Anisotropic Mechanical Behaviors and Microstructural Evolution of Thin-Walled Additively Manufactured Metals

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**Anisotropic mechanical behaviors and
microstructural evolution of thin-walled additively
manufactured metals**

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Abstract

Additive manufacturing (AM), also known as 3D printing, is a concept and method of a manufacturing process that builds a three-dimensional object layer-by-layer. Opposite to the conventional subtractive manufacturing, it conquers various limitations on component design freedom and raises interest in various fields, including aerospace, automotive and medical applications. This thesis studies the mechanical behavior of thin-walled component manufactured by a common AM technique, laser powder bed fusion (LPBF). The studied material is Hastelloy X, which is a Ni-based superalloy, and it is in connection to a component repair application in gas turbines. The influence of microstructure on the deformation mechanisms at elevated temperatures is systematically investigated. This study aims for a fundamental and universal study that can apply to different material grades with FCC crystallographic structure.

It is common to find elongated grain and subgrain structure caused by the directional laser energy input in the LPBF process, which is related to the different printing parameters and brands of equipment. This thesis will start with the study of scan rotation effect on stainless steel 316L in an EOS M290 equipment. The statistic texture analysis by using neutron diffraction reveals a clear transition when different level of scan rotation is applied. Scan rotation of 67° is a standard printing parameter with intention to lower anisotropy, yet, the elongated grain and cell structure is still found in the as-built microstructure. Therefore, the anisotropic mechanical behavior study is carried out on the sample printed with scan rotation of 67° in this thesis.

Thin-walled effects in LPBF are investigated by studying a group of plate-like HX specimens, with different nominal thicknesses from 4mm down to 1mm, and a reference group of rod-like sample with a diameter of 18mm. A texture similar to Goss texture is found in rod-like sample, and it becomes $\langle 011 \rangle // \text{BD}$ fiber texture in the 4mm specimen, then it turns to be $\langle 001 \rangle$ fiber texture along the transverse direction (TD) in the 1mm specimen. Tensile tests with the strain rate of 10^{-3} s^{-1} have been applied to the plate-like specimens from room temperature up to

700 °C. A degradation of strength is shown when the sample becomes thinner, which is assumed to be due to the overestimated load bearing cross-section since the as-built surface is rough. A cross-section calibration method is proposed by reducing the surface roughness, and a selection of proper roughness parameters is demonstrated with the consideration of the calculated Taylor's factor and the residual stress. The large thermal gradient during the LPBF process induces high dislocation density and strengthens the material, hence, the LPBF HX exhibits better yield strength than conventionally manufactured, wrought HX, but the work hardening capacity and ductility are sacrificed at the same time.

Two types of loading condition reveal the anisotropic mechanical behavior, where the vertical and horizontal tests refer to the loading direction being on the BD and TD respectively. The vertical tests exhibit lower strength but better ductility that is related to the larger lattice rotation observed from the samples with different deformation level. Meanwhile, the elongated grain structure and grain boundary embrittlement are responsible for the low horizontal ductility. A ductile to brittle transition is traced at 700 °C, so a further study with two different slow strain rates, 10^{-5} s^{-1} and 10^{-6} s^{-1} , are carried out at 700 °C. Creep damage is shown in the slow strain rates testing. Deformation twinning is found only in the vertical tests where it forms mostly in the twin favorable $\langle 111 \rangle$ oriented grain along the LD. The large lattice rotation and the deformation twinning make the vertical ductility remain high level under the slow strain rates. The slow strain rate tensile testing lightens the understanding of creep behavior in LPBF Ni-based superalloys.

In summary, this thesis uncovers the tensile behavior of LPBF HX with different variations, including geometry-dependence, temperature-dependence, crystallographic texture-dependence and strain rate-dependence. The generated knowledge will be beneficial to the future study of different mechanical behavior such as fatigue and creep, and it will also enable a more robust design for LPBF applications.

Keyword: hot tensile testing, slow strain rate tensile testing, creep, anisotropy, crystallographic texture evolution, deformation twinning

Populärvetenskaplig sammanfattning

Additiv tillverkning, eller 3D-utskrift, är tillverkningsmetoder där man skapar ett tredimensionellt objekt genom att tillföra material lager för lager. Till skillnad från konventionella avverkande tillverkningsmetoder elimineras många geometriska begränsningar vilket ger större designfrihet och metoderna har därför väckt stort intresse inom en rad olika områden, inklusive flyg-, fordons- och medicinska tillämpningar. I denna avhandling studeras mekaniska egenskaper hos tunnväggiga komponenter tillverkade med en vanligt förekommande laserbaserad pulverbäddsteknik, laser powder bed fusion (LPBF). Det studerade materialet är Hastelloy X, en Ni-baserad superlegering som är vanligt förekommande för både nytillverkning och reparation av komponenter för gasturbiner. Inverkan av mikrostruktur på deformationsmekanismerna vid förhöjda temperaturer undersöks systematiskt. Detta arbete syftar till att ge grundläggande och generisk kunskap som kan tillämpas på olika materialtyper med en kubiskt tätpackad (FCC) kristallstruktur.

Det är vanligt att man hittar en utdragen kornstruktur orsakad av den riktade tillförseln av laserenergi i LPBF-processen, vilket kan relateras till olika processparametrar och kan variera mellan utrustningar från olika leverantörer. Denna avhandling inleds med studien av effekten av scanningsstrategi vid tillverkning av rostfritt stål 316L i en EOS M290-utrustning. En statistisk texturanalys med hjälp av neutrondiffraction påvisar en tydlig övergång mellan olika mikrostrukturer när olika scanningsstrategier tillämpas. En scanningsrotation på 67 mellan varje lager är en typisk standardinställning med avsikt att sänka anisotropin i materialet, dock finns den utdragna kornstrukturen oftast kvar. I denna avhandling studeras därför de anisotropa egenskaperna hos material tillverkade med 67 scanningsrotation.

Effekten av tunnväggiga strukturer i LPBF undersöks genom att studera en uppsättning platta HX-prover, med olika nominella tjocklekar från 4 mm ner till 1 mm, samt en referensgrupp med cylindriska prov med en diameter på 18 mm. Kristallografisk textur som liknar den av Goss-typ återfinns i de cylindriska

proverna vilket gradvis övergår från en fibertextur med $\langle 011 \rangle$ i byggriktningen för 4mm-proven till en fibertextur med $\langle 001 \rangle$ i tvärriktningen för 1mm-proven. Dragprovning med en töjningshastighet på 10^{-3} s^{-1} har utförts på de platta provstaverna från rumstemperatur upp till 700°C . En sänkning av styrkan uppvisas när proven blir tunnare, vilket kan antas bero på att det lastbärande tvärsnittet överskattas på grund av den grova ytan. En metod för tvärsnittskalibrering föreslås genom att kompensera för ytråheten, och valet av lämplig ytfinhetsparameter motiveras med hänsyn till den beräknade Taylor-faktorn och förekomsten av restspänningar. Den stora termiska gradienten som uppstår för LPBF-processen inducerar en hög dislokationstäthet vilket höjer materialets styrka och följaktligen uppvisar LPBF HX högre sträckgräns än konventionellt tillverkad, smidda HX, men förmågan till deformationshårdnande samt duktiliteten i materialet sänks samtidigt.

