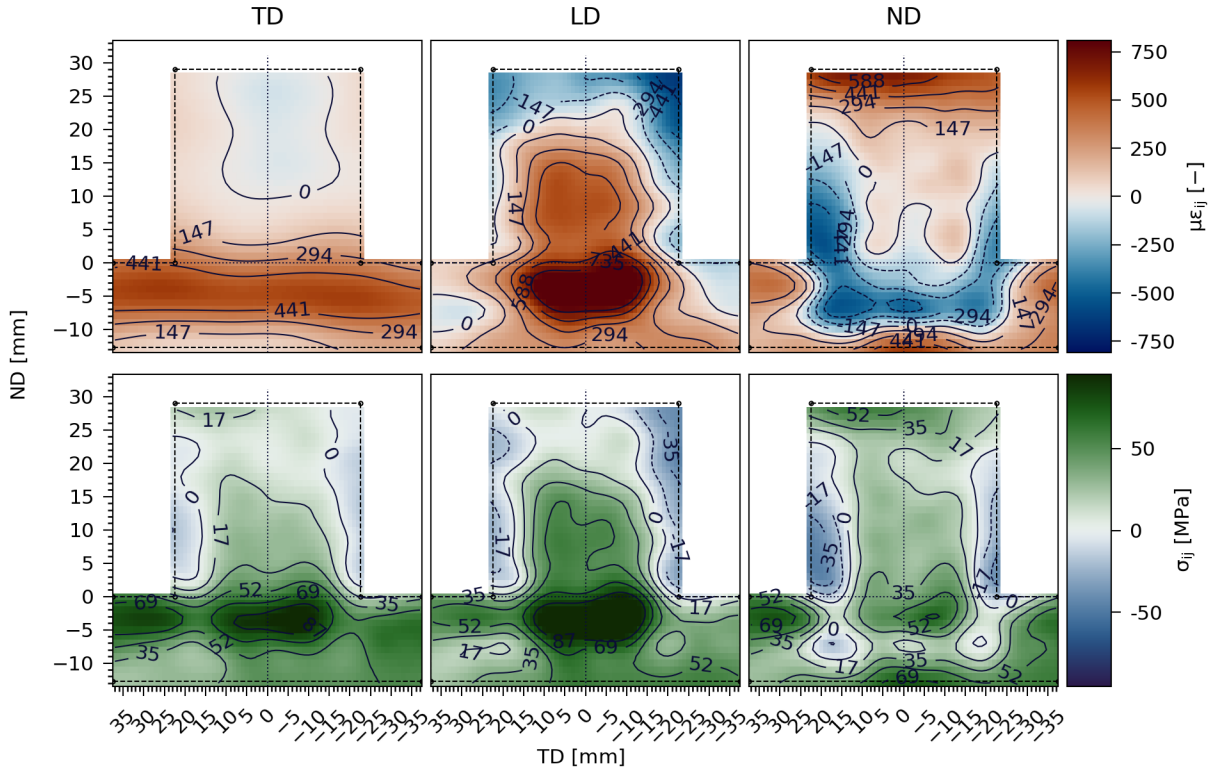


Figure 4.27: Calculated microstrain and residual stress in the three principal directions of the 100 hr, reheat deposit. The advancing side is on the left of the figures.

4.3.2 Contour Method

Contour data were collected on a ZEISS CMM and plotted in Python. Further analysis was conducted on this data in Abaqus FEA software. The results of this implementation are plotted in Fig 4.28. In this implementation, the 100 hr pause, no reheat sample exhibited the largest tensile and compressive stress values when compared to the other samples. In each sample, compressive balancing stresses are found in the outer region of the base plate. The data for each sample are plotted in Appendix ??.

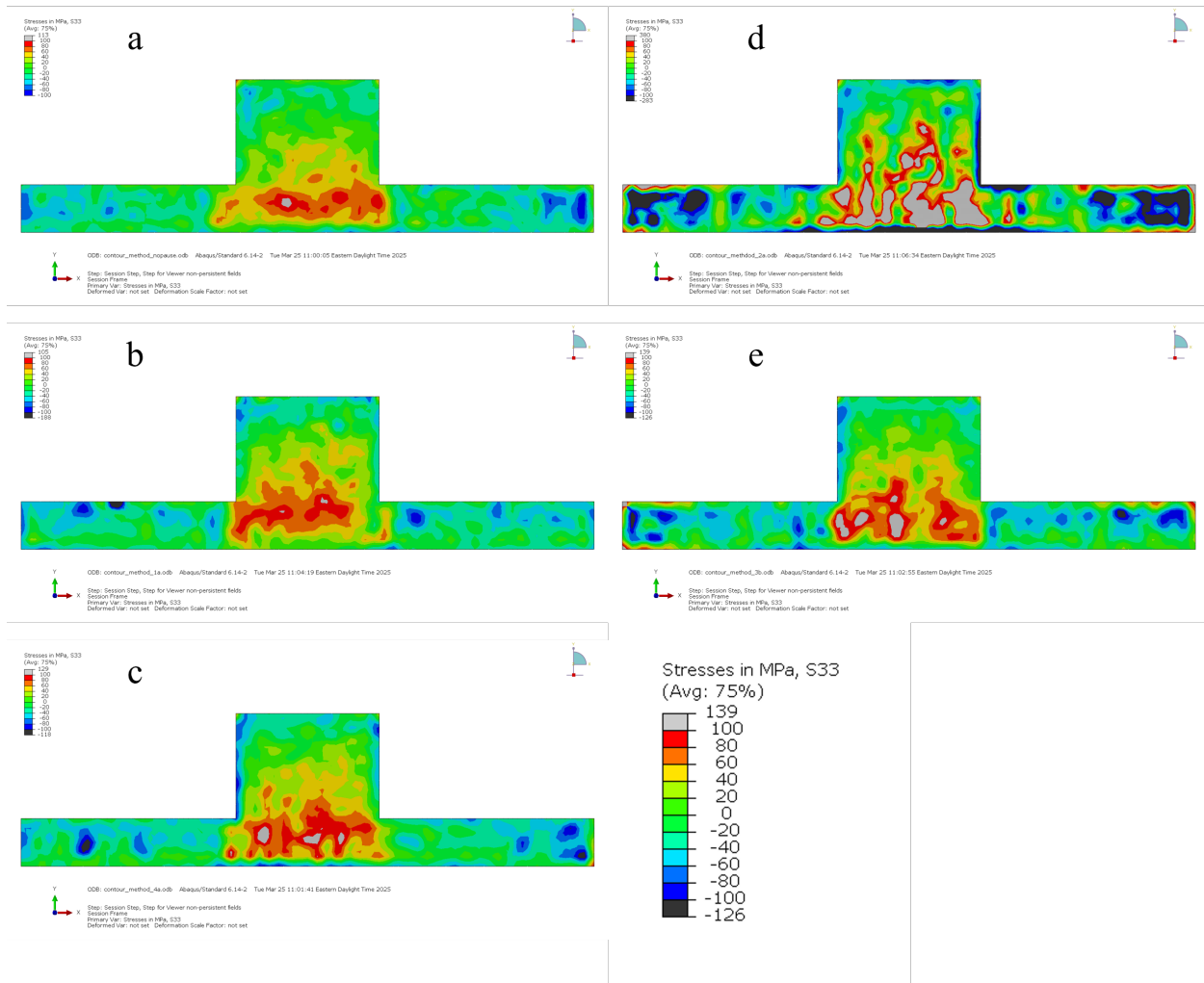


Figure 4.28: Residual stress calculated in Abaqus from contour measurements on the cut surfaces of the samples, (a) No pause, (b) 15 hr, no reheat, (c) 15 hr, reheat, (d) 100 hr, no reheat, and (e) 100 hr, reheat.

Chapter 5

Discussion

5.1 Age Hardening Behavior of AFSD AA7075

A hypothesis for the variation in aging behavior through the build height is that the additional heat the deposit is exposed to as subsequent layers are deposited creates an in-situ heat treatment. Sigworth et al. (43) discussed that 7XXX series alloys can only attain a stable maximum strength using a two-step aging process comprised of a low temperature treatment, below 125 deg C, followed by a high temperature aging step of 140 deg C or higher. They specifically highlighted that artificial aging at temperatures above 160 C will result in loss of strength. Similarly, Polmear (42) found that aging an Al-4%Zn-2%Mg alloy at 90-100 deg C resulted in Vickers hardness of 140 HV, whereas the same alloy aged between 130-240 C was only able to attain a Vickers hardness of 60-70 HV. AA7075 is sensitive to heat treatment conditions due to its quench sensitivity, where prolonged exposure to elevated temperatures promotes the formation of chromium-containing intermetallics that reduce the capacity for slowly cooled material to age harden (79; 38; 43). Therefore, the

layers that were deposited first have a lower capacity for the development of nano-scale precipitates and age hardening when compared to the final layers deposited. In contrast, the uppermost layers do not experience as much time at an elevated temperature, with the final layer only receiving the heat input during deposition taking approximately 3 min. per layer. The thermomechanical processing inherent to AFSD generates dislocations, which serve as nucleation sites for nano-scale precipitate formation that further promotes age hardening (43). The Hall-Petch effect of grain size on microhardness is not assumed to be significant in this material, as the grain size variation through the build height in previous AFSD studies is minimal and the Hall-Petch coefficient for aluminum alloys is low (19; 80).

In the study reported by Schmitz et al. on AFSD AA7075, temperature data captured from thermocouples embedded in the base plate ranged from 67.4 deg C to 301.7 deg C, remaining above 125 deg C for 83% of deposition time (Fig. 4.3) (77). Since similar processing conditions and materials were used in both studies, it was assumed that similar temperature ranges were present in the base plate material studied for aging. This means that the first layer deposited remained between 125 deg C and the deposition temperature of 440 deg C for at least 42 minutes of the 50-minute deposition duration. This is within the range of Polmear and Sigworth's studies, where maximum achievable hardness is reduced to 70 HV, similar to the hardness range observed in the present sample (42; 43). In contrast, the final deposited layer had only a single exposure to elevated temperatures and was immediately quenched in room temperature water within the wire EDM for post-processing, retaining the high dislocation density induced during the AFSD thermomechanical processing and promoting precipitate formation and subsequent material hardening. From this understanding of the temperature range present within the sample

during deposition, the spatial variation in hardness within AFSD AA7075 is interpreted to be an outcome of the layer-by-layer processing. These results are significant as they show region-dependent mechanical behavior within a deposit. Furthermore, these variations in mechanical properties increase in magnitude over time. Given the established correlation between the yield strength and microhardness of aluminum alloys, it is expected that the strength of material deposited at different time intervals will also differ accordingly (81).

5.2 Metallography

The EBSD results (Fig. 4.19) indicate recrystallized grains in both the upper and lower regions. The KAM maps indicate that extensive recovery has taken place with little intergranular misorientation, though GROD maps show some regions with high GROD compared to the bulk of the studied area (Fig. 4.20). Regions of high GROD are suspected to be caused by plastic strain during deposition creating geometrically necessary dislocations. The variation in grain size between the lower and upper regions may be attributed to the variation in thermo-mechanical processing in each region (29). In the first layer deposited, the average spindle rotational speed was 143 rpm and the average tool temperature was 379 deg. C. In the final layer deposited, spindle rotational speed was 101 rpm and the average tool temperature was 410 deg C. In addition to a 30 deg C variation in tool temperature, the different spindle velocities will cause different strain rates, which offers an explanation for the variation in grain size between the first and final deposited layers (2).

5.3 Paused Sample Behavior

With the understanding of the age hardening behavior from the first part of the study, a hypothesis is presented for the cause of the variation in hardness within the material deposited with manufacturing pauses. It is suspected that these variations are a result of age hardening occurring during the hours before the deposition was restarted. All of the paused samples exhibited an increase of hardness near 110 HV within the 3rd - 6th layers deposited before the pause, as opposed to the 95 HV seen in the same layers of the sample produced without a pause. In the paused region, the hardness within the uppermost layers (7-9) deposited before the pause softens when compared to the previous layers. This is likely a result of the thermo-mechanical processing induced during deposition that remixes the uppermost material from the previous layers. The final nine layers in each sample with different pause durations exhibit nearly equivalent hardness at a given layer height. The material within the base plate immediately below the deposit does not appear to age harden even with few layers deposited before a manufacturing pause and remained the softest point in all samples. Nominal d_0 spacing values vary spatially within individual samples and between the no pause, 100 hr pause, no reheat, and 100 hr pause, reheat, another indication that the precipitate and microstructure changes as a result of a pause and restart.

The tensile testing and microhardness results indicate the deposited material microstructure is somewhere between an O temper and a T73. In the O temper state, AA7075 YS is 103 MPa, UTS is 228 MPa, and percent elongation is 17%. In the T73 state, the YS is 434 MPa UTS is 503 MPa, and percent elongation is 13% (38; 75). The results of the deposited material being between a heat treated and an overaged state is consistent with the thermal

histories of the material. By introducing a manufacturing pause and allowing the region to age before reheating, precipitate formation occurs from the near solutionization temperature during deposition. Upon resumption of deposition, the hardened material retains some hardness up to 110 HV, compared to material that has not had time to harden before subsequent deposition cycles losing much of the hardening capacity, down to a minimum hardness near 90 HV. This minimum hardness value appears to be process-dependent, as the 12.7 mm feedstock sample tested for age hardening behavior, which was produced with a different tool and different parameters to accommodate for the different feedstock geometry, had a minimum hardness of 77 HV.

To further understand the importance of the base material properties in AFSD, comparisons may be drawn from FSW literature. In their work studying the mechanical properties of FSW AA7075, Zhu et al. found that FSW of O-temper AA7075 promotes grain refinement and an increase in tensile strength when compared to the base material, while FSW of T6 AA7075 significantly reduces tensile strength with no ductility improvement (82). As the process fundamentals of stirring two materials together are similar for AFSD and FSW, these results indicate that the temper of the base material affects the mechanical performance, and that softer material (such as hot, recently deposited material that has not had a chance to age at room temperature) will result in improved ductile mechanical performance, as demonstrated in this study.

The 100 hr pause, no reheat sample has significant differences in mechanical behavior from the other samples studied - the residual stress, and particularly maximum tensile stress, as calculated by the contour method for this sample was larger. The microhardness in the layers deposited before the pause and restart is noticeably higher than the hardness of the

other paused samples. Under tensile loading, three of the five tensile samples in the 100 hr, no reheat sample set failed prematurely, two while being loaded into the tensile testing load cell and a third very early on during failure, with little ductility. These results indicate that the pause in manufacturing, cold restart, and duration of pause before resuming deposition all influence the mechanical performance of AFSD material. A reduction in ductility is to be expected in harder materials, but this particular sample does not exhibit significantly improved tensile strength over the other samples, indicating that there is little benefit gained by the improvement in microhardness compared to the other samples (38).

Overall, the UTS and YS are similar across all of the samples. Despite their varied thermal histories, the upper regions of the samples have similar microhardness, and therefore, it is expected that the tensile samples extracted from this region will have similar tensile properties. However, most sample sets had repeated failures at a specific Z height, indicating that each AFSD deposit has a weak point in it (Table 4.2). In the 15 hr, no reheat and 15 hr, reheat samples, failure occurred near the commanded 7th layer. The no pause sample failure occurred in the 6th layer of the deposit, and the 100 hr pause, no reheat failure occurred near the commanded 8th layer. Four of the five 100 hr, reheat samples failed in the 10th layer area, and one in the 7th.

XRD data shows a smaller volume fraction of $MgZn_2$ in the 100 hr pause, no reheat sample compared to the no pause sample with an increase in Al lattice size in the 100 hr pause, no reheat sample in the lowest layers compared to the no pause sample (Fig. 4.21, indicating that the introduction of a manufacturing pause changes the precipitation behavior of the deposited material. The introduction of a pause allows for precipitate formation, creating dislocations that slightly widen the lattice volume. Meanwhile, in the sample

produced without a pause, the thermal input encourages the growth of $MgZn_2$ phase to overage the sample to larger, incoherent precipitate particles, increasing the phase volume fraction while simultaneously reducing the regional hardness of the material.

Macrographic analysis of the fracture surfaces indicates that some ductility was present in the failure of each material. While there is a general correlation between the reduction in area and the elongation at failure in each sample, some spread in the results indicates that localized necking occurred in certain samples. The 100 hr, no reheat samples tested exhibited the widest spread in results, with a 4.1% change in area in the sample that failed during loading in the tensile frame, while the other measured sample exhibited a 14.9% change in area and 4.6% elongation at failure. This was the highest measured change in area, indicating that significant localized necking occurred without bulk sample elongation at failure in this specific 100 hr, no reheat tensile sample.

Previous studies have shown that the layer interfaces are compressed during subsequent layer deposition (83), so any effects of the manufacturing pause on mechanical performance might not directly correlate with the Z height at which the pause was commanded on the machine. Instead, they are likely 1-3 mm below the Z height at which the layer was commanded, making the height of 7-8 mm the region of the pause. This offers an explanation for the sample failure locations for the 15 hr pause samples with and without reheats, and the 100 hr, no reheat samples. However, the 100 hr, reheat sample appears to have another weak area in the uppermost layers.

The bulk residual stress analysis from neutron diffraction shows some variation in the deposits. The largest magnitude of tensile stress values occurs in the sample produced with a reheat, but overall, the cold start sample has more material volume in residual tensile stress.

The cold restart region exhibits discontinuities in the residual stress development within the deposited material, whereas the warm restart material exhibits a continuous transition from tensile to compressive stress. By subtracting the cold restart residual stress values from the hot ones, the differences in stress development with the cold restart are visually more apparent (Fig. 5.1). Contour analysis of the AFSD deposits corroborates the neutron residual stress results, with the 100 hr, no pause sample exhibiting the largest tensile stress and the paused sample tensile stresses occurring in higher layers than the stress in the sample produced without a pause.