Tester utförda i två olika belastningsriktningar, vertikalt respektive horisontellt mot byggriktningen, demonstrerar det anisotropiska mekaniska beteendet. De vertikala testerna uppvisar lägre hållfasthet men bättre duktilitet vilket kan relateras till en större benägenhet för kristallstrukturen att rotera när deformationsgraden ökar. Samtidigt är den utdragna kronstrukturen ansvarig för den lägre duktiliteten för de horisontella proverna. En övergång från ett duktilt till ett mer sprött beteende noterades vid 700°C , och därför initierades ytterligare en studie där tester med två lägre töjningshastigheter, 10^{-5} s^{-1} och 10^{-6} s^{-1} , utfördes vid 700°C . Det kan noteras att krypskador återfinns i tester med en långsam deformationshastighet och deformationstvillingar uppstår endast i de vertikala provstaverna där det främst bildas tvillingar i korn orienterade med $\langle 111 \rangle$ riktningen längs belastningsriktningen. Den stora förmågan till rotation i kristallstrukturen och deformationstvillingarna bidrar till att den vertikala duktiliteten förblir hög även i testerna med en låg deformationshastighet. Testerna med en långsam draghastighet bidrar därför till en bättre förståelse av krypbeteendet i LPBF Ni-baserade superlegeringar.

Sammanfattningsvis så bidrar denna avhandling till bättre förståelse av de mekaniska egenskaperna hos LPBF HX i olika utföranden och förhållanden, inklusive geometriberoende, temperaturberoende, deformationshastighetsberoende samt inverkan av kristallografisk textur. Den genererade kunskapen kommer att vara till stor nytta vid fortsatta studier av olika mekaniska egenskaper som utmattning och kryp, samt bidrar till att möjliggöra en mer robust design för LPBF-tillämpningar.

Nyckelord: Varmdragprov, Dragprov med låg töjningshastighet, kryp, anisotropi, kristallografisk textur, deformationstvillingar

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A handwritten signature in black ink, appearing to read 'Hank Yu' in a cursive, stylized script.

Hank Yu
Linköping, October 2020

List of papers

The papers related to this Licentiate thesis are as follow:

Appended papers

- I. A. Leicht, C. H. Yu, V. Luzin, U. Klement, and E. Hryha, “Effect of scan rotation on the microstructure development and mechanical properties of 316L parts produced by laser powder bed fusion,” *Materials Characterization*, vol. 163, p. 110309, 2020.

In this paper paper, Cheng-Han Yu contributes the statistic texture analysis by using neutron diffraction. The main contributor is Alexander Leicht at Chalmers University, Göteborg, Sweden.

- II. C.-H. Yu, R. L. Peng, J.-E. Lundgren, and J. Moverare, “Anisotropic mechanical properties of thin-walled Hastelloy X manufactured by laser powder bed fusion,” *European Congress and Exhibition on Powder Metallurgy*, 2019.
- III. C.-H. Yu, R. L. Peng, V. Luzin, M. Sprengel, M. Calmunger, J.-E. Lundgren, H. Brodin, A. Kromm, J. Moverare, “Thin-wall effect and anisotropic deformation mechanisms of an additively manufactured Ni-based superalloy,” submitted manuscript.
- IV. C.-H. Yu, R. L. Peng, H. Brodin, and J. Moverare, “Anisotropic deformation and fracture mechanisms of an additively manufactured Ni-based superalloy,” in *TMS Superalloys 2020*.

In the appended papers II-IV, Cheng-Han Yu is the main contributor, including mechanical testing, microstructure analysis and manuscript writing, under the supervision of Johan Moverare and Ru Lin Peng. The studied materials are provided by Siemens Energy AB, Finspång, Sweden. The texture

measurement by using neutron diffraction is contributed by Vladimir Luzin at Australian Nuclear Science and Technology Organization (ANSTO), Sydney, Australia. The residual stress measurement is contributed by Maximilian Sprengel and Arne Kromm at Bundesanstalt für Materialforschung und -prüfung (BAM), Berlin, Germany. The mechanical testing of wrought Hastelloy X is contributed by Mattias Calmunger at Linköping University.

Abbreviations

Additive Manufacturing	AM
Building Direction	BD
Computer Aided Design	CAD
Critical Resolved Shear Stress	CRSS
Dynamic Strain Aging	DSA
Electical Discharge Machining	EDM
Electron Backscatter Diffraction	EBSD
Electron Beam Melting	EBM
Face-Centered Cubic	FCC
Grain growth Direction	GD
Hastelloy X	HX
Hexagonal Close-Packed	HCP
Inverse Pole Figure	IPF
Laser Powder Bed Fusion	LPBF
Light Optical Microscope	LOM
Loading Direction	LD
Orientation Distribution Function	ODF
Pole Figure	PF
Powder Bed Fusion	PBF
Scanning Electron Microscope	SEM
Slow Strain Rate Testing	SSRT
Transmission Electron Microscope	TEM
Transverse Direction	TD
Time-Temperature-Transformation	TTT
Topological Close-Packed	TCP
Ultimate Tensile Strength	UTS
Wall Direction	WD

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Part I

Background and Theory

1.1 Background

Additive Manufacturing (AM), also known as 3D printing, has recently raised high interest in various applications in virtue of potential advantages such as component design freedom and manufacturing energy efficiency. In an AM process, the component is built layer-by-layer guided by 3D computer aided design (CAD) model, which breaks the geometrical limitation in the conventional manufacturing, and it enables a complex geometry to be fabricated in one-step. The concept of AM was brought up in the 70's and executed in the 80's, and it was started with the Stereolithography process on polymer materials [1]. The AM in metallic materials began with the sintering or melting by laser-based techniques from 90's, and after a long period of development, various processes became relatively applicable in 10's. For decades it has been called "rapid prototyping", but thanks to the successful development of AM techniques in the recent years, AM components become functional and able to be used as critical parts in different applications, such as aerospace, automotive and medical surgery [2]. The concept of mass production has dominated over decades, but the idea of mass customization gradually takes the place of it in the recent industrialization revolution. Without a doubt, AM will play as a "game changer" in mass customization, and it is expected to prevail in a wide variety of applications. However, the functionality needs to pass a complete examination and fulfill standard requirements, therefore, this thesis systematically investigates the mechanical behavior and deformation mechanism of LPBF materials with the focus on the anisotropy and thin-wall effects.

This PhD study is within the framework of CAM2, a competence center for additive manufacturing of metals funded by Vinnova, and it includes seven different research areas. This work is under the research area of material behavior and

design of AM components, and a specific investigation has been carried out on the anisotropic material properties and thin-walled structure of a Ni-based superalloy for high temperature applications. An application connected to this study is the burner tip repair in gas turbines [3], in which the tip can be built directly on the remained nozzle after the worn part being removed instead of manufacturing the entire component. The manufacturing and machining processes of Ni-based superalloy require high energy consumption, so an entire component remanufacturing is definitely a waste of energy and material. The AM process produces the part directly with one step, and the strength of energy efficiency makes AM a great potential for the development of sustainable environment.