In summary, manufacturing pauses influence residual stress and mechanical behavior within the material by allowing the deposited material to age harden before continuing deposition, and introducing a thermal gradient in the deposition upon restarting. The residual stress development in material deposited with and without a reheat before restarting is slightly different, with a smoother stress distribution through the build in those restarted with a preheat.

A hypothesis for the higher residual stress and lower ductility of the 100 hr pause, no reheat sample when compared to the samples produced with 15 hr pauses and the reheated 100 hr pause sample is that the 100 hr pause duration allowed significantly more age hardening compared to the 15 hour pause, as evidenced by the age hardening study results. The lack of a reheat will have forced the longer paused material to experience a larger thermal gradient compared to the 100 hr, reheat pause. The combination of a longer pause and no reheat caused the largest variation in microhardness and residual stress within the samples, creating material prone to premature failure with little ductility.

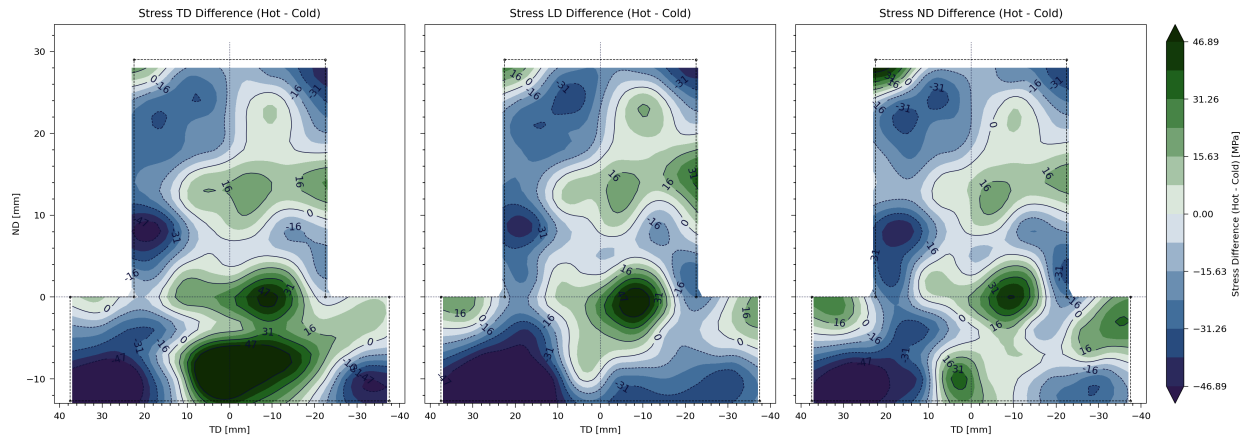


Figure 5.1: The difference between the neutron diffraction residual stress calculations from the samples generated with and without a reheat after the manufacturing pause.

5.4 Influence of Manufacturing Pauses on AFSD Aluminum 7075 Material

By understanding the age hardening behavior of AFSD material, with the final layers of the deposit retaining hardness after deposition, the upper ~ 20 mm of the deposit steadily hardening after deposition, and the material deposited prior to that staying in the O-temper hardness regime over the period of months, the cause of the defects present in the large component which motivated this study (Fig. 1.1) can be better understood. The deposition of hot material onto cold material, coupled with part size being more than 10x greater than those studied here, will have ensured that the hardest material nearing T7 properties is intimate contact with the softest material with O-temper qualities. Combining this knowledge with the significant tensile stresses found in as-deposited AFSD aluminum, and the results of previous studies finding that stresses in additively manufacturing components will increase in magnitude with increased number of layers and component size (84), provides evidence that the delamination that motivated this study likely occurred due to a combination of the varied mechanical properties in the as-deposited material and high tensile residual stresses.

Chapter 6

Conclusions and Recommendations

6.1 Summary

A series of AFSD AA7075 samples were produced using a closed-loop temperature control mechanism to create single-walled geometries with pauses of approximately 15 and 100 hr durations introduced half way through some builds. Some paused samples were reheated to 120 deg C before restarting, while others were restarted without the reheats. The resultant depositions were scanned with structure light scanning to measure base plate deflections. Select samples were studied using neutron diffraction for residual stress characterization. After bulk properties were captured, samples were cut in half via wire EDM, and the surface displacement on the cut surfaces was measured on a CMM. Samples were extracted from the bulk material and prepared for tensile testing, hardness testing, and metallographic analysis.

In an AFSD AA7075 sample produced without a pause, microhardness in the central region of the deposit was tested through the build height after 10 hrs, 1 week, 4 weeks, and 15 weeks. The base plate material was softest in the region immediately below the base

plate and deposit interface. The material in the base plate did not have a change in hardness following deposition. However, the maximum hardness in the deposited material increased from 113 HV to 147 HV over the fifteen weeks of testing. The aging behavior varied spatially through the build height of the material, with subsequent heating of each layer reducing the hardening capacity of the previous layers.

The paused AA7075 samples were tested for microhardness and tensile strength. Baseline hardness was measured across the build width and height in the no pause sample, finding a spatial variation in microhardness in the build height and additionally, a trend toward harder material in the uppermost layers on the retreating side compared to the advancing side. All samples were then tested in the central region of the deposit through the base plate and deposited material. In the paused samples, the layers below the pause interface hardened before deposition resumption. This resulted in a difference in mechanical hardness between the no pause and paused samples in the first nine layers, with the paused sample hardness reaching between 100 and 110 HV in the first 6 layers while the no pause hardness range was between 90 and 100 HV. In the 100 hr pause, no reheat sample, the hardness was slightly higher.

All of the samples UTS and YS were between the O-temper and T73 values, as is to be expected in W-temper material which has been allowed to age. Tensile testing of the samples demonstrated that the AFSD material had low ductility, with elongation at failure ranging between 1.2%-9.1%. The 100 hr pause, no reheat sample overall had the greatest reduction in ductility, hardest material in the first nine layers, and largest residual stress and surface deflection on the EDM surface measured via CMM.

Neutron diffraction analysis of the deposited material shows all samples have a concentration of tensile residual stress in the base plate and deposit interface. The addition of a manufacturing pause with a warm restart increased the residual tensile stress in the central region of the layers deposited before the restart. In the deposit with a cold restart, the longitudinal direction residual stress within the first half deposited and second half deposited both are tensile in nature, with a visible variation in stress in the immediate region of the manufacturing pause seen in mapped residual stress contours. The measured d_0 spacing in each of the studied samples varies, indicating the changes in thermo-mechanical processing history affected the evolution of microstructure and atomic spacing.

6.2 Significance of Study

This dissertation presents a first of its kind study characterizing the impact of manufacturing pauses on the mechanical properties and residual stress in AFSD. It was motivated by observations of delamination in a large scale AFSD part at the location of a manufacturing pause and resumption. The results of this study indicate that the thermo-mechanical processing nature of AFSD generates material prone to age hardening. However, deposition of additional layers acts as an in-situ heat treatment of the material, reducing the age hardening capacity with each passing layer. The introduction of a manufacturing pause between layers provides the material with time to cool and begin aging, inducing mechanical and microstructural heterogeneity in the deposit. The results of this study advance the currently available literature, providing insights into practical implementation of AFSD AA7075.

6.3 Study Limitations

The focus of this study was quite narrow - AFSD of AA7075, using single wall builds with a maximum of eighteen layers. Practical application of AFSD will involve larger, more complex geometries and a variety of different alloy systems. In addition, this study was limited to the available AFSD equipment, a discrete feed machine that had to be reloaded every 3-4 layers. These reloads change the thermal history of deposition compared to deposits manufactured on continuous feed machines.

6.4 Future Work

Future studies should focus on more practical geometries of AFSD, and how the thermo-mechanical process variation with overlaps and re-stirring of material influences aging and mechanical property development in AFSD material. Studies characterizing the impact different layer heights and number of layers have on aging behavior and mechanical property development in AFSD are recommended. Additional studies are recommended on modeling the residual stress development in AFSD material. The results of this study offer baseline experimental data from which models could be tuned. Additional work is also recommended characterizing the impact of manufacturing pauses on AFSD of other alloy systems.

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Appendix A

Additional Figures - Paused Samples

A.1 No Pause Data

A.1.1 Images

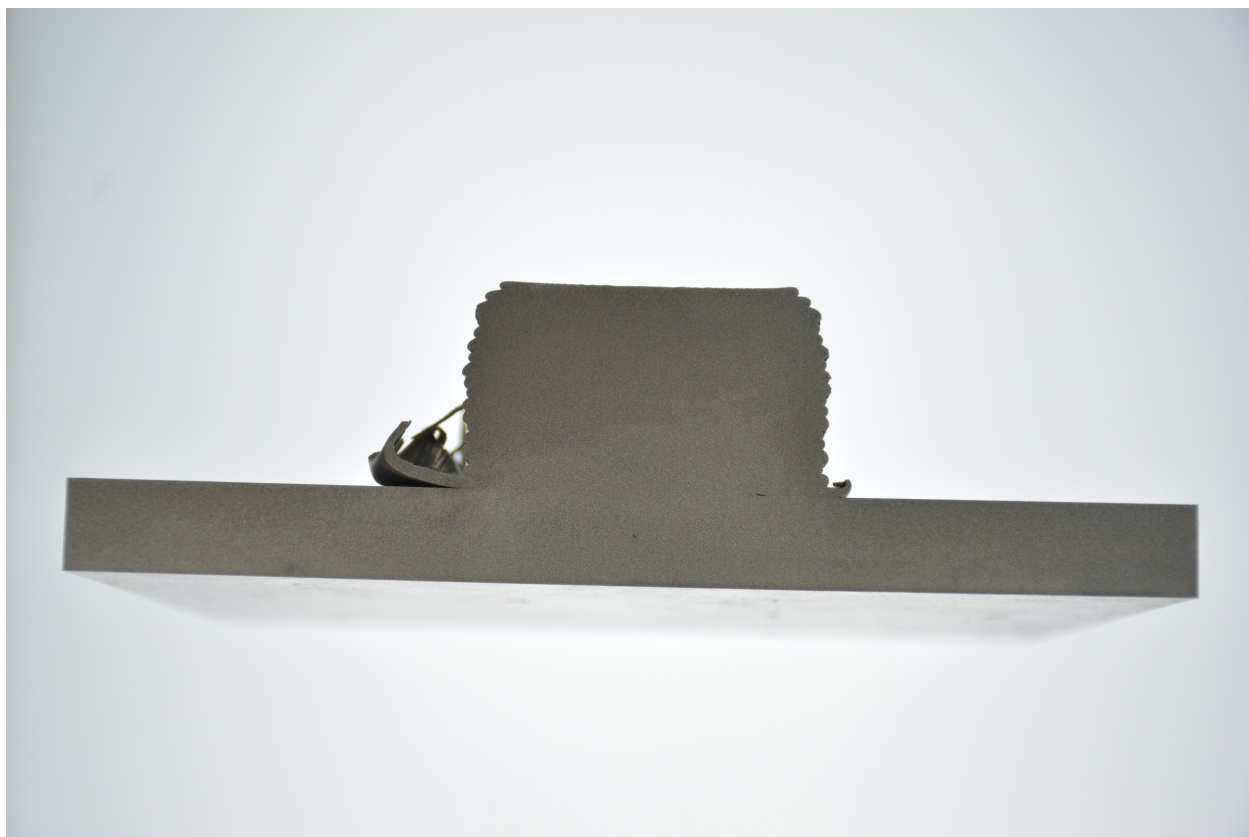


Figure A.1: Photo of no pause sample after cutting it in half for contour measurement.