1.2 Aims and research questions

A general aim of this thesis is to generate knowledge of mechanical behavior of AM materials and bring AM a step further from prototyping toward functional component. In addition, this thesis aims for a fundamental and universal study that applies to different material grades with a Face-Centered Cubic (FCC) crystallographic structure and manufactured by Laser powder bed fusion (LPBF). A detailed plan is to uncover the tensile behaviors of Hastelloy X, a Ni-based superalloy, with different variations, including geometry-dependence, temperature-dependence, strain-rate dependence and crystallographic texture-dependence. To investigate the mechanisms behind the tensile behaviors, microstructure study and statistic texture analysis are carried on the as-built and deformed materials.

The following research questions will first address the commonly found features in AM materials, and then find the mechanisms for the material behaviors.

1. What are the typical microstructure and anisotropic mechanical properties in LPBF materials?
2. How does the microstructure and mechanical behavior evolve when it comes to thin-walled structure in LPBF?
3. How does the mechanical behavior of LPBF FCC materials respond at elevated temperatures and different strain rates?
4. Based on the questions above, what are the mechanisms for anisotropic mechanical behaviors and how does it relate to elongated grain and subgrain structure?

1.3 Outline

The thesis is structured into two parts. **Part I** is the *Background and Theory*, and it consists of seven chapters. It starts with a brief introduction of the PhD research project, followed by the addressed research questions that structure this Licentiate thesis. A general presentation of additive manufacturing from the points of view of process, limited material applicability, microstructure and thin-walled structure

is brought up. Right after that, the mechanical behaviors and several important theories of deformation mechanism are given. Then, the highlights of the related papers are summarized, responding to the research questions mentioned in the beginning. Finally, the conclusions that answer the research question are given in four themes. **Part II** is the *Publications*, which presents the main part of the PhD research so far.

2.1 Additive manufacturing processes

According to the standard, ISO/ASTM52900-15, the AM processes are defined in seven categories,

- Vat photopolymerization
- Material jetting
- Binder jetting
- Powder bed fusion
- Material extrusion
- Directed energy deposition
- Sheet lamination

Among above, powder bed fusion (PBF) is a commonly used technique for metallic materials, and there are two major categories of PBF depending on different input energy, laser source (laser powder bed fusion, LPBF) or electron beam source (electron beam melting, EBM). The two techniques share similarity of working principles that the input energy melts the powders at the top surface of the powder bed in the commanded area and the component is built layer by layer, but the detailed parameters are different. For example, the used powder in LPBF is smaller than EBM, the vacuum chamber and heating is need in EBM, and the support structure is required in LPBF [4].

Figure 2.1. Schematic illustration of laser powder bed fusion.

This study will focus on LPBF and a simplified schematic illustration is demonstrated in Figure 2.1. It is a continuously circular process where the laser source melts along the designed pattern on the top layer of the powder bed, then the base plate lowers down the entire powder bed, and the recoater brings a new layer of powder from the powder delivery to the powder bed for the next round of laser melting. For each layer, the laser moves linearly and parallelly along the same direction, melting the powder in the region commanded by the designed pattern. The designed pattern can be briefly categorized to two types, hatching (the region of main body) and contouring (the linear boundary), and the input energy density is different between each other. Scan rotation corresponds to the directional change of the moving laser between consecutive layers. Scan rotation of 67° is a commonly used parameter in the standard process, which means that the laser moving direction will continuously shift 67° after each layer and the center of powder bed is approximately the rotation center. During the printing process, an inert gas flow is applied in the direction perpendicular to the recoater moving direction, where the proper parameters can optimize the interaction between the ambient environment and the melt pool [5], and it further improves the quality of the component such as lower amount of porosity, better surface roughness and mechanical properties [6, 7]. The support structure in Figure 1 is only placed between the component and base plate, but in a real case the support structure is often designed beneath the fragile part, for instance, the overhang. The support structure is for the prevention of distortion during the printing process, the position and geometry of the support structure can be suggested by the software and decided by the designer [8, 9].

Various process parameters and scanning strategies can be tuned for the desired material properties, for example, laser moving speed, hatch distance, layer thickness and scan rotation [10–14]. The influence of scan rotation for texture control will be discussed later on in this chapter.

2.2 Materials

The material for this study is Hastelloy X (HX), a Ni-Cr-Fe-Mo alloy, as a material grade of Ni-based superalloy it exhibits excellent corrosion resistance and mechanical properties for high temperature applications [15]. HX exists as a single phase of austenitic γ matrix at as-built state, which is a FCC crystallographic structure, and the nominal chemical composition is listed in Table 2.1.

Ni	Cr	Fe	Mo	W	Co	C	Si
Bal.	20.5-23	17-20	8-10	0.2-1	0.5-2.5	≤ 0.1	≤ 1
Mn	S	P	B	Se	Cu	Al	Ti
≤ 1	≤ 0.03	≤ 0.04	≤ 0.01	≤ 0.005	≤ 0.5	≤ 0.5	≤ 0.15

Table 2.1. Nominal composition of EOS NickelAlloy HX, in wt%

Since each alloy requires a development of specific printing parameters for its own, there are only limited numbers of printable material grades in the state-of-the-art LPBF and EBM techniques. The printability depends on various factors, including dimension accuracy, composition variation, lack of fusion defects and more, and multiple material properties of each composition need to be taken in consideration, for example, thermal expansion coefficient, diffusion coefficient and thermal conductivity [16]. For Ni-based superalloy, the printability highly depends on the amount of γ' phase [17], which is an ordered coherent intermetallic precipitate with a chemical formula $\text{Ni}_3(\text{Al,Ti})$. γ' precipitation is an important strengthening mechanism for Ni-based superalloy, especially for good creep resistance it requires 40-80% volume fraction of γ' [18]. However, the higher content of Al and Ti reduces the weldability of an alloy and it will be prone to form defects by various mechanisms, such as solidification cracking, liquation cracking and strain-age cracking [17], therefore, the content of Al and Ti is an indication of the printability. As a result, it is difficult to print alloys with high volume of γ' , for instance, Inconel 738 [19, 20], CM247LC [21], CMSX-4 [22, 23] and René N5 [24]. On the other hand, the solid solution strengthened alloys such as Inconel 625 and Hastelloy X, and the γ'' strengthened alloy like Inconel 718, exhibit better printability. Given the good printability of HX, it is reasonable to assume that the as-built specimens are nearly defect free, so the influence such as porosity and lack of fusion is not within concern when it comes to the discussion of deformation mechanism in this study.

2.2.1 Solid solution strengthened alloy

HX is a majorly solid solution strengthened alloy, in which the solute atoms induce lattice distortion and create coherent strain field to hinder the dislocation movement [25]. Each element contributes differently to the solid solution strengthening where the eventual strengthening can be quantified by equation (2.1) [26–28],

$$\Delta\sigma = \sum_i \frac{d\sigma}{\sqrt{dc_i}} \sqrt{c_i} \quad (2.1)$$

where c is the solute concentration and $\frac{d\sigma}{\sqrt{dc_i}}$ is a strengthening coefficient varying with different element. The yield strength correlates well with the solid solution strengthening, and the strengthening coefficient can be obtained by fitting the Feltham equation to the experimental data [27]. With the consideration of ternary and higher order of alloy system, the strengthening coefficients in the nickel γ matrix of the alloyed element in HX are shown in Table 2.2, where Mo and C exhibit high strengthening capability.

Element	Fe	Cr	Mo	W	Co	C	Si	Mn	Co	Al	Ti
$\frac{d\sigma}{\sqrt{dc_i}}$	153	337	1015	977	39.4	1061	275	448	86.7	225	775

Table 2.2. Solid solution strengthening coefficients of alloyed element in nickel γ matrix, $\frac{d\sigma}{\sqrt{dc_i}}$.

Precipitation strengthening of the various secondary phases in γ matrix is an important strengthening mechanism in Ni-based superalloys [29]. The alloys with formation of intermetallic compounds, γ' and γ'' , are classified as the principal precipitation strengthened material group, which can fulfil high requirement of mechanical properties at elevated temperatures. For the material group that is essentially solid solution strengthened, the formation of carbides provides additional but limited strengthening. The formation of carbides at grain boundaries stabilizes the local grain boundaries against the excessive shear, which is a common case, meanwhile, the formation of carbides within grains can also strengthen the material via dispersion hardening.

No precipitation is shown in the as-built state of HX [30], but with different heat treatment conditions there are four types of precipitation being observed [31, 32], $M_{23}C_6$, M_6C , σ and μ . The time-temperature-transformation (TTT) diagram of conventionally manufactured HX can be found in Figure 2.2. $M_{23}C_6$ and M_6C are carbides with FCC crystal structure where it is common to find $M_{23}C_6$ in Cr-rich region and M_6C in Mo-rich region. σ and μ are so-called topological close-packed (TCP) phases, and both are detrimental to mechanical properties due to the incoherent crystal structures.

Figure 2.2. TTT diagram of conventionally manufactured Hastelloy X reprinted from J. Zhao et al. [31]. Four different precipitations are observed and labeled, including $M_{23}C_6$, M_6C , σ and μ .

2.3 Typical microstructure for LPBF

The directional and concentrated laser energy input induces high thermal gradient, guiding the continuous grain growing approximately along the building direction (BD). As a result, elongated grain structure and cell structure are common features in LPBF, which can be found from different perspectives in the scanning electron microscope (SEM), electron backscatter diffraction (EBSD) and light optical microscope (LOM) images in Figure 2.3. In the lower magnification, a roughly elongated grain structure is shown from the side view and an equiaxed grain structure is shown from the top view, and a cell structure exists within the high angle grain boundaries ($> 10^\circ$) under the further higher magnification. The cell structure consists of primary dendrite arms formed during the rapid cooling process. The grain growth direction (GD) of the dendrite arm follows the energy-favorable crystallographic $\langle 001 \rangle$ direction, hence, the general texture behavior can be estimated from the cell structure. Different input energy source and printing parameters influence the grain growth process and lead to different final texture [33–35]. Take EBM for a comparison, a strong $\langle 001 \rangle$ texture component along the BD is commonly found in EBM materials, however, different textures along the BD in LPBF materials are reported in the literature [36–40]. The influence of scanning rotation and the geometrical effect will be discussed later in this chapter.

Figure 2.3. The as-built microstructure of LPBF HX. The coordinate system in this study is given here. (a) EBSD grain orientation mapping in the side-view where the BD is along the vertical direction. The rough as-built surfaces are at the left and right end. The color legend corresponds to the BD. The black lines refer to $> 10^\circ$ grain boundaries and the gray lines refer to $> 2^\circ$ grain boundaries. (b) EBSD grain orientation mapping in the top-view where the BD is out-plane, and the surface is at the top end. The color legend and grain boundaries settings are the same as in (a). (c) A SEM image from the selected area in (a), showing the elongated cell structure. (d) The coordinate system of the sample, where the building direction (BD), wall direction (WD) and transverse direction (TD) are defined. The different thickness is the width variation on the wall direction. (e) Melt pool structure from LOM images. The grain growth directions are labeled with white arrows by the elongated direction of cell structure. A selected area showing the relation between the grain growth and melt pool boundaries.

Melt pool structure is a common feature in LPBF generated due to the localization of concentrated laser energy input. Melt pool structure can be revealed after being electro-etched, hence, it suggests that element segregation takes place around melt pool boundaries. As shown in Figure 2.3e, the sizes of melt pools are

not identical. The size can be larger than the powder layer thickness brought by the recoater in each printing step, pointing out that the laser can melt through multiple powder layers while passing the surface of powder bed. In addition, the grain growth can continuously pass through multiple melt pool boundaries, and the GD constantly changes the direction. The shape and curvature of melt pool can be an indication of general texture behavior since the GD varies in different melt pool morphology [41, 42], see the schematic illustration in Figure 2.4. A strong $\langle 001 \rangle$ texture can be found in the shallow melt pool as the GD is mostly along the BD. For the deeper melt pool, the deviation of the GD from the BD becomes larger, and it leads to a more random texture. However, the cases in Figure 2.4 are only valid when the melt pool morphology are identical over a large area. Various printing parameters can influence the melt pool morphology, for instance, the laser energy density and scan rotation. The melt pool with various sizes and morphology shown in Figure 2.3e is a good example for the case that the scan rotation of 67° was applied. The 67° scan rotation is one of the standard process in an EOS equipment, and it serves the purpose for reaching isotropic material properties by increasing the randomness of heat flow [11]. A further discussion with the respect to grain growth, melt pool boundaries and microstructure can be found in **Paper III** and **Paper IV**.

Figure 2.4. Schematic illustration of melt pool structures with different morphology. The blue and red arrows refer to the grain growth with the preferable $\langle 001 \rangle$ crystallographic direction.

The microstructure of LPBF materials is far from homogeneous compared to conventional manufactured materials, therefore, the statistic texture analysis is a good way to make fair comparison among different printing parameters. The effect of different scan rotation on a LPBF stainless steel 316L is shown in Figure 2.5 and Figure 2.6 [12]. In Figure 2.5, a strong $\langle 011 \rangle$ texture component is found in the sample with no scan rotation being applied, and the asymmetrical intensity between the transverse direction (TD) and the wall direction (WD) shows the similarity with the Goss texture that is commonly found in the rolling steel [43]. When the scan rotation is applied, the texture become weaker and the intensity turns to be more symmetrical, which exhibits a $\langle 011 \rangle$ fiber texture along the BD. The orientation distribution function (ODF) in Figure 2.6 shows a clear transition from a texture component to a fiber texture as the level of scan rotation increasing, where the strongest fiber texture is found in the sample with 67° scan rotation. A further investigation on the mechanical behaviors of different as-built texture can be found in **Paper I**.

Figure 2.5. The (111), (200) and (220) pole figures of LPBF stainless steel 316L printed with different scan rotation, 0°, 90°, 45° and 67°. The pole figures are measured from neutron diffraction. The center refers to the BD, the vertical poles refer to the TD and the horizontal poles refer to the WD. The color legend is shared between the different samples with the same pole figure respectively.