A.1.2 3D Scanning Data

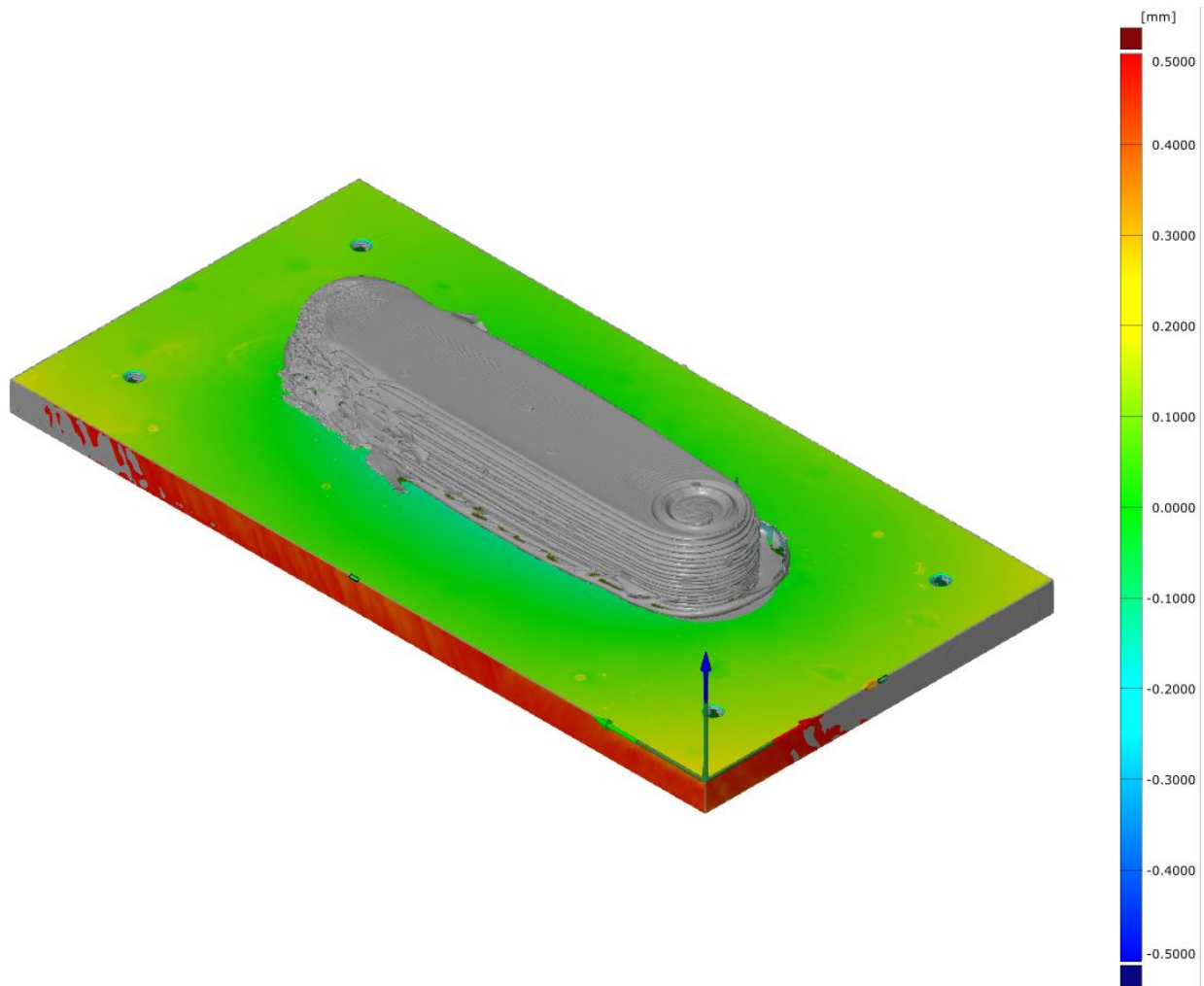


Figure A.2: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

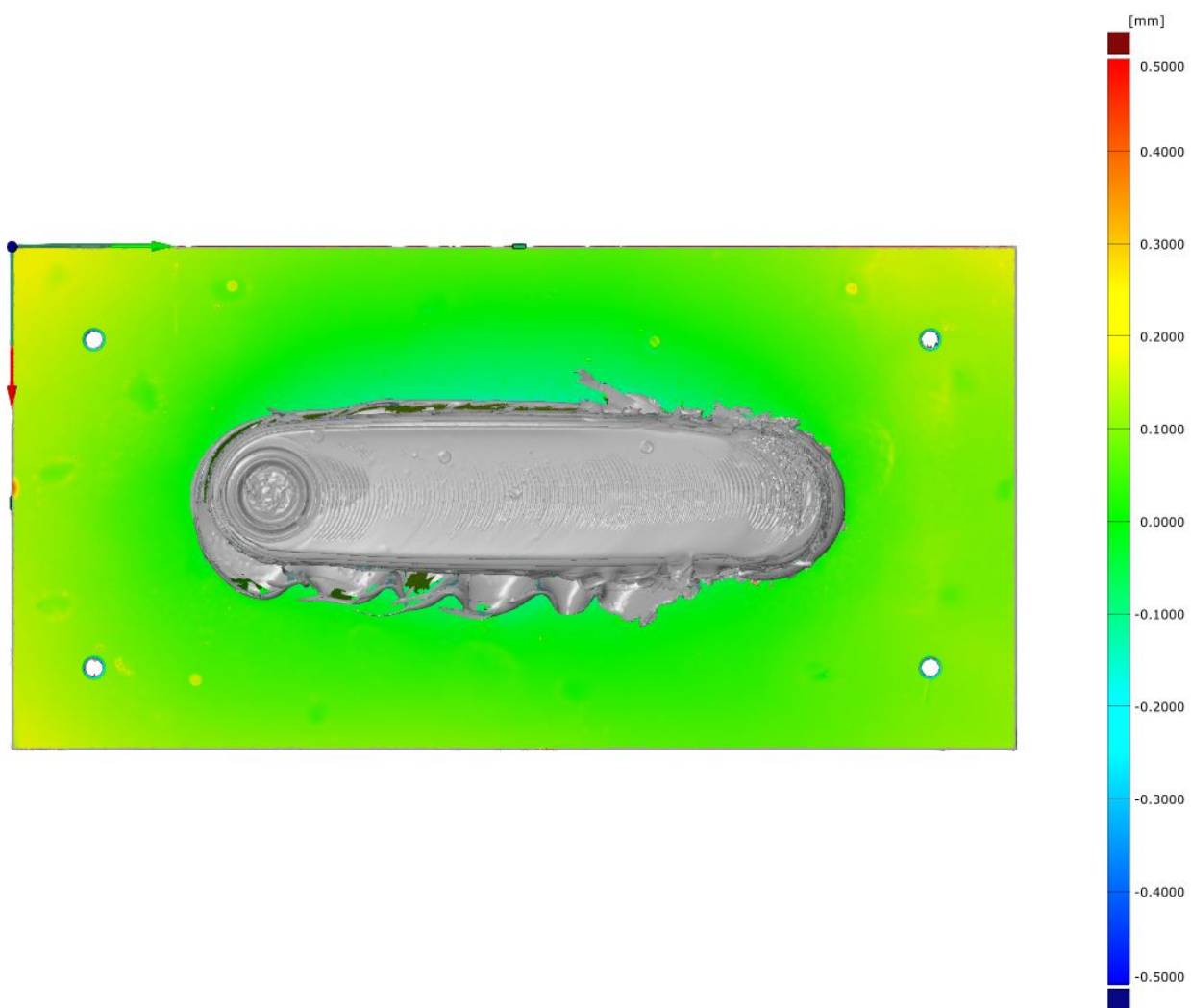


Figure A.3: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

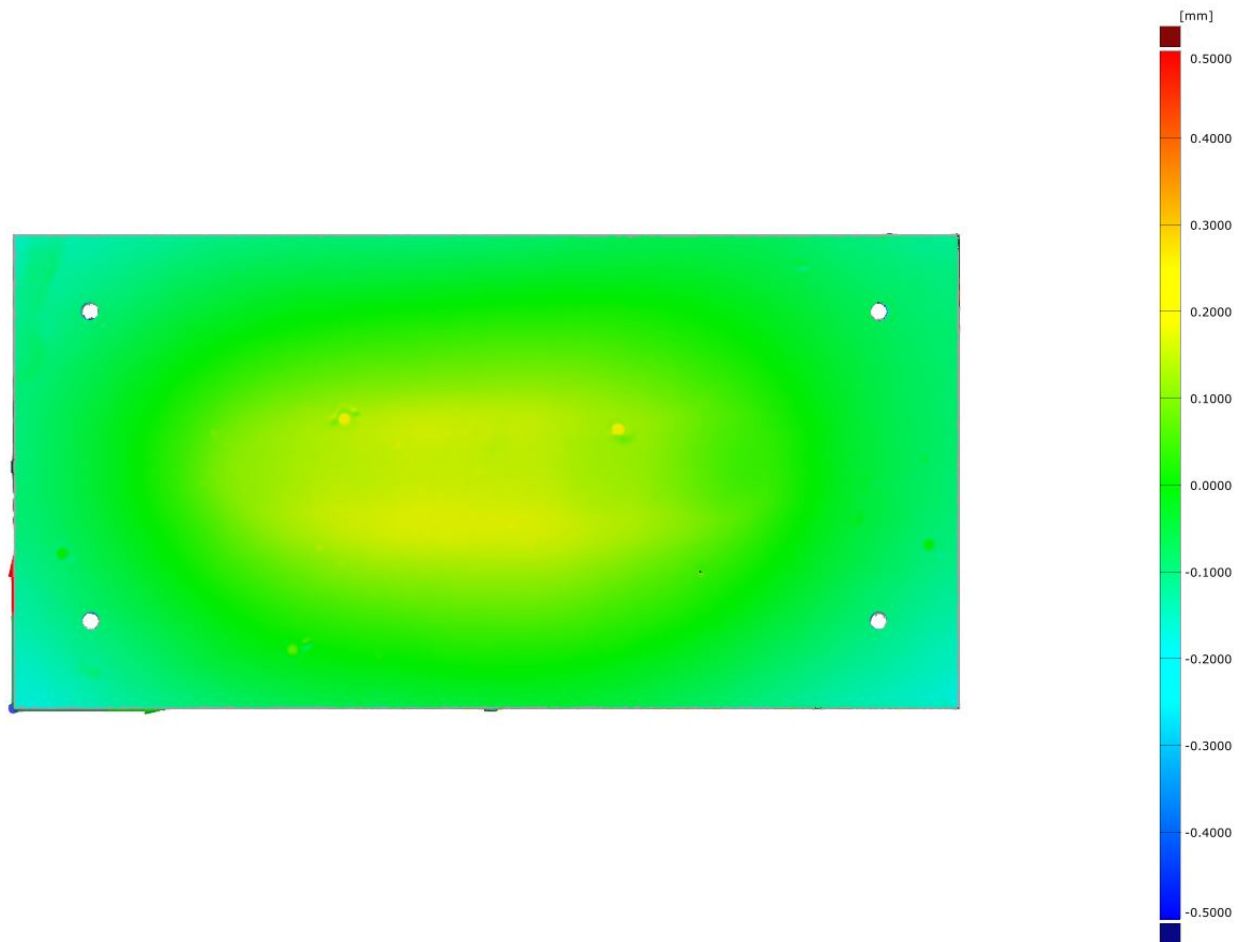


Figure A.4: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.1.3 Contour Data

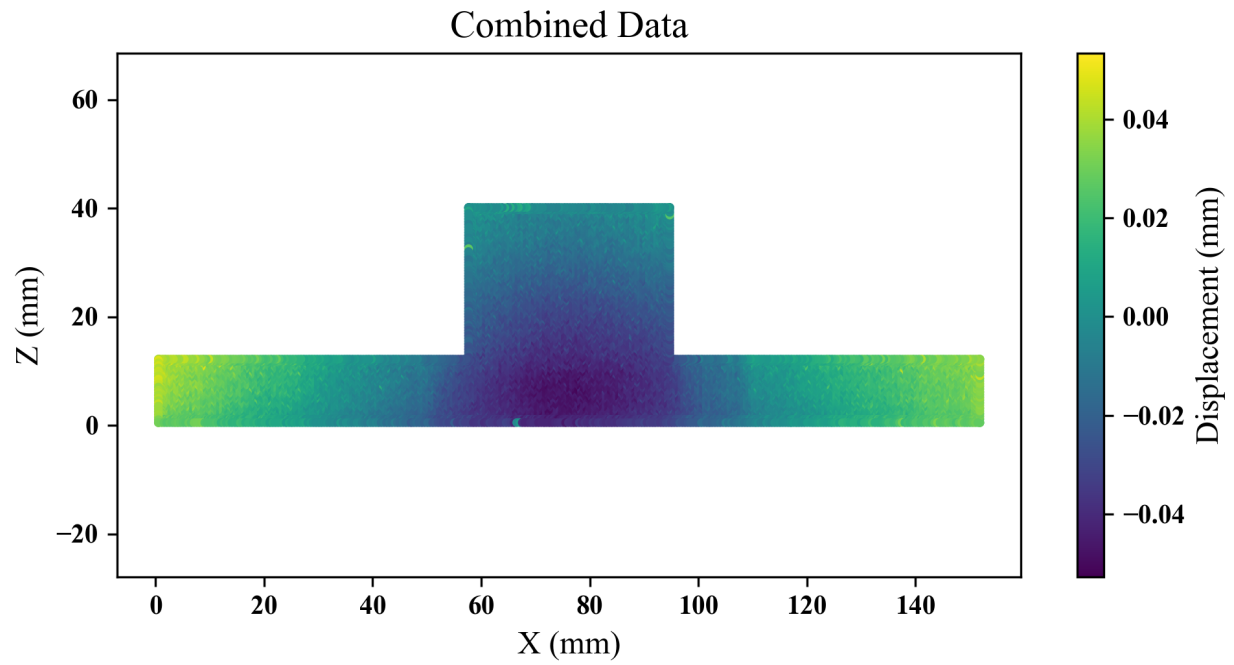


Figure A.5: The raw CMM data combined into a single file.

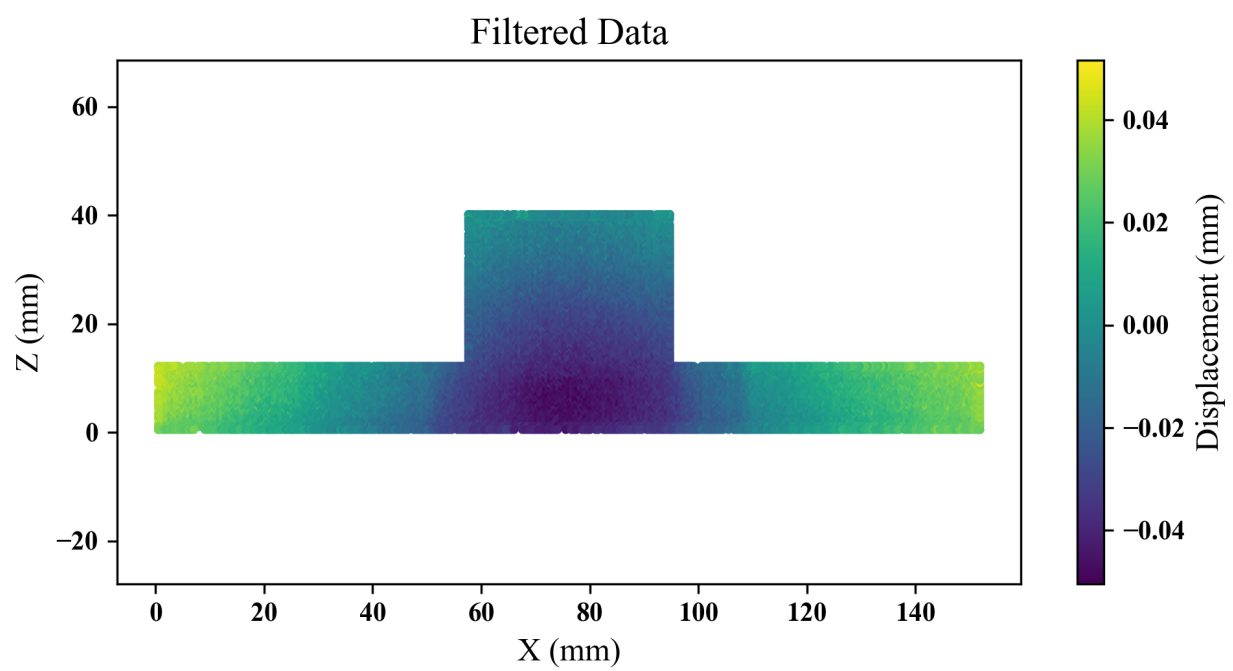


Figure A.6: Outlier elimination filtered data from the no pause sample.

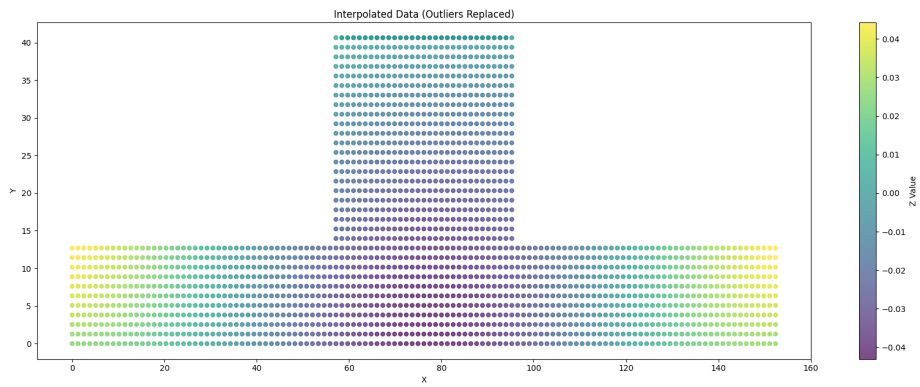


Figure A.7: No pause data interpolated to a grid of 1.27 mm.

A.2 15 hr Pause, No Reheat Data

A.2.1 Part a

A.2.2 Sample Photos



Figure A.8: Photo of sample 1a after cutting for contour measurement.