Figure 2.6. The orientation distribution function (ODF) of LPBF stainless steel 316L printed with different scan rotation, 0° , 90° , 45° and 67° . Each ODF is reconstructed from five poles measured from neutron diffraction, including (111), (200), (220), (311) and (222).

2.4 Thin-walled structure

An important topic of this thesis is to investigate the thin-wall effects in LPBF materials. It is assumed that the geometrical variation will lead to different thermal boundary condition and result in the evolution of microstructure. Therefore, a set of plate-like specimens with different nominal thicknesses, 1mm, 1.5mm, 2mm, 2.5mm and 4mm, are printed for microstructure and mechanical behavior study, where the flat dog-bone tensile testing specimens are taken directly from the plates by electrical discharge machining (EDM), see Figure 2.7. In addition, a rod-like specimen has been printed and set as a reference for the texture study, where the diameter is 18mm and the length is 125mm. In order to conduct an universal study of the geometrical effect in LPBF, the standard printing parameters in EOS equipment are applied for a fair comparison to the other relevant research.

Figure 2.7. (a) The plate-like specimens of LPBF HX with different thicknesses, 1mm, 1.5mm, 2mm, 2.5mm and 4mm. The dog-bone tensile testing specimens are taken directly from the plates by using EDM. (b) The reference group, rod-like specimen of LPBF HX with a diameter of 18mm and a length of 125mm.

2.4.1 Thickness and height-dependent

To reveal the statistic microstructure behavior, the texture measurement by neutron diffraction has been carried out on both plate-like and rod-like specimens. The measured gauge volume for the plate-like specimen covered the entire thickness including the rough surface. For the rod-like specimens, the measured samples were taken from the bottom and the top, which represent the initial state and stable state of the printing process, and the samples were machined to the diameter of 10mm and the height of 10mm to show the “bulk texture”.

Figure 2.8a shows the (200), (220) and (222) pole figures (PF) of each sample, and the sample coordinate system is the same as the one shown in Figure 2.3d. The texture of plate-like specimen exhibits a clear thickness-dependence. An approximate $\langle 001 \rangle$ fiber texture along the TD is shown in 1mm thick sample, and it becomes a $\langle 011 \rangle$ fiber texture with weaker orientation along the BD in 4mm thick sample. Fiber texture stands for a feature that a certain crystallographic orientation aligns toward a direction with respect to the sample coordinate system. Take the PFs of 4mm for example, a strong point-like intensity locates at the center of (220) PF and ring-like intensities are found at different corresponding polar angles in each PF, which indicates a $\langle 011 \rangle$ fiber texture. The $\langle 001 \rangle$ and $\langle 111 \rangle$ crystallographic orientations are randomly distributed with the axis of $\langle 011 \rangle$ direction, hence, it results in a ring-like intensity in the PFs.

A comparison to the rod-like sample in Figure 2.8b can further reveal the geometrical effect since the thermal boundary condition is symmetric on the plane normal to the BD where the grain growth is expected to be homogeneous on the planar directions. However, ring-like intensity is not found in the PFs, instead, clear point-like intensities with two-fold symmetry appear. The texture of rod-like sample shows similarity with Goss texture $\{011\} \langle 100 \rangle$, which is a texture commonly found in silicon-steel from rolling processes [44], and it has been reported in other LPBF Ni-based superalloy as well [43]. Yet, the spatial relation between the rod and the building chamber was not recorded and only the BD is known, so

the effect such as the laser movement to the final texture remains unclear. On the other hand, the texture from the top of the build is stronger and sharper than the bottom of the build where the bottom is close to the base plate. Hence a slightly less identical microstructure caused by the unstable thermal field in the initial state of the printing is indicated. A further discussion correlating the microstructure and statistic texture can be found in **Paper III**.

Figure 2.8. The (200), (220) and (222) pole figures of the as-built specimens in Figure 2.7. The pole figures are obtained from neutron diffraction, and the color legend is shared by the three pole figures of each specimen respectively. (a) The plate-like specimens. The center refers to the BD, the vertical poles refer to the TD and the horizontal poles refer to the WD. (b) The rod-like specimens. The center refers to the BD. The specimen is built from the bottom to the top, hence, the bottom is close to the base plate.

2.4.2 Surface roughness

It is common to find rough surface in the as-built LPBF and EBM materials due to the unmelted and semi-melted powders attached on the surface as shown in Figure 2.3a. The surface roughness can cause stress concentration and be detrimental to mechanical properties, such as fatigue [45–47]. In addition, the surface roughness also influences the size of measured net cross-section that carries the loading, which is the primary concern in this study. With the thinner sample, the proportion of surface roughness in the entire cross-section becomes larger. Given that the attached powder at the surface has no contribution to the loading bearing, a cross-section calibration method is proposed by reducing a selected surface roughness parameter. The selection of proper surface roughness parameter is demonstrated in **Paper III**. This method can be applied when the length of a certain dimension is close to the minimum limit.

Deformation and fracture mechanisms

To bring AM in high temperature applications such as gas turbine, it is necessary to examine the mechanical properties at elevated temperatures. Tensile tests from room temperature up to 700 °C have been carried out on the flat dog-bone specimens shown in Figure 2.7. Two types of tensile specimen were set for the anisotropy study, one was the “Vertical” where the loading direction (LD) was applied along the BD, and the other one was “Horizontal” where the LD was applied perpendicular to the BD. The mechanical behavior under different strain rates at 700 °C has also been investigated with the comparison to a wrought HX set as a reference group of conventional manufacturing.

Anisotropic mechanical properties are commonly found in AM materials where the strength along the BD is slightly weaker than the TD [36, 37, 48]. This thesis studies the anisotropic mechanical behavior with different influencing factors. The influence of temperature variation will be discussed in **Paper II** and **Paper III**, where a ductile to brittle transition and a weakening trend of anisotropy at elevated temperature have been traced. The specimen thickness also plays an important role as a clear texture difference has been found, and the texture evolution will be discussed in **Paper III** and **Paper IV**. Finally, the influence of strain rate at 700 °C will be discussed in **Paper IV** after a discovery of ductility transition. Multiple factors result in the mechanical behaviors, and each of them will be discussed in the following sections.

3.1 Anisotropic mechanical behaviors

The stress-strain curves in Figure 3.1 reveal the anisotropic mechanical behaviors where the yield strength, ultimate tensile strength (UTS) and work hardening of the horizontal tests are better than the vertical test. However, the vertical ductility

outperforms the horizontal ductility, especially at 700 °C, the horizontal ductility shows a significant drop while the vertical ductility remains at the same level as the lower temperature tests. A further investigation at 700 °C by using slow strain rate testing (SSRT) is shown in Figure 3.2. A continuous softening right after the yield point is present in both vertical and horizontal tests. In addition, a further drop of horizontal ductility is found in both the strain rate of 10^{-5} s^{-1} and 10^{-6} s^{-1} .

Figure 3.1. Tensile stress-strain curves of LPBF HX at elevated temperatures with the strain rate of 10^{-3} s^{-1} . (a) The vertical tests. (b) The horizontal tests.

Figure 3.2. Slow strain rates tensile testing of LPBF HX at 700 °C. Three different strain rates are compared, including 10^{-3} s^{-1} , 10^{-5} s^{-1} and 10^{-6} s^{-1} .