A.2.3 3D Scanning Data

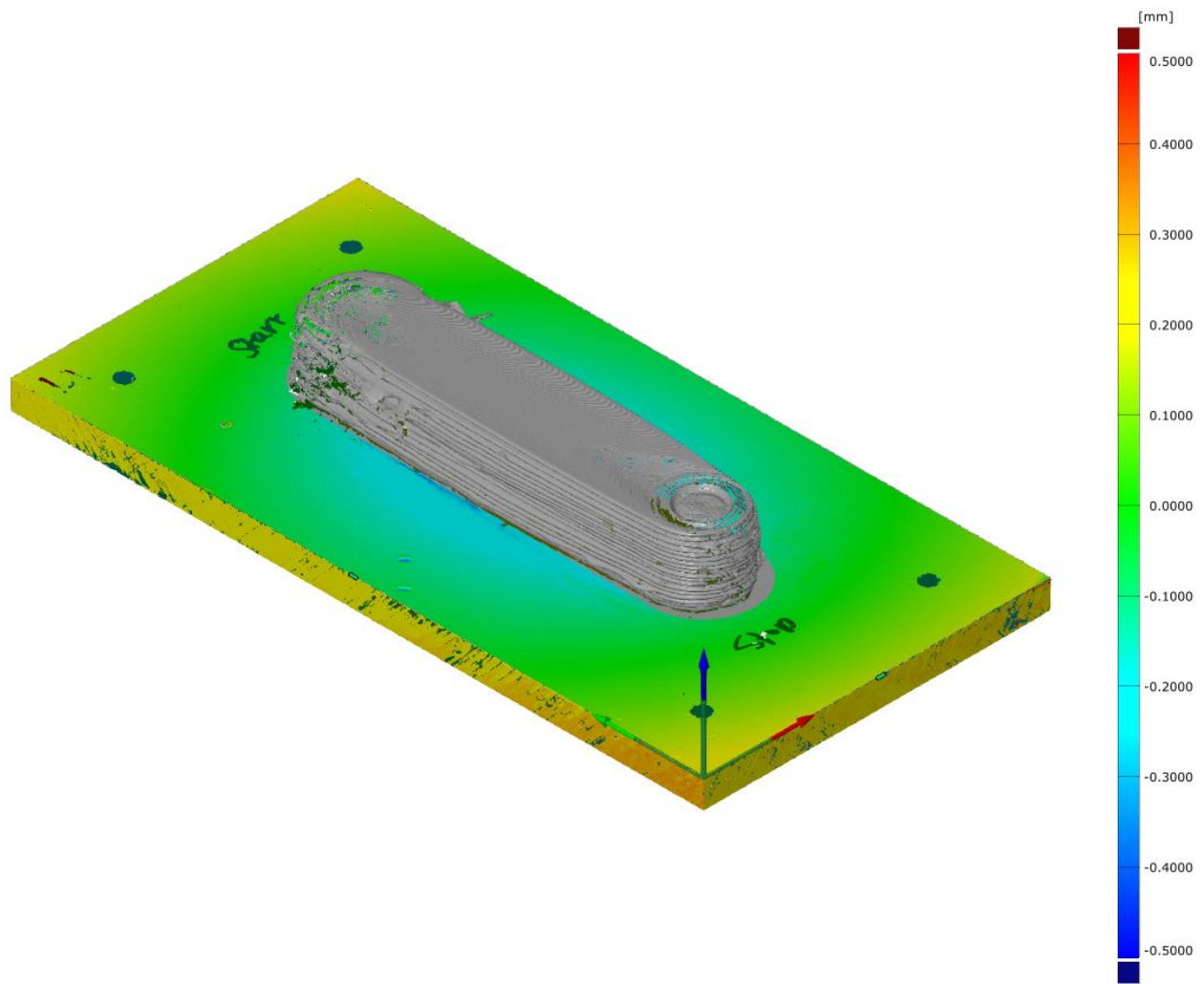


Figure A.9: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

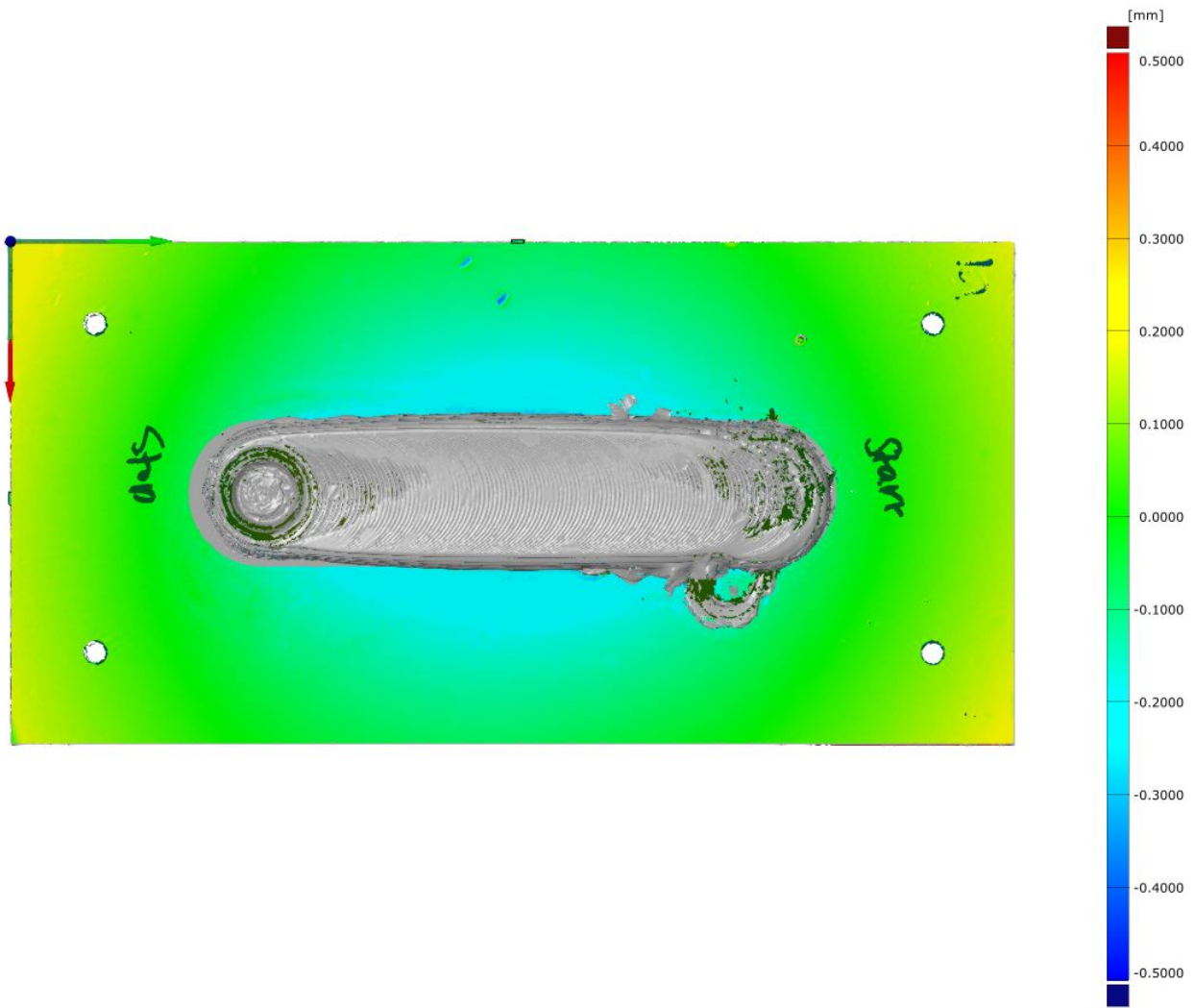


Figure A.10: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

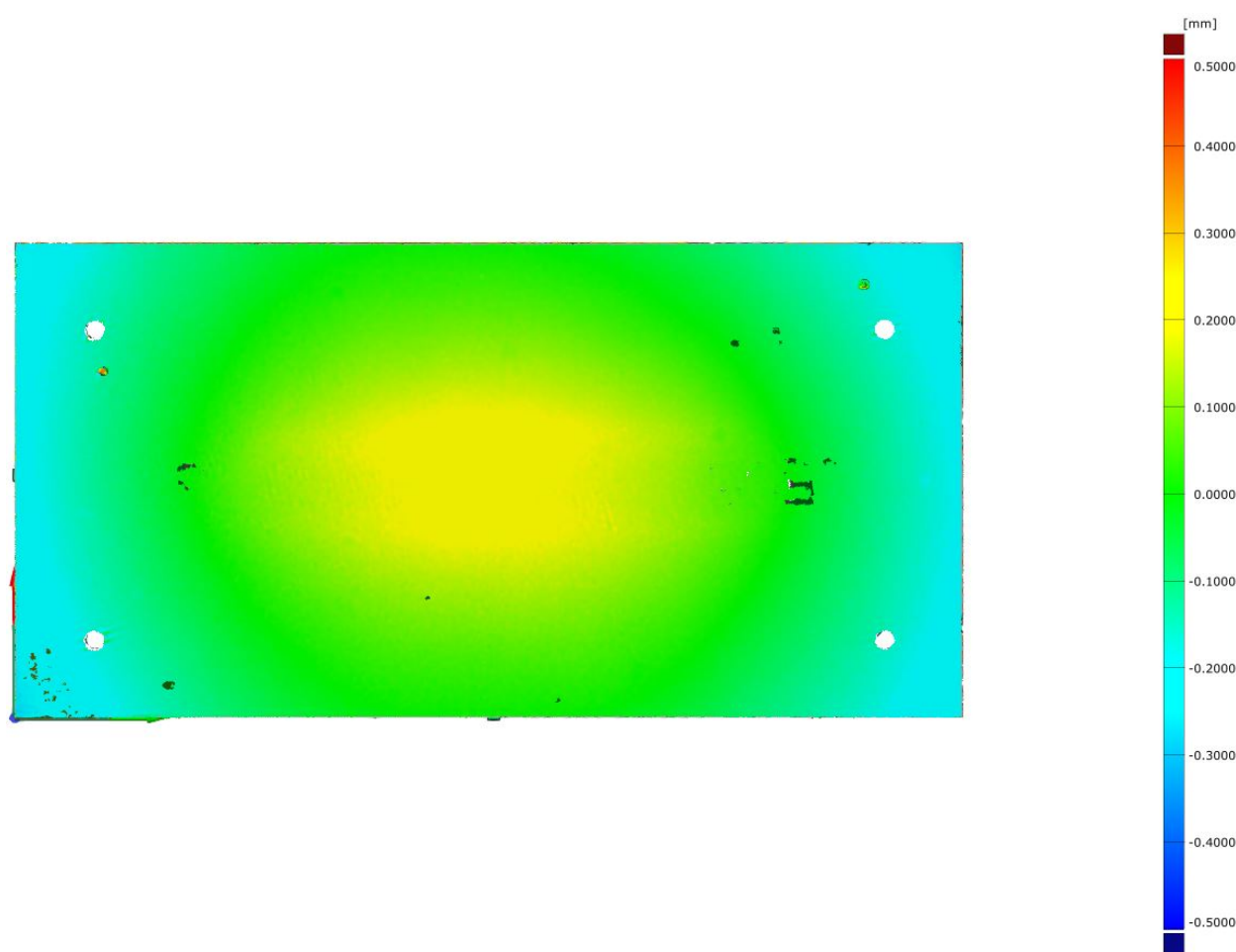


Figure A.11: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.2.4 Part b

A.2.5 Sample Photos

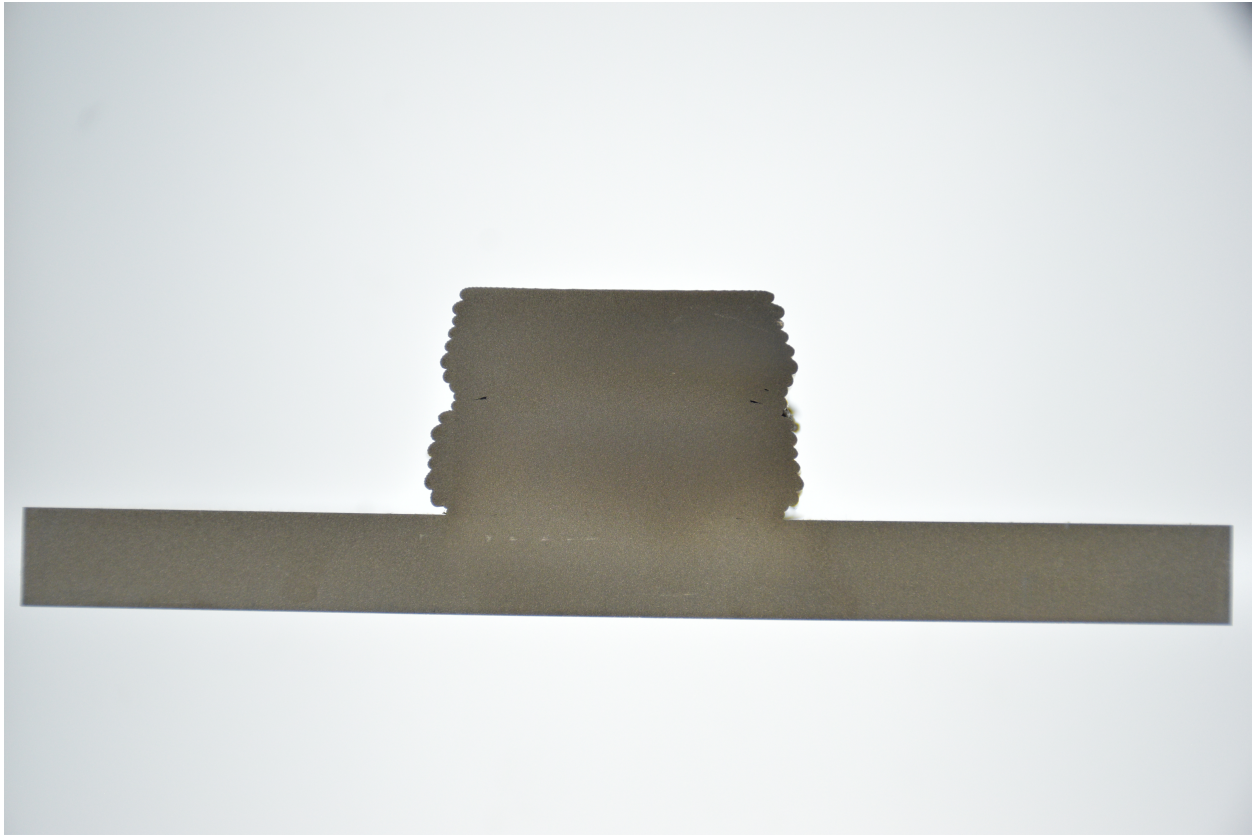


Figure A.12: Photo of sample 1b after cutting for contour measurement.

A.2.6 3D Scanning Data

A.3 15 hr Pause, Reheat Data

A.3.1 Part a

A.3.2 Sample Photos

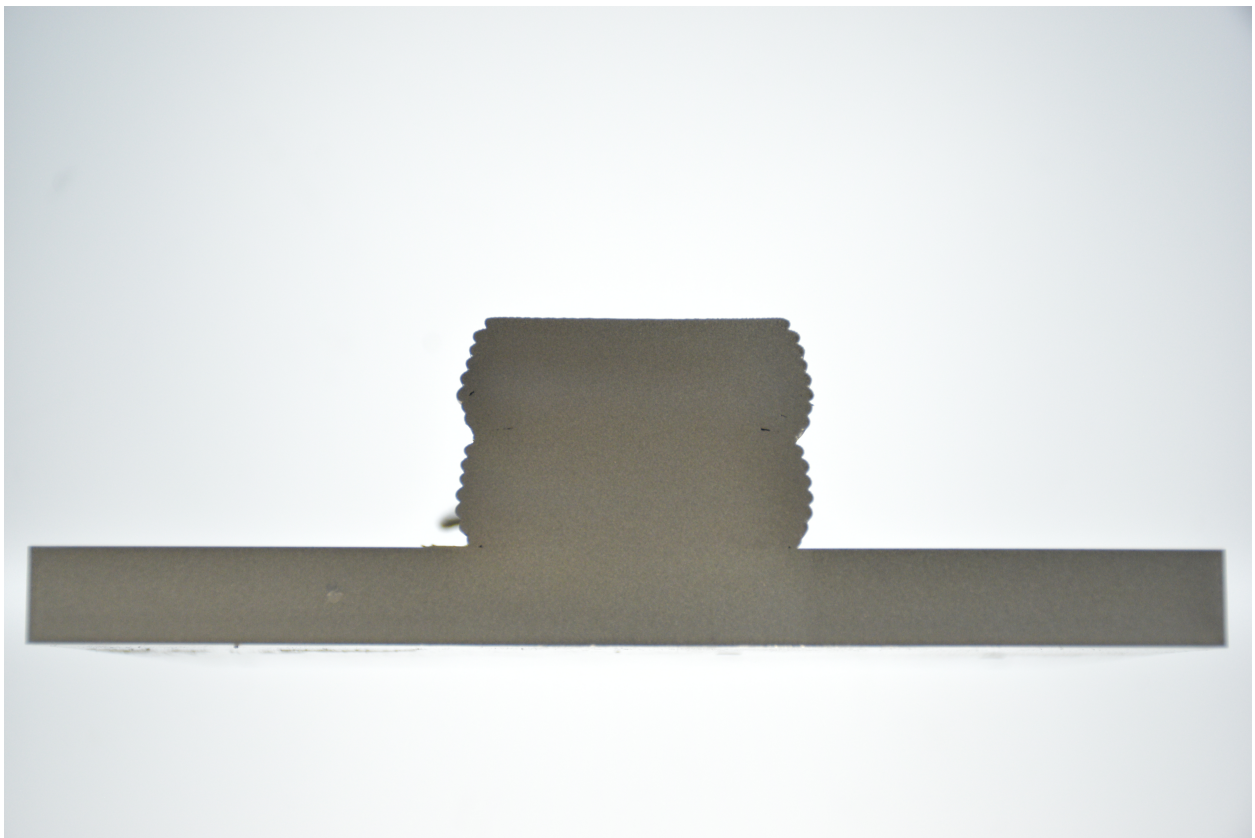


Figure A.13: Photo of sample 4a after cutting for contour measurement.