3.2 Grain morphology effect

The elongated grain and cell structure shown in Figure 2.3 is an important factor contributing to the anisotropic mechanical behavior. A simplified schematic

illustration of the LD and the grain structure is proposed in Figure 3.3, where the green elongated grains represent the major $\langle 011 \rangle$ texture component and the red part stands for the minor $\langle 001 \rangle$ texture component along the BD. For the horizontal tests, as the loading is applied on the longitudinal side of the elongated grain, it is relatively easier for the grain boundary detachment to take place and results in lower ductility. Grain boundary of Ni-based superalloys is supposed to be stable at elevated temperatures, however, the increasing creep damage and weaker chemical bonding [49] from intermediate to high temperature embrittle the grain boundary. The enhancement of grain boundary embrittlement from 500 °C to 700 °C is clearly indicated from the fracture surface study shown in **Paper II**, and the further time-dependent creep damage can be found in the SSRT study in **Paper IV**. For the vertical tests, the elongated grains are approximately along the BD in the as-built state, and they will align further closer to the BD after the deformation, for which a discussion of grain rotation can be found in **Paper III** and **Paper IV**.

Figure 3.3. Schematic illustration of the spatial relationship between the LD and the grain structure. The green elongated grains refer to the major $\langle 011 \rangle$ texture component along the BD, and the red elongated grains refer to the minor $\langle 001 \rangle$ texture component along the BD.

3.3 Dislocation movement and texture strengthening

Dislocation is an important crystallographic defect in crystalline metallic materials since the plastic deformation usually undergoes via the movement of dislocation. Dislocation is a linear crystallographic defect or irregular atomic arrangement, which can be categorized into two major types, edge dislocation and screw dislocation. There are different types of dislocation movement at various conditions, such as planar slip, cross-slip and climb, and the planar slip will be discussed first [50].

For most of the crystallographic metals, the slip of dislocation takes place on the densest lattice plane with the direction pointing toward the shortest distance between atoms, hence, the $\langle 011 \rangle \{111\}$ is the favorable slip system for materials with FCC crystallographic structure [51]. To activate a slip, the shear stress on

a slip system needs to reach critical value, τ_c , which is often named as critical resolved shear stress (CRSS), and the spatial relationship between the applied load and slip system is determined by Schmid's law in equation 3.1 [52],

$$\tau_c = \sigma_x \cos \lambda \cos \phi \quad (3.1)$$

where σ_x is the stress of the uniaxial load, λ is the angle between slip direction and load direction, ϕ is the angle between slip plane normal and load direction. However, the LPBF material is not a single crystal, the polycrystal deformation behavior has to be taken into consideration. As proposed by G. I. Taylor [53], the deformation of each grain in polycrystal needs to be compatible with the neighbor grains, so the shape change of each grain should be the same as the entire polycrystal. In Taylor's assumption, the critical shear stress causing the slip is the same on all active system, and for the case of FCC crystals, each grain with different orientation experiences the deformation consisted of the summation of 5 out of 12 shear strain, which is the 5 lowest shear strain in the $\langle 011 \rangle \{111\}$ slip system. The external applied stress and the internal shear stress can be determined with the Taylor's factor by equation 3.2,

$$\sigma_x = M\tau \quad (3.2)$$

where σ_x is the external uniaxial tension, M is the Taylor's factor and τ is the shear stress. Slip is easier to be activated in orientation-favorable grain, so crystallographic texture plays a key role in anisotropic mechanical properties where the yield strength is a direct indication [54, 55]. The orientation-dependence of Taylor's factor is computed and plotted in Figure 3.4 under the axisymmetric deformation of FCC crystals [50, 56], and the average Taylor's factor of all orientations is 3.067 which represents a FCC polycrystal with random texture. To analyze the anisotropy in this study, the Taylor's factors of the plate-like samples with different thickness and the rod-like samples are calculated from the pole figures shown in Figure 2.8, and the strain tensor is applied corresponding to the vertical and horizontal loading in this study with axisymmetric flow, see Figure 3.5. With different texture in the as-built state of each specimen, the stronger texture shows larger anisotropy of Taylor's factor between the vertical and horizontal loading conditions, which means that the top of the rod-like specimen shows the largest difference. The correlation between the yield strength and calculated Taylor's factor can be found in **Paper III**.

Figure 3.4. Computed orientation dependence of Taylor's factor for FCC crystals undergoing axisymmetric deformation reprinted from G. Y. Chin et al. [50, 56].

Figure 3.5. Calculated Taylor's factor for the plate-like and rod-specimens, with the strain tensor of axisymmetric flow corresponding to the vertical and horizontal loading condition. The calculation is based on the ODF, which is reconstructed from five pole figures measured by neutron diffraction.

The major difference between edge dislocation and screw dislocation is the movement mechanism [50]. Edge dislocation can only move on the unique slip plane that contains its Burgers vector, but screw dislocation is parallel to its Burgers vector and it can change from one slip plane to another. For edge dislocation to change slip plane, it needs a process called “climb” with the assistance of diffusional flux of atoms, and climb becomes important at elevated temperature during creep deformation since it provides an alternative mechanism to avoid obstacle [57]. When a screw dislocation slips from one plane to another plane, the process is called “cross-slip”, which is an energy-favorable way to pass through hindrance. A screw dislocation can dissociate into two partial dislocations and create a stacking fault in between, which can lower the total energy, but the partial

dislocations need to recombine to form screw dislocation if it tends to cross-slip to another plane. The recombination of partial dislocations is not energy-favorable since it increases the total energy, which is aided by the applied stress and thermal activation, and the stacking fault energy determines the difficulty of the process [58]. The higher the stacking fault energy the lower applied stress it needs to cause the recombination, hence, the slip trace is wavy in metals with high stacking fault, for instance aluminum, due to the more frequently happening cross-slip; for the opposite case the slip trace is straight in metals with low stacking fault, for instance copper [50, 59–61]. However, it is even more complicated in the real cases, different partial dislocation movement can be found in the literature [18, 62][18], [62]. At high temperature, multiple slip systems and mechanisms can be activated, so the anisotropy such as the texture-dependent mechanical behavior becomes weaker [63].

3.4 Texture evolution

Lattice rotation often occurs gradually during the slip of dislocation, and it will lead to different crystallographic textures along the loading direction after deformation, depending on the crystal system and the loading condition. For example in FCC crystals, the end preferred orientation of single crystals is $\langle 112 \rangle$ after tensile deformation and $\langle 110 \rangle$ after compressive deformation. In addition, the end preferred orientations for polycrystals are mostly the same as single crystals, however, the end preferred orientations under tensile deformation become $\langle 110 \rangle$ and $\langle 111 \rangle$ for the case of FCC polycrystals. As tensile deformation mechanism of FCC crystals is the major interest in this study, the following discussion will focus on the texture evolution of the vertical and horizontal tensile loading in LPBF materials.

Figure 3.6. The texture evolution of FCC single crystals under tensile deformation adapted from W. F. Hosford [50]. (a) The primary slip system is activated and the texture evolves toward the [101] corner in the adjacent triangle. (b) When the texture reaches the [100]-[111] boundary, the favorable slip system in the adjacent triangle will be activated, which the texture tends to evolve toward [110] corner in the original triangle. The two conjugate slip systems are equivalent along the [100]-[111] boundary, and the end preferred orientation will evolve toward [112].

The evolution of preferred orientation in tension of FCC single crystals is schematically illustrated in Figure 3.6. With the consideration of the primary slip system $\{111\}\langle 011\rangle$ and given a basic stereographic triangle with [100], [110] and [111] at the corners, the texture will evolve toward the [101] corner in the adjacent triangle. When the texture passes through the [100]-[111] boundary of the triangle, the conjugate slip system will be activated, and the texture will evolve back to the [110] corner of the original triangle. The two conjugate slip systems are in competitive relationship while the texture being on the [100]-[111] boundary, and the texture moving gradually toward the end preferred orientation along the boundary [50].

To trace the texture evolution in this study, texture analysis by using neutron diffraction has been carried out on the sample with different deformation level, see Figure 3.7. Like the different mechanical behavior in the stress-strain curves, the vertical loading exhibits a significantly different texture evolution with higher level of change compared to the horizontal loading. For the vertical loading, a clear texture evolution toward the [100]-[111] boundary in the inverse pole figure (IPF) of BD is shown at the half-deformed point ($\Delta\epsilon = 19.5\%$), and it further splits into two texture components, $\langle 112\rangle$ and $\langle 001\rangle$, at the fully deformed point. The end preferred orientation of $\langle 112\rangle$ indicates that the deformation behavior of the vertical loading is similar to single crystals. However, less lattice rotation is expected in the horizontal loading since the deformed textures are rather close to the as-built state, and the deformation behavior shows similarity to polycrystals due to the end preferred orientations of $\langle 001\rangle$ and $\langle 111\rangle$.

Figure 3.7. Inverse pole figures reconstructed from five pole figures obtained by neutron diffraction, with respect to the WD, TD and BD. (a) As-built texture. (b) Texture evolution under tensile deformation at different strain level at 700 °C.

In addition, the initial state of the texture has a great influence on the texture evolution, see Figure 3.8. As a strong $\langle 001 \rangle$ texture component exists along the TD in 1mm as-built sample, the end preferred orientation stays at $\langle 001 \rangle$ along the TD even though the LD has been applied on the BD, which is quite different compared to the 4mm deformed sample. A further discussion on the grain structure evolution under the lattice rotation, the effects of temperature and strain rates can be found in **Paper III** and **Paper IV**.

Figure 3.8. Inverse pole figures reconstructed from five pole figures obtained by neutron diffraction, with respects to the WD, TD and BD. The as-built texture and the fully deformed texture of different loading condition at 700 °C are shown. (a) Texture evolution of 1mm plate-like specimen under tensile deformation. (b) Texture evolution of 4mm plate-like specimen under tensile deformation.

3.5 Deformation twinning

Twin is a feature in crystals that the orientation of a certain region of lattice is mirror-symmetric to the neighbor lattice with a specific angular relationship. Twinning is also a deformation mechanism for metals, especially acting as an important alternative for hexagonal close-packed (HCP) metals due to the limited number of easily activated slip systems. There are two common types of twins, annealing twins are created during recrystallization, and deformation twins are formed by uniform shearing [50]. For conventionally manufactured HX, it is possible to observe annealing twins in the as-received material, and the annealing twins play a good role in hindering the dislocation movement during deformation [64–66], but it is not within the discussion in this study since annealing twins were not observed in LPBF HX.

Deformation twinning is caused by shear stress where the twinning occurs on $\{111\}$ planes and $\langle 11\bar{2} \rangle$ directions, and the direction of the twinning shear is strict, so for example, the $(111)[11\bar{2}]$ shear can cause deformation twinning but not the opposite direction $(111)[112]$. The lattice rotation of deformation twinning is abrupt and it can be considered as a nucleation and growth process [67]. The local

stress concentration are important nucleation sites for deformation twinning, where the response of dislocation to the local concentration plays a decisive role. For example, Lomer-Cottrell lock of partial dislocations [68], the dislocation reaction in the region of grain boundaries [69, 70] and the microtwinning when partial dislocations encounter precipitation [71]. The mechanisms of dislocation reaction for triggering deformation twinning are complicated and there are more different scenarios in the literature.

The tendency of materials to twin depends on several factors, for instance, temperature, strain rate, grain size, texture and stacking fault energy [67, 72, 73]. In general, for FCC materials, the lower temperature and lower strain rate are favorable for the formation of deformation twins, while the stacking fault energy is an even more important factor where the tendency to twin increases with lower stacking fault energy. Stacking fault energy is highly composition dependent. For Ni-based superalloys, the stacking fault energy for pure Ni is 125 mJ/m^2 [58], each element in Ni-based superalloy exhibits different level of influence [74], and Mo serves as a leading factor in HX in this study due to the high potential to lower the stacking fault energy [75]. Besides, the texture also plays a significant role, for example, it is more common to observe deformation twins in grains with $\langle 111 \rangle$ orientations along the LD rather than the slip favorable $\langle 001 \rangle$ oriented grains in twinning-induced plasticity (TWIP) steel [76]. A further discussion on the favorable texture for deformation twinning in LPBF materials can be found in **Paper IV**.

3.6 Creep damage

Creep is a time-dependent plastic deformation behavior that is composed of multiple temperature-dependent mechanisms, and creep damage becomes significant at high temperature, which is needed to be taken into consideration in the slow strain rate tensile testing in this study.

A creep test is carried out on a material usually with constant applied engineering stress (constant load) and temperature, and creep behavior can be divided into three stages shown in Figure 3.9. At the first stage, the material reacts elastically as the load being applied, followed by a transformation to plastic deformation with rapid strain rate. Then the strain rate gradually decreases to a steady-state where the second stage starts. Finally, the strain rate increases again at the third stage due to the shrinkage of cross-section and the possible porosity and cavitation development in the tertiary creep.

Figure 3.9. Schematic illustration of time-dependent creep deformation behavior, which is consisted of three stages, primary creep, steady-state creep and tertiary creep.

Creep deformation can be described as viscous flow of which the viscosity depends on the thermal activation energy. In polycrystals, the grain boundary sliding facilitates the viscous flow, at the same time, the compatibility among grains is maintained by the diffusional flow, dislocation movement and grain boundary migration [50]. The tensile stress separates the atoms in the grain boundaries, and it becomes a driving force for the diffusional flow that the atoms diffuse from the grain boundaries parallel to the stress axis to the grain boundaries normal to the stress axis. The atomic diffusion can either pass through the lattice (Nabarro-Herring creep, particularly at high temperature) or along grain boundaries (Coble creep, particularly at low temperature). The various dislocation movement can also dominate the creep deformation, for instance, the diffusion-assisted climb of dislocation is activated at high temperature, and the obstruction to the partial dislocation movement by the precipitation in Ni-based superalloy. Multiple factors contribute to the strain rate, including the mentioned above, and the overall strain rate is led by the most rapid mechanism. The overall steady-state creep strain rate is often described as follow in equation 3.3 [18, 77, 78],

$$\dot{\epsilon} = A\sigma^n \exp \frac{-Q}{RT} \quad (3.3)$$

where $\dot{\epsilon}$ is the strain rate, A is a constant, σ is the applied stress, n is the stress exponent, R is ideal gas constant, and T is the temperature. Q is the activation energy deduced from the experimental data, it refers to all the mechanisms mentioned above, and it often correlate with activation energy for self-diffusion. Given an applied stress and temperature, one can estimate the rupture time of a creep test from the experimental data itself by using different assumptions, such as Sherby-Dorn, Monkman-Grant relationship and Larson-Miller parameter.