A.3.3 3D Scanning Data

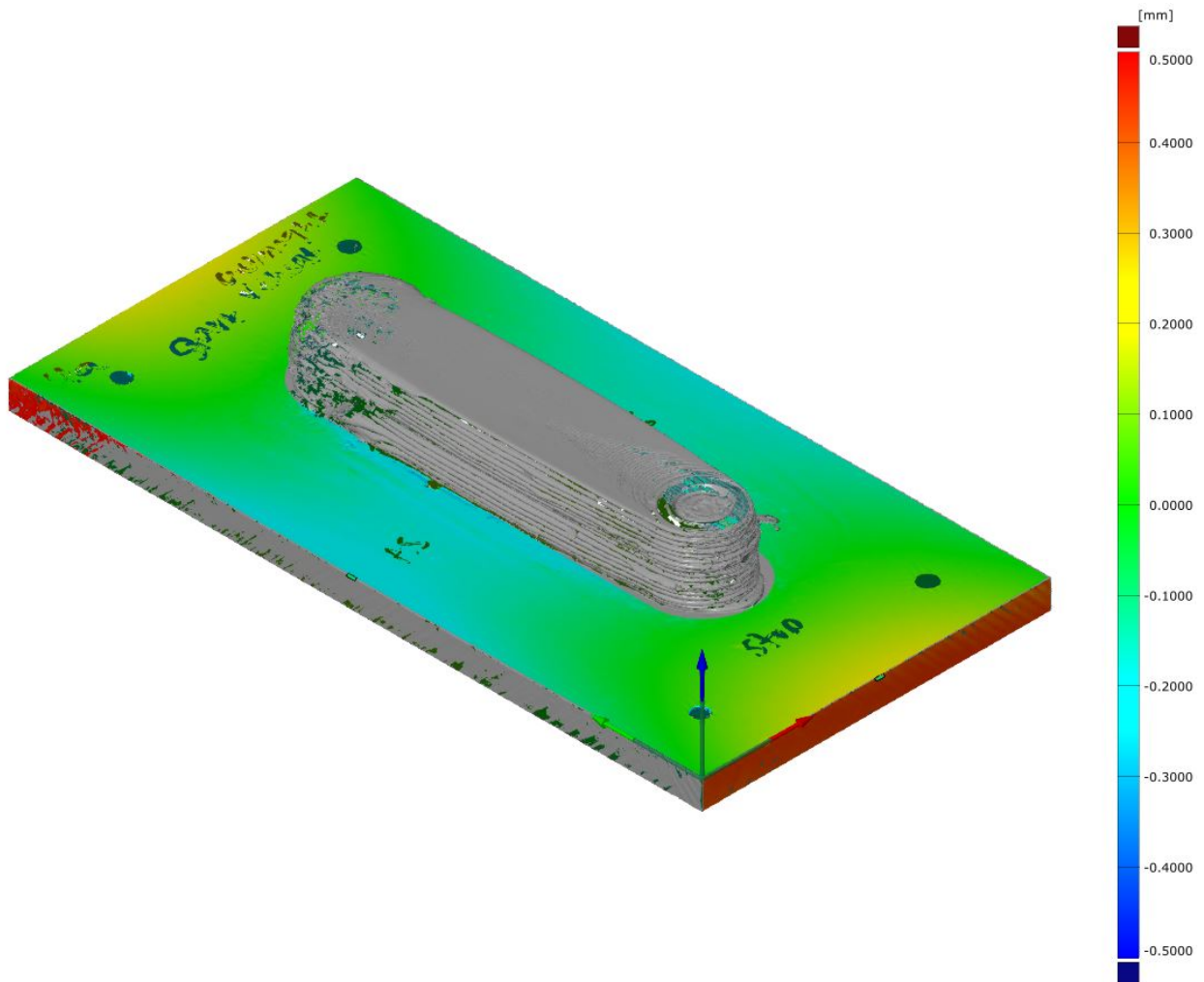


Figure A.14: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

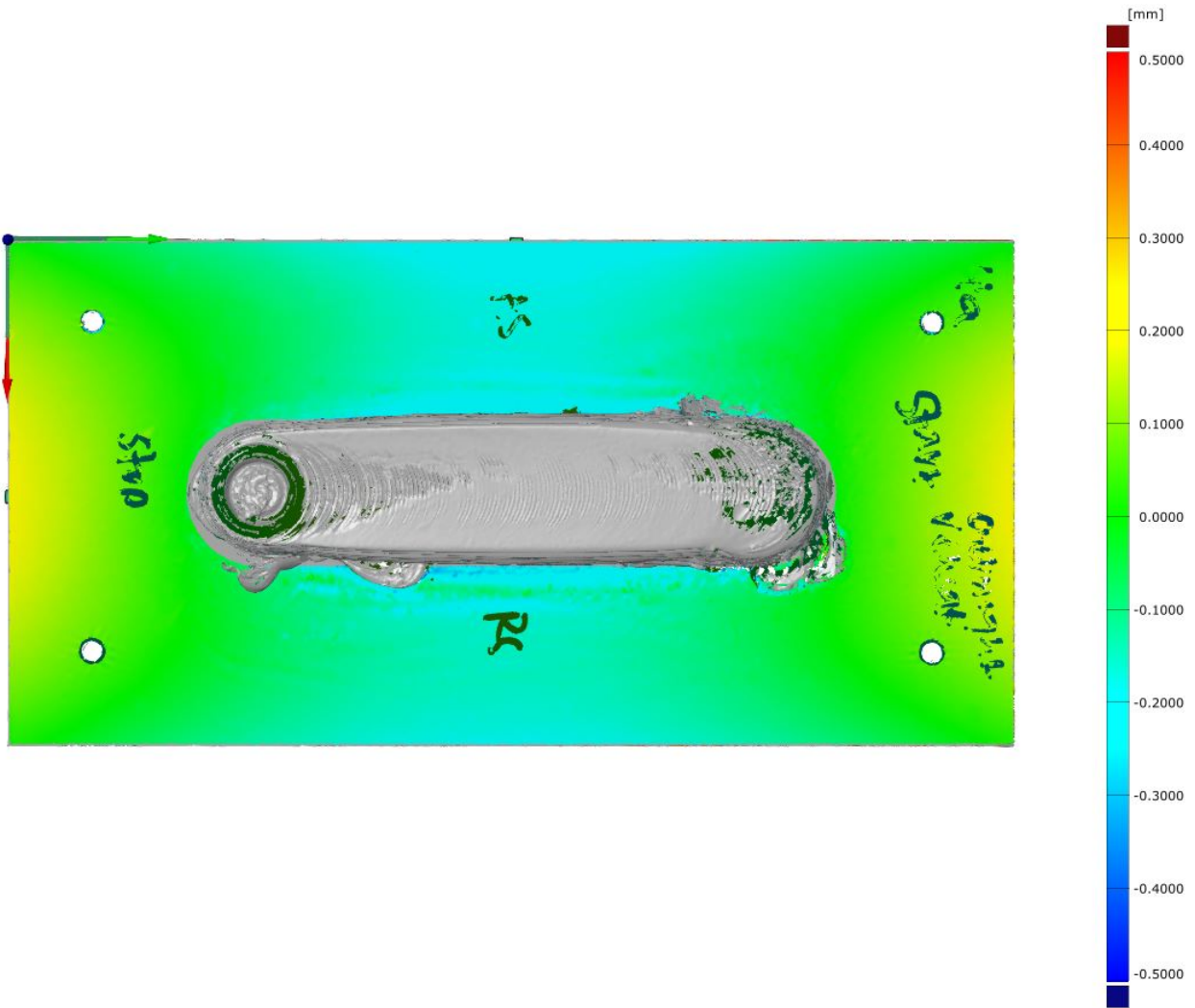


Figure A.15: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

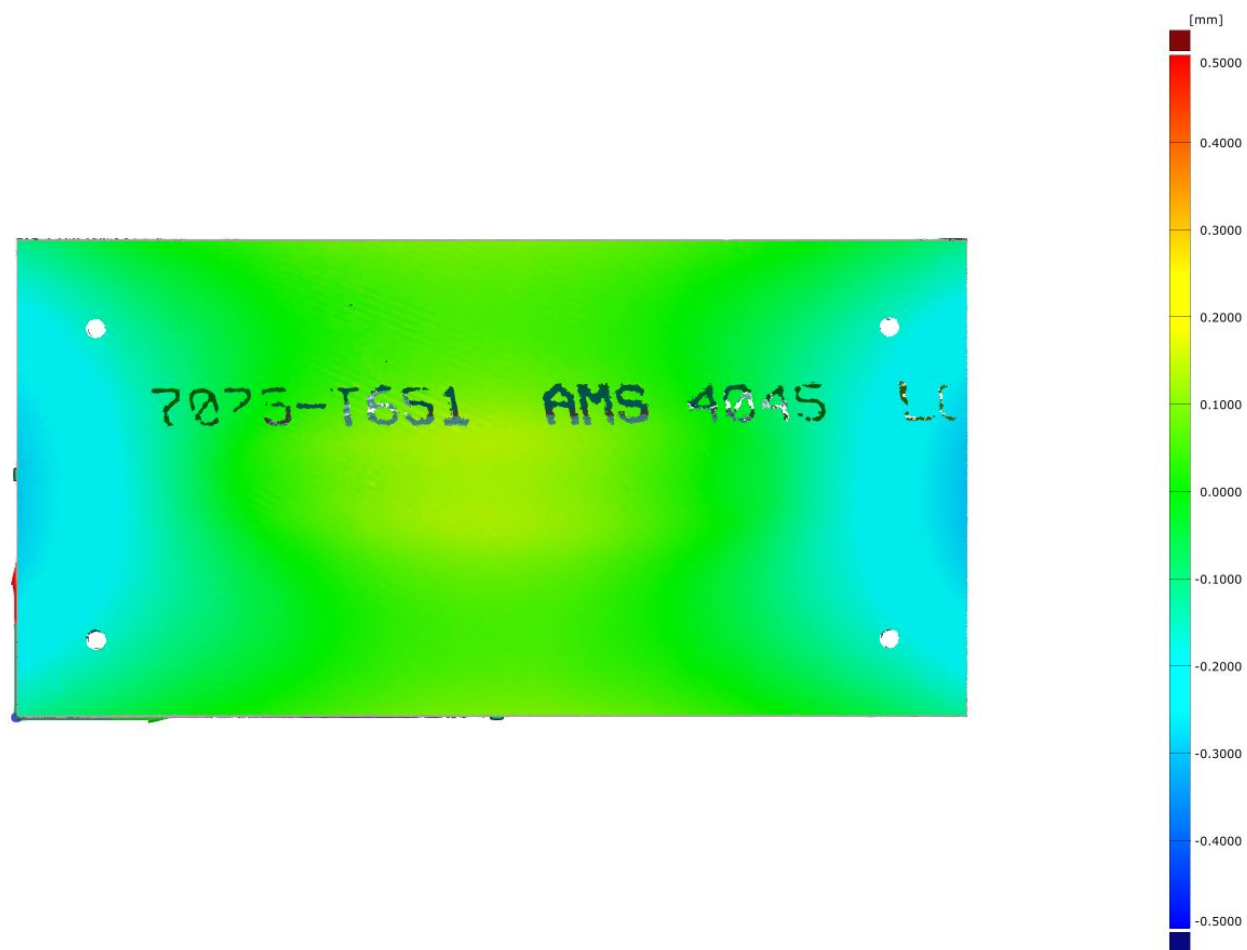


Figure A.16: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.3.4 Part b

A.3.5 Sample Photos

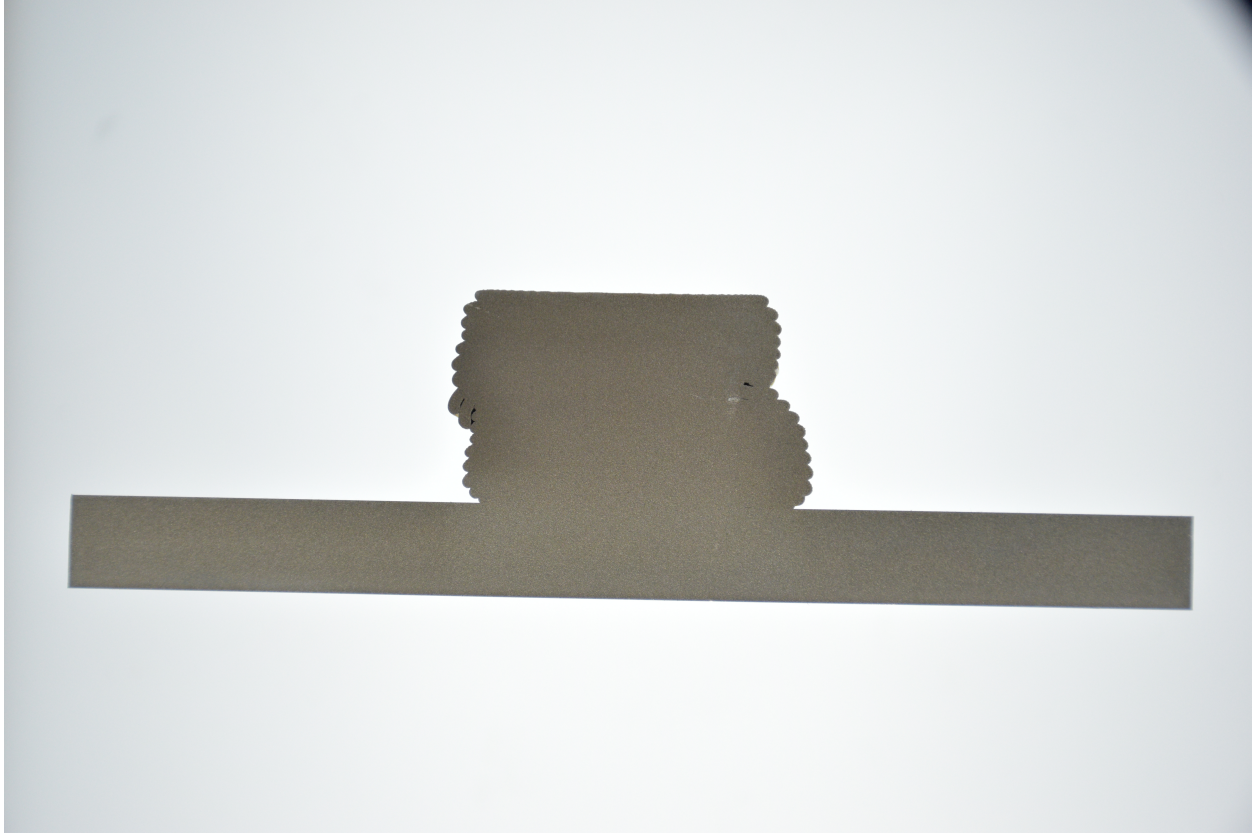


Figure A.17: Photo of sample 4b after cutting for contour measurement.

A.3.6 3D Scanning Data

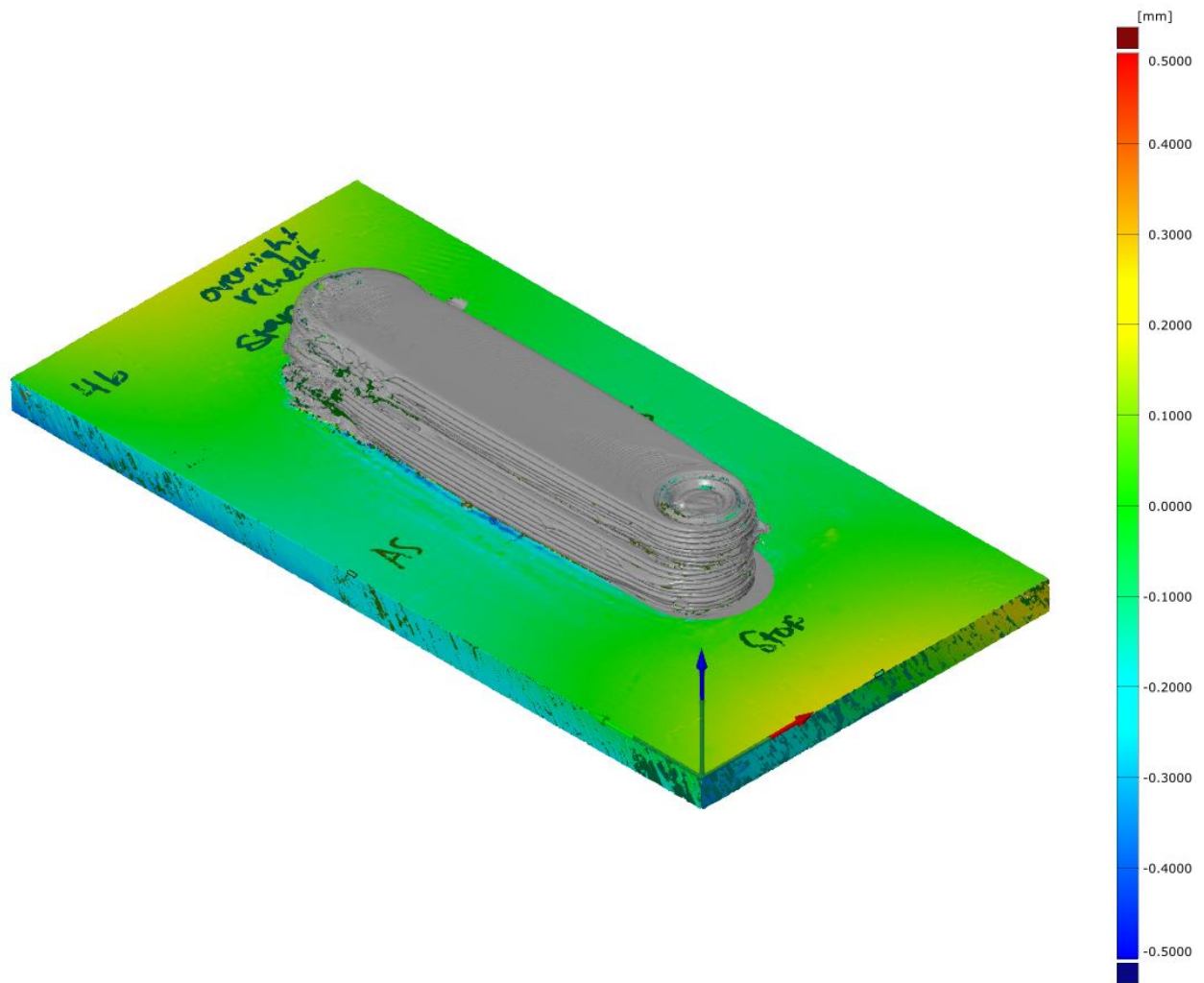


Figure A.18: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

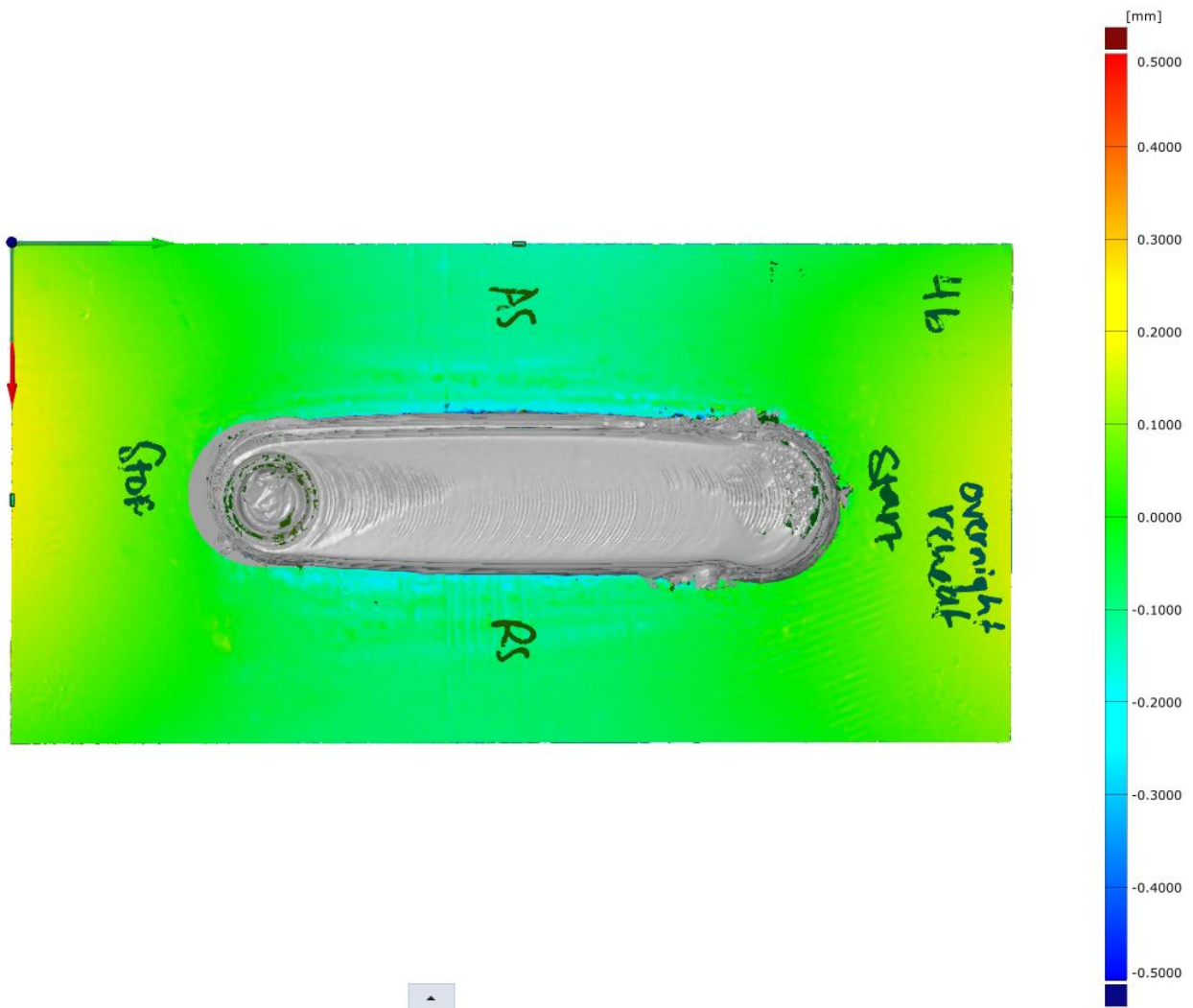


Figure A.19: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

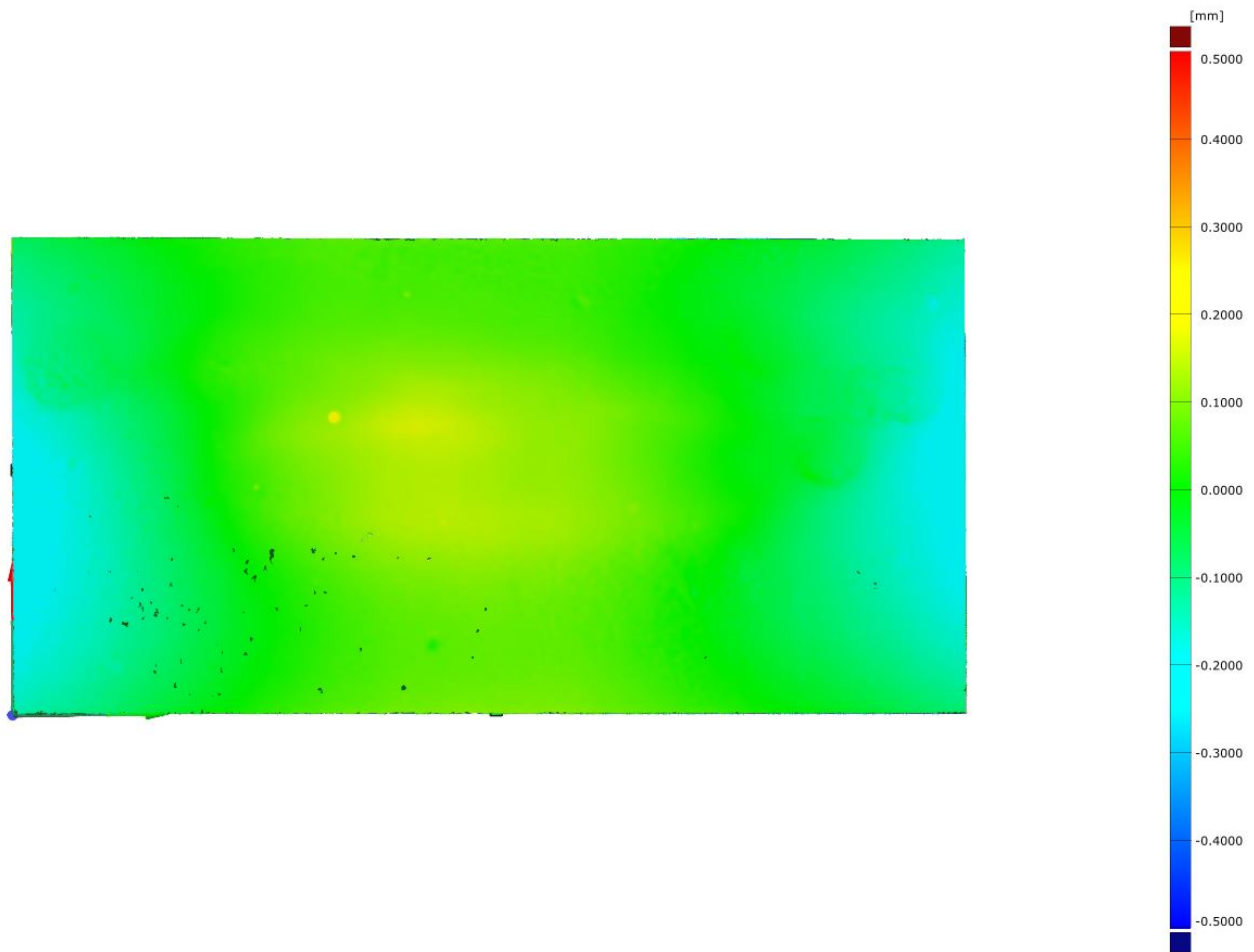


Figure A.20: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.4 100 hr Pause, no Reheat Data

A.4.1 Part a

A.4.2 Sample Photos



Figure A.21: Photo of sample 2a after cutting for contour measurement.

A.4.3 3D Scanning Data

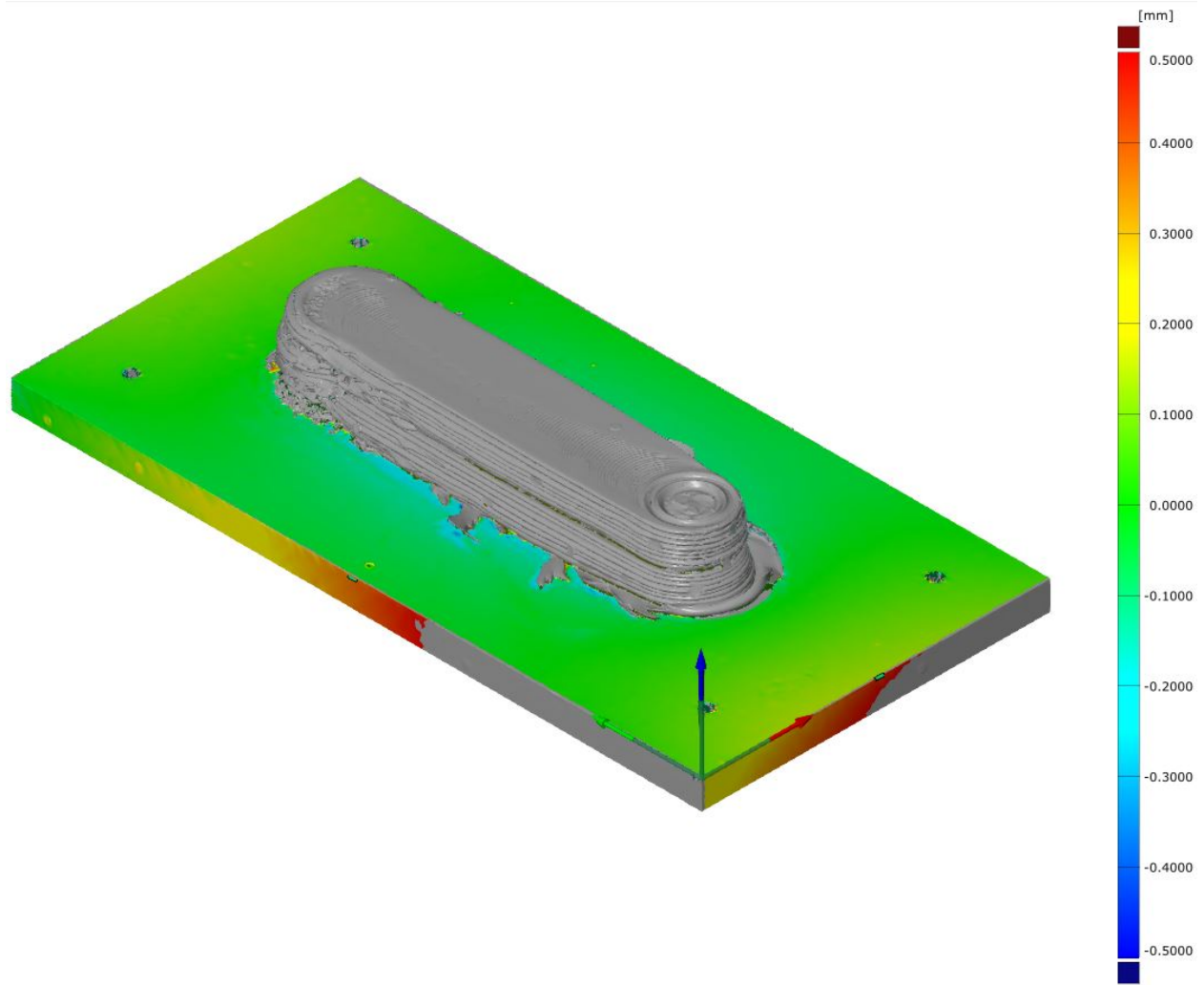


Figure A.22: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

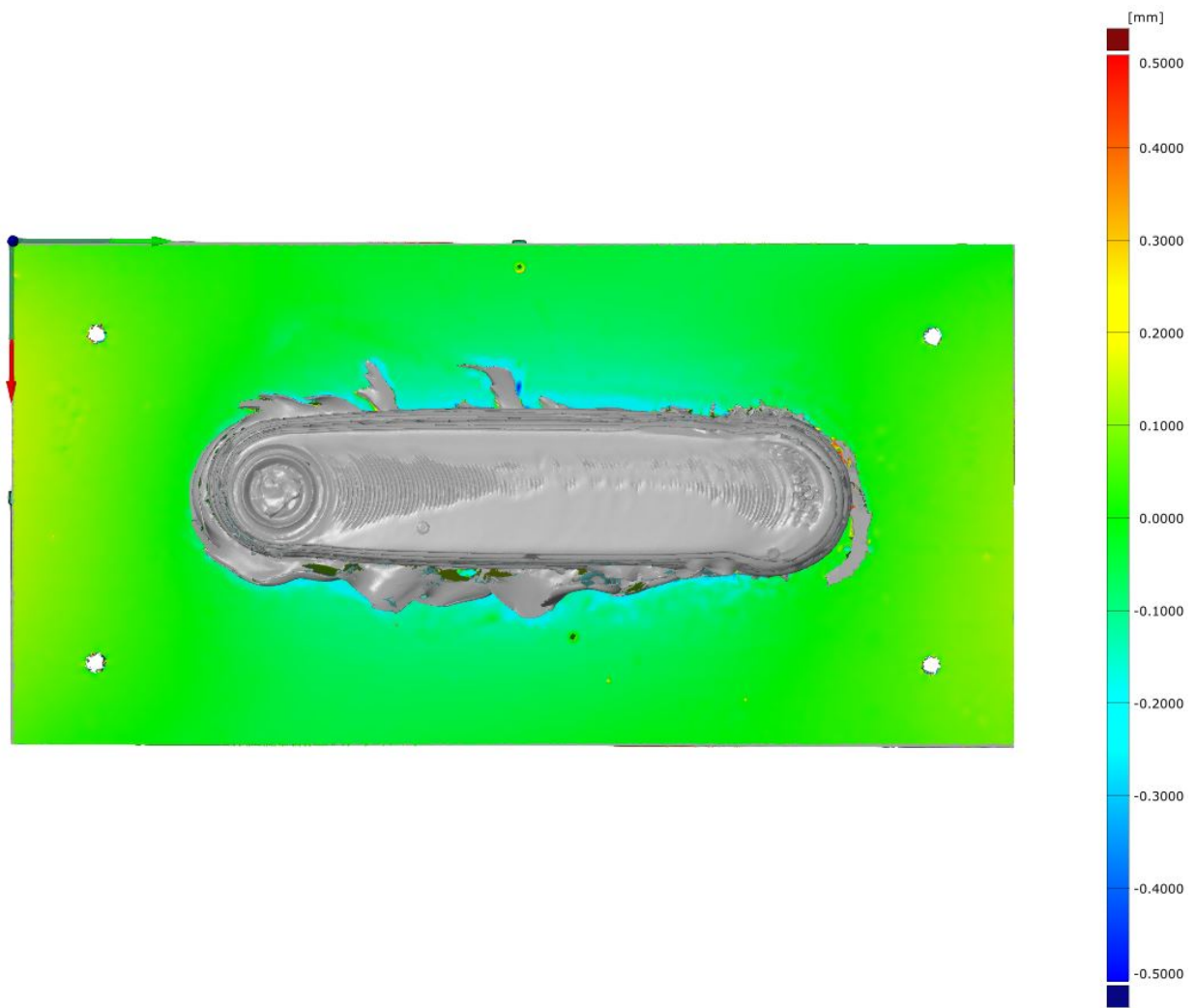


Figure A.23: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

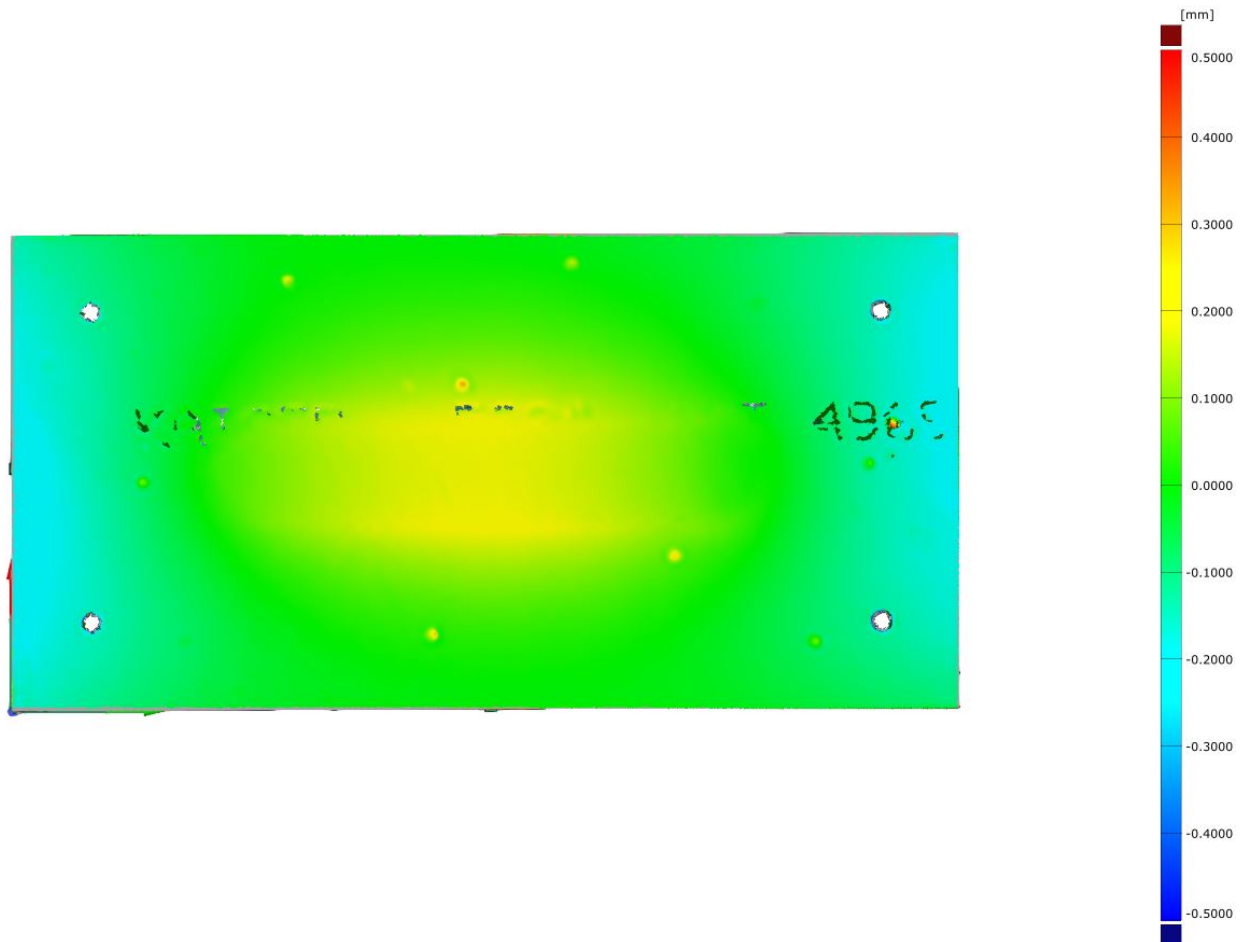


Figure A.24: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.4.4 Part b

A.4.5 Sample Photos

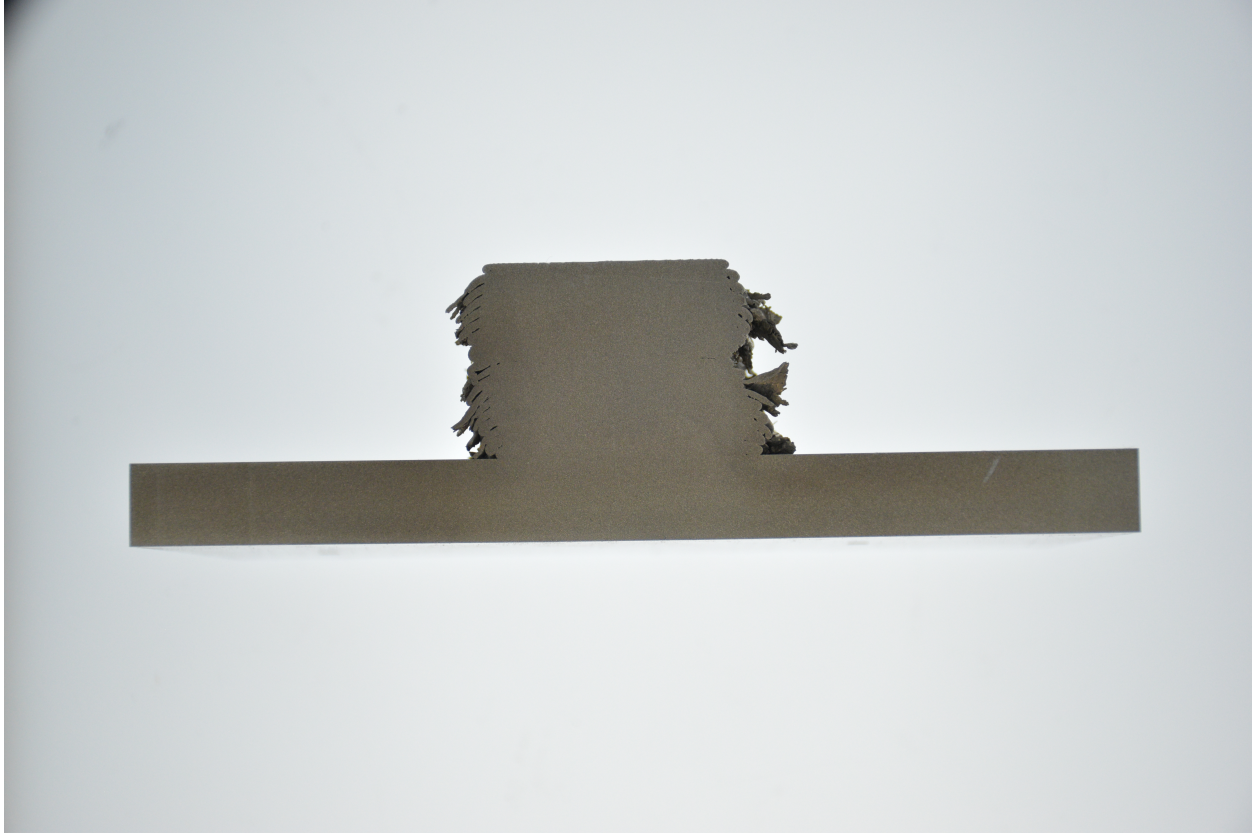


Figure A.25: Photo of sample 2b after cutting for contour measurement.