As mentioned earlier that creep deformation can be seen as a viscous flow, which includes grain rotation. Hence, texture shows a great influence on the creep behavior since a grain responds differently depending on its orientation, for instance, the different level of grain rotation and texture evolution [63, 79], and the favorable orientation for deformation twinning [80]. A further study on the

creep damage in LPBF materials from the SSRT study can be found in **Paper IV**.

The main purpose of this study is to investigate the anisotropic material properties of thin-walled structure manufactured by LPBF. The study material is the plate-like specimen with different thicknesses shown in Figure 2.7, and it is provided by Siemens Energy AB, Sweden. The experimentation details will be present in this chapter, including LPBF process parameters, mechanical test and microstructure analysis.

4.1 Laser Powder Bed Fusion

The study material was built with standard printing parameters in an EOS M290 machine, and the used powder was EOS NickelAlloy HX. The thickness of each building layer was set as 20 μ m, and the 67° scanning rotation was applied. In each building layer, the hatching was first and then followed by two times of the contouring with the width of 0.15mm. The material for the entire study was meant for as-built state, so no post treatment has been applied. Plate-like specimens with different nominal thicknesses, 1mm, 1.5mm, 2mm, 2.5mm and 4mm, were built and shown in Figure 2.7.

The reference group for this study, a wrought HX, has similar nominal composition as EOS NickelAlloy HX, and a solution annealing of 20 minutes at 1175 °C followed by water quenching was applied on the material.

4.2 Mechanical test

Tensile tests with the strain rate of 10^{-3} s^{-1} were carried out at room temperature and elevated temperatures, from 400 °C to 700 °C; tensile tests with the strain rates

of 10^{-5} s^{-1} and 10^{-6} s^{-1} were done at 700°C . Under the standard ISO 6892-2, the tensile tests were performed by using an Instron 5582 - 100kN universal testing machine with strain-control using an Instron 7361C extensometer where the gauge length was 12.5mm, and the tensile specimens were placed in a heating furnace with a type K thermocouple attached to the center of the specimen for the temperature control within the variation less than $\pm 2^\circ\text{C}$. The flat dog-bone tensile specimens with the dimensions given in Figure 4.1 were taken from the plate-like specimens by using electrical discharge machining (EDM). Two types of tensile specimen were conducted in each test condition, one was “Vertical” where the LD was applied along the BD, and another one was “Horizontal” where the LD was applied perpendicular to the BD.

Tensile test specimen

Figure 4.1. The dimensions of flat dog-bone tensile specimens.

4.3 Microstructure characterization

4.3.1 Light optical microscope

A light optical microscope, Leica DM6, was used for imaging the melt pool structure and the cross-section for roughness measurement. For the melt pool structure, the image was taken from the Twin-jetted TEM thin foil where the melt pool boundaries were etched during the electropolishing process. Another alternative to reveal the melt pool structure is to electro-etch the well-polished sample with the solution of 10 vol.% oxalic acid and 10 V of the applied voltage [81].

4.3.2 Scanning electron microscope

A Hitachi SU 70 field emission scanning electron microscope (FE-SEM) equipped with an electron back scatter diffraction (EBSD) detector from Oxford Instrument was used for the microstructure analysis. Secondary electron (SE) and backscattered electron (BSE) modes were operated at 10 KV for microstructure imaging, and EBSD was operated at 15 or 20 KV for grain orientation mapping. The imaging parameters vary slightly under different circumstance where the details can

be found in each appended paper. An open source MATLAB toolbox, MTEX, was used for EBSD data analysis [82].

For the metallographic preparation, the cross-section of interest was first cut by using EDM and mounted into an electrically conductive resin, PolyFast, from Struers. Then the sample was mechanically ground step-by-step from 500 Grit up to 4000 Grit and followed by fine polishing with diamond suspension from 3 μ m down to 0.25 μ m. Finally, the sample was polished by using OP-U colloidal silicon suspension, and water was used as the last step to take away the residual particle from the suspension as much as possible.

4.3.3 Transmission electron microscope

The dislocation structure was studied by using transmission electron microscope (TEM), FEI Tecnai G2 microscope, operating at an accelerating voltage of 200 KV. The sample preparation of TEM thin foil started with grinding down to the thickness of 50-100 μ m. To further thin down the sample below 100nm, ideally 30nm for high resolution analysis, an electropolishing was applied by using Struers TenuPol-5 electrolytic machine. The solution was 10 vol.% perchloric acid with 90 vol.% ethanol, it was operated at the applied voltage of 23 V and ambient temperature of -25 °C.

4.4 Neutron diffraction

The texture analysis of bulk materials was carried out by using KOWARI, a neutron diffractometer at ANSTO [83]. Each PF was measured by monochromatic beam with a wavelength of 1.5 Å on an approximately 5 $\times$ 5° polar grid where the gauge volume covered the entire sample, and the measurement time per angular position was 5 seconds. Five PFs, (111), (200), (220), (311) and (222) were obtained and used for the reconstruction of orientation distribution function (ODF) for quantitative analysis. An open source MATLAB toolbox, MTEX, was used for texture analysis [84].

4.5 Roughness determination

The surface roughness was determined from the cross-section image analysis of the specimen by using an in-house MATLAB script [85]. The desired cross-section was cut and mounted, then multiple images was taken from a light optical microscope for statistical analysis. Different roughness parameters were calculated from the two-dimensional roughness analysis. The definition of each parameters from the standard ISO 4287 is schematically illustrated in Figure 4.2, where R_a is the arithmetic mean values of the Z-coordinates, R_c is the mean value of Zt_i in each S_i , R_p is the mean value of Zp_i in each S_i , R_z is the maximum Zt_i , R_v is the maximum Zv_i and R_q is the root mean square of the Z-coordinates.

Figure 4.2. Schematic illustration of a roughness profile.

Summary of the appended papers

- I. A. Leicht, C. H. Yu, V. Luzin, U. Klement, and E. Hryha, “Effect of scan rotation on the microstructure development and mechanical properties of 316L parts produced by laser powder bed fusion,” *Materials Characterization*, vol. 163, p. 110309, 2020.

This paper serves as an initiation of this Licentiate thesis since the effect of scan rotation on the materials properties has been carried out, which is the primary concern of printing parameters influence on as-built microstructure and mechanical behavior of LPBF materials.

The degree of scan rotation refers to the angular relationship of the laser moving direction between each adjacent scanning layer. A comparison has been drawn among the samples applied with 0°, 45°, 67° and 90° scan rotation, and it turns out that the 67° scan rotation gives relatively weaker mechanical properties (yield strength, UTS and ductility) but a more homogeneous grain