A.4.6 3D Scanning Data

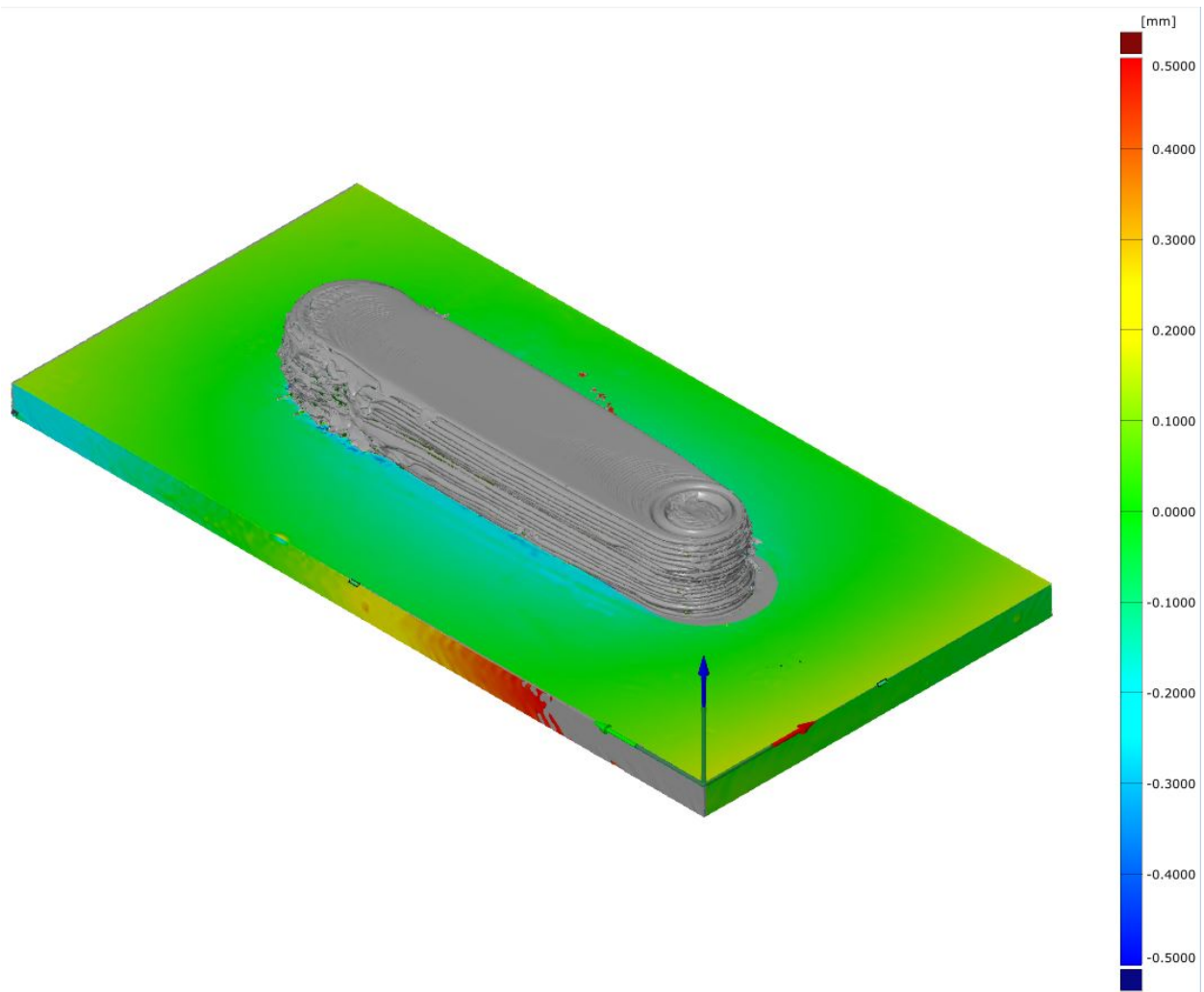


Figure A.26: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

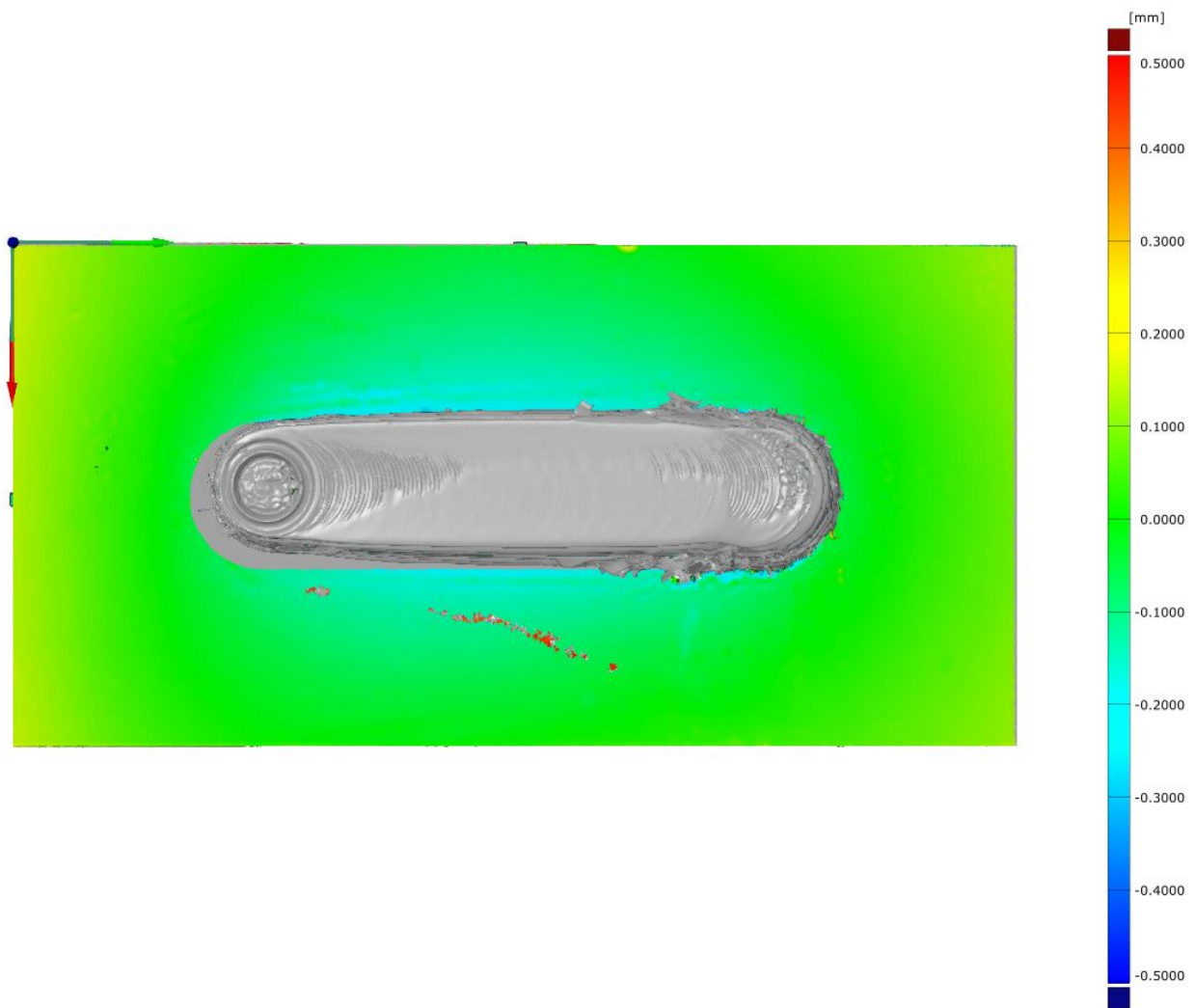


Figure A.27: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

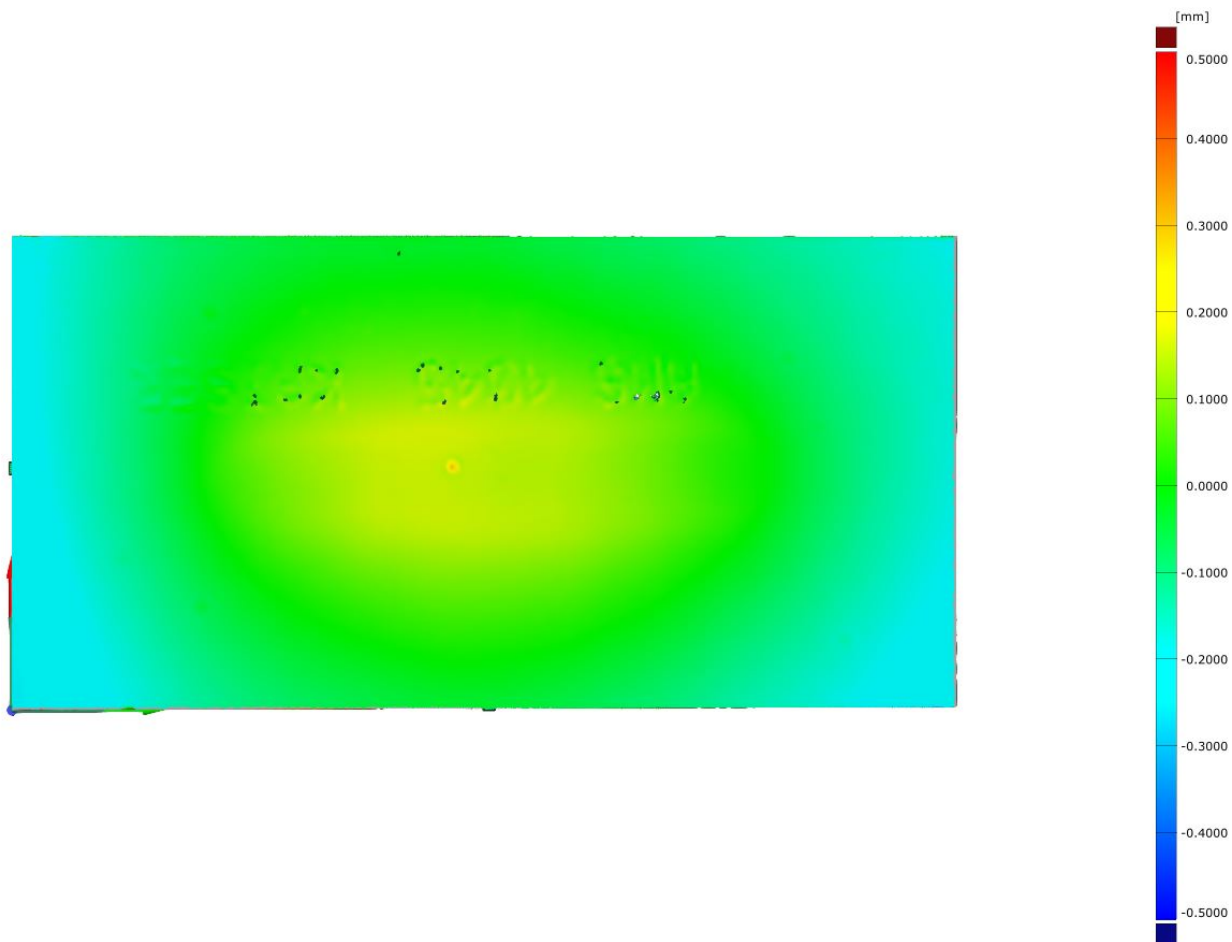


Figure A.28: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.5 100 hr Pause, Reheat Data

A.5.1 Part a

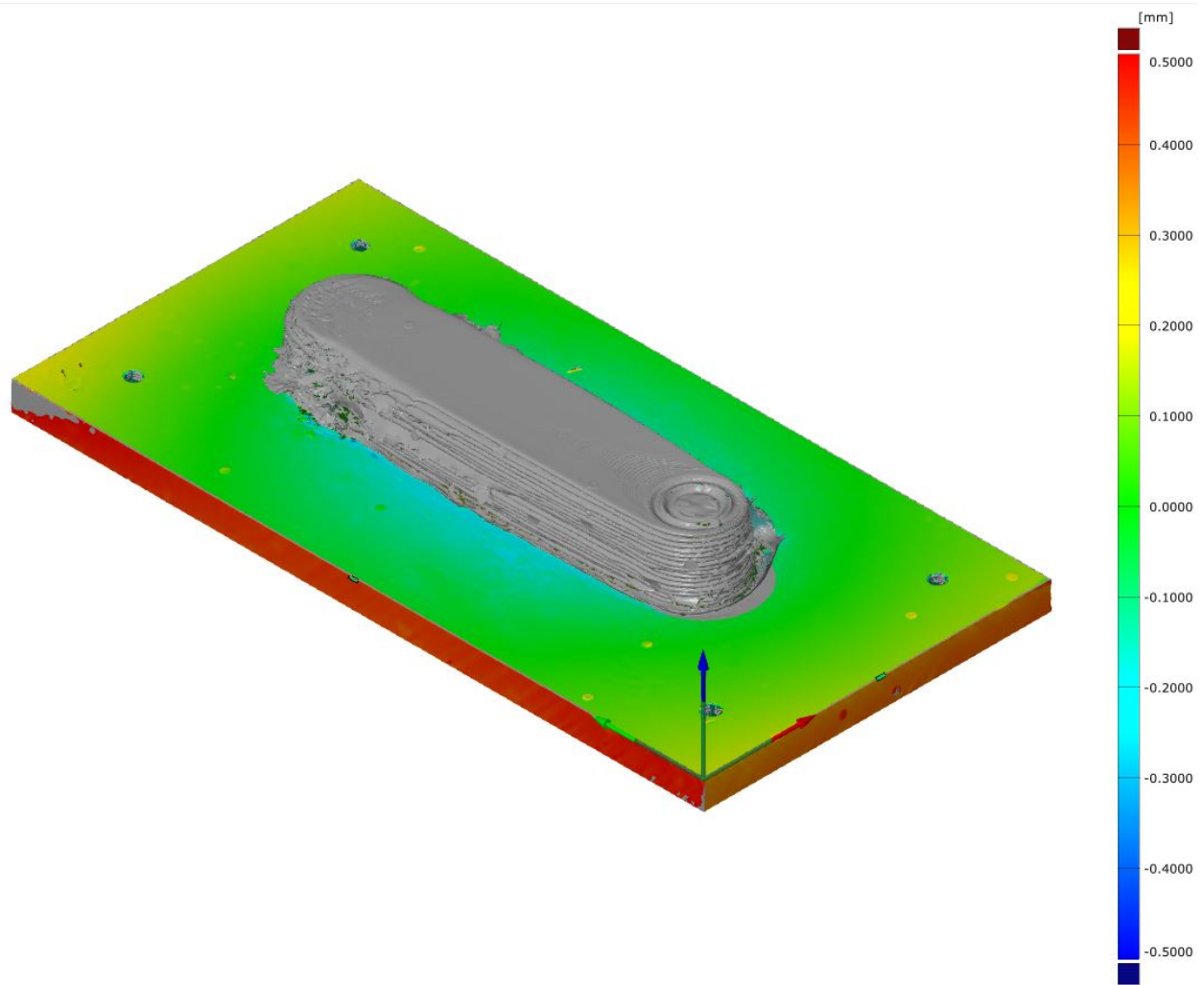


Figure A.29: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

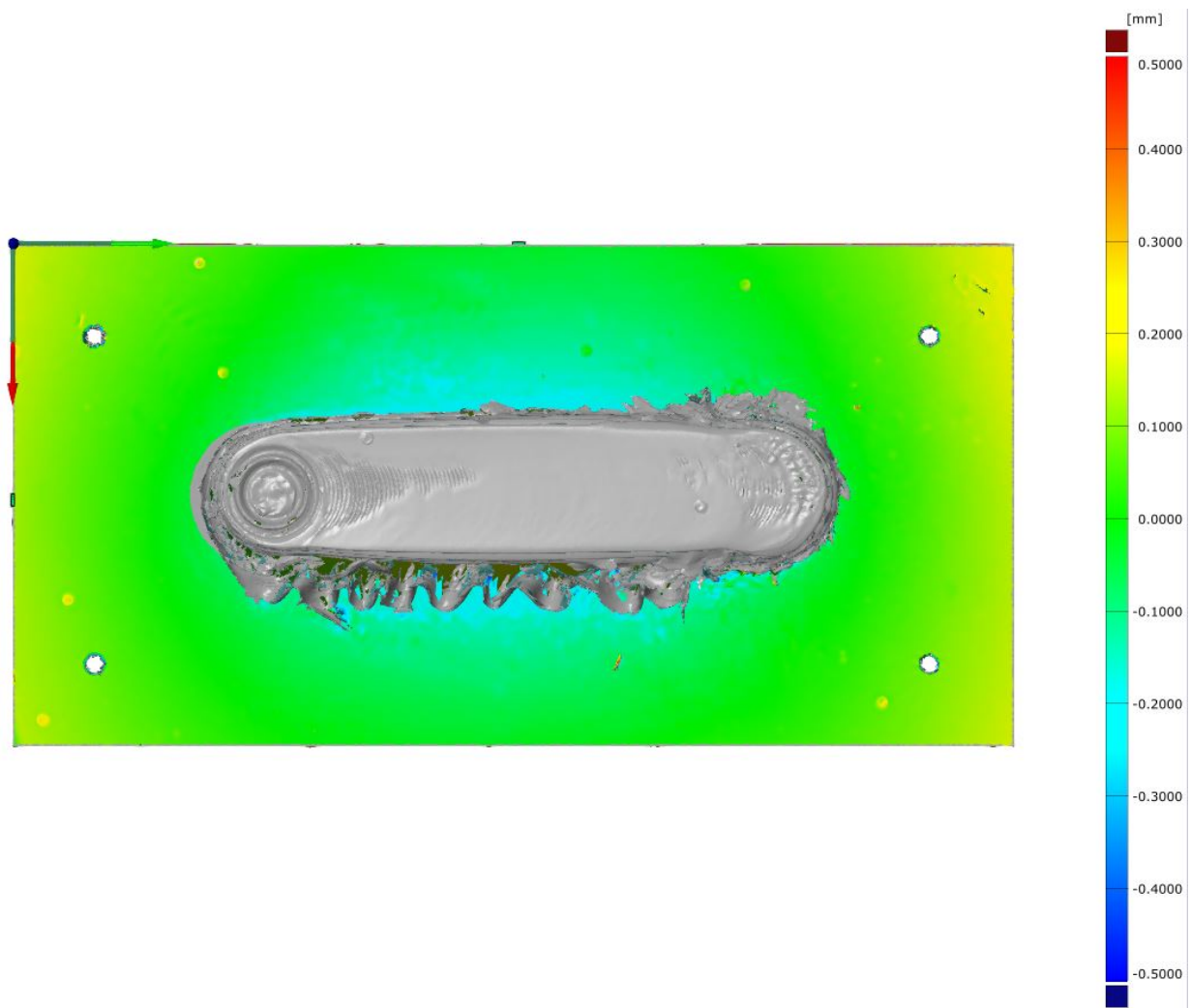


Figure A.30: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

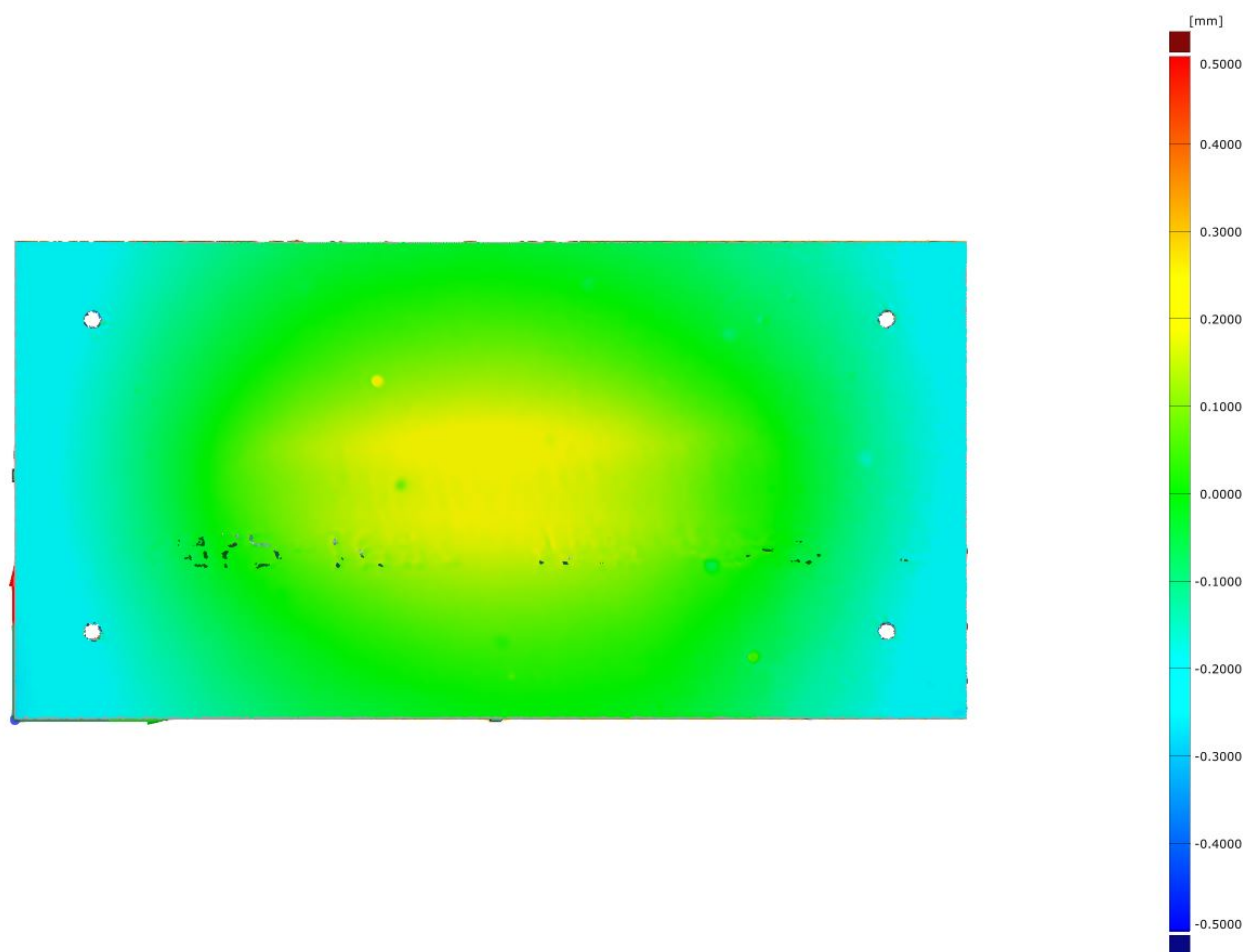


Figure A.31: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

A.5.2 Part b

A.5.3 Sample Photos



Figure A.32: Photo of sample 3b after cutting for contour measurement.

A.5.4 3D Scanning Data

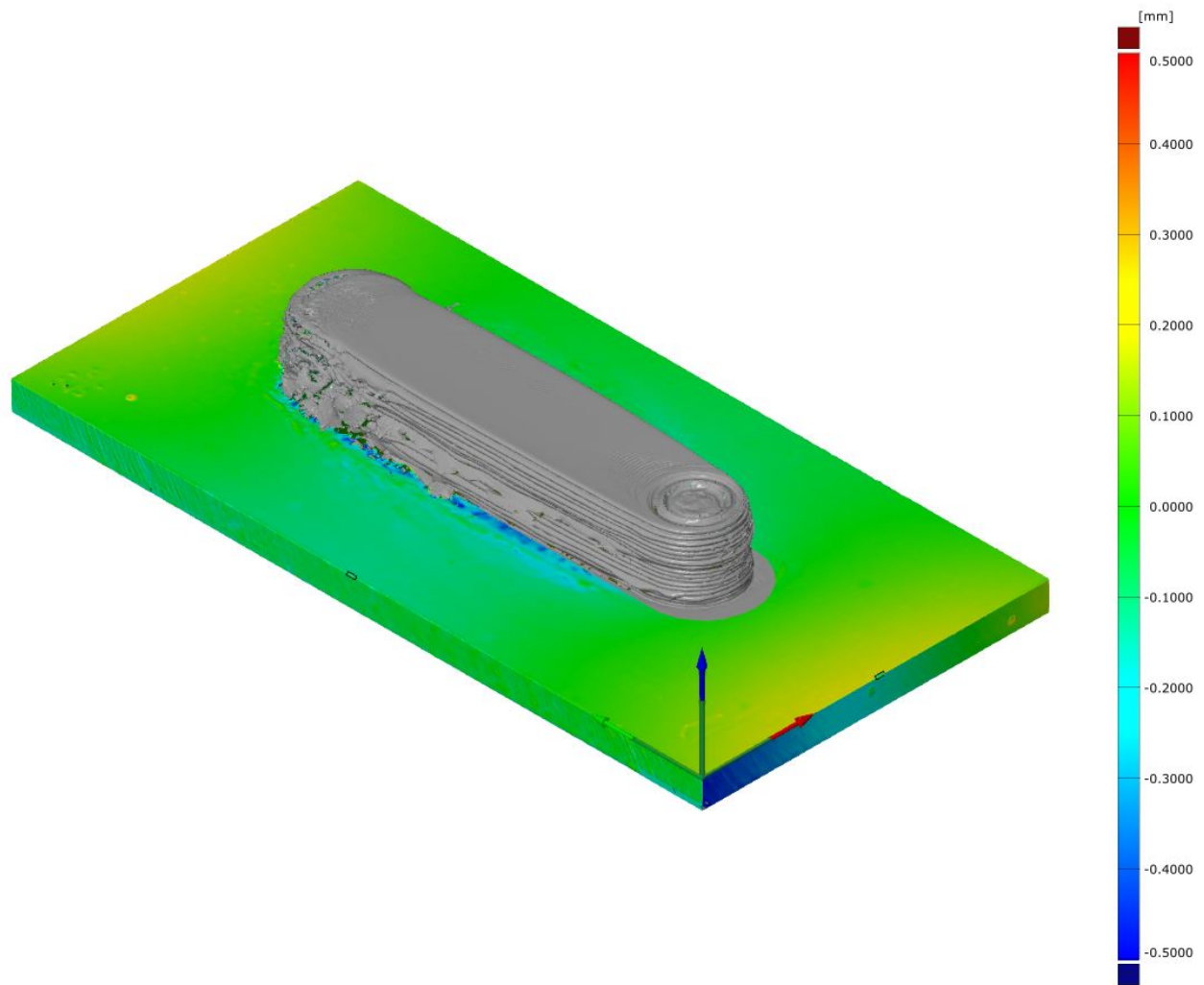


Figure A.33: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate.

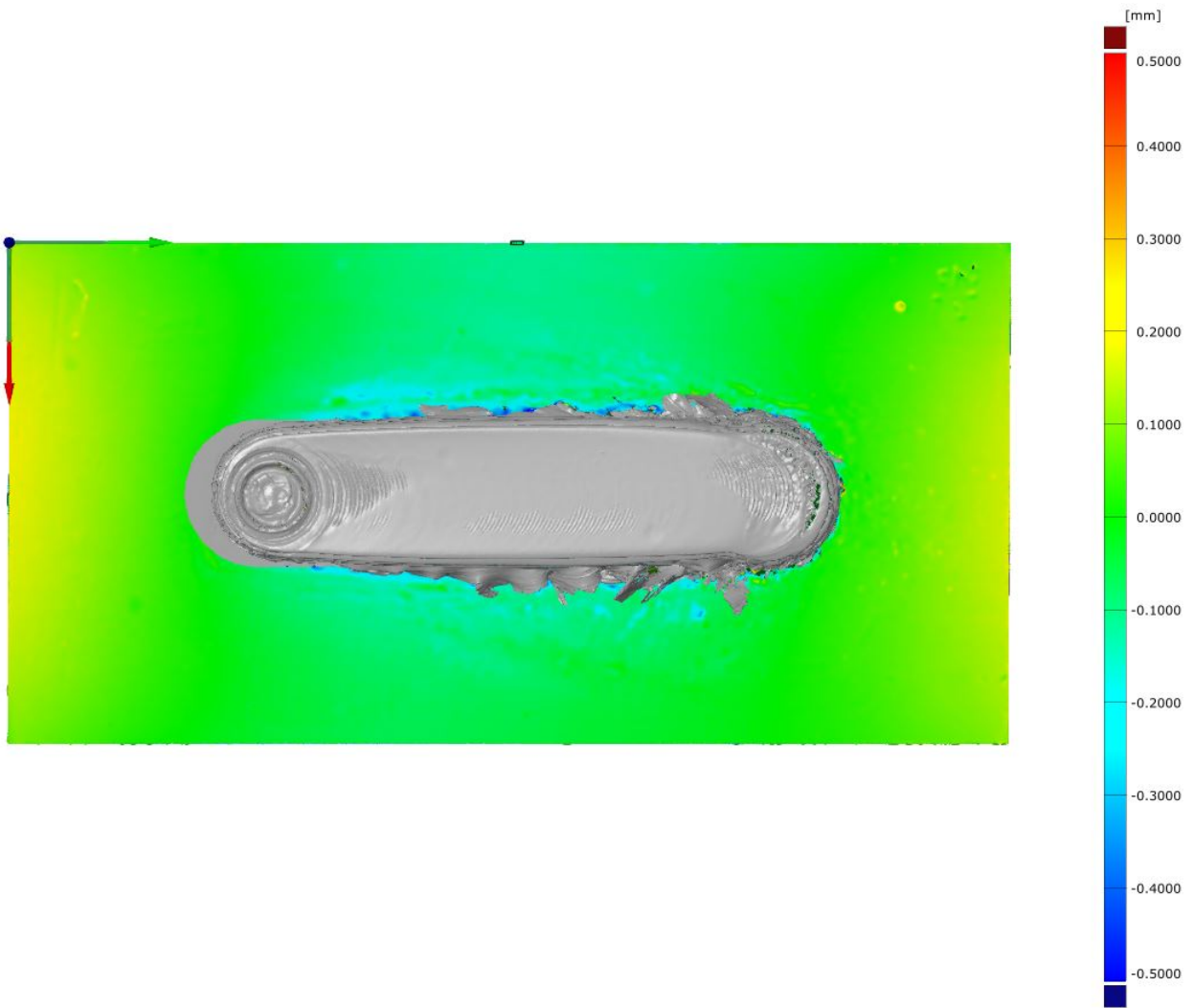


Figure A.34: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the top view.

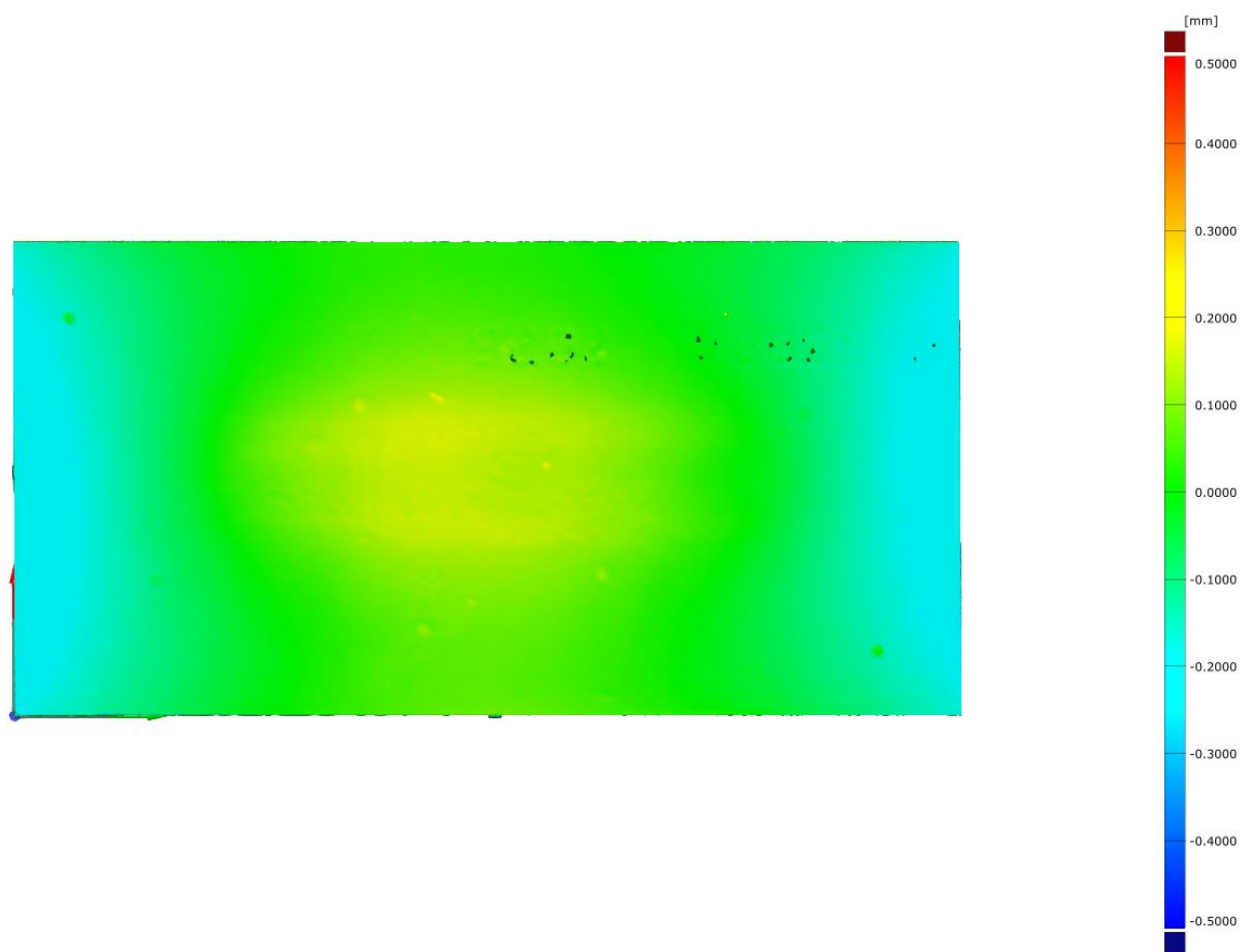


Figure A.35: Screen capture from ZEISS GOM 3d scan comparing the nominal CAD value of the base plate to the deposited plate from the base view.

Vita

Lauren M. Miller (née Van Beek) was born on August 21st, 1994 in Arlington Heights, Illinois. She graduated from Monticello High School (Minnesota) in 2012 and received her Bachelor of Science in Mechanical Engineering from the University of St. Thomas, St. Paul, Minnesota in May 2016. Following her undergraduate studies, Lauren worked in the metrology and manufacturing industries before starting graduate school at the University of Tennessee, Knoxville, in August 2020. Her dissertation is focused on Additive Friction Stir Deposition of aluminum 7075. Outside of work, Lauren is an active member of the Graduate Society of Women Engineers at UTK, where she has previously served as events chair and vice president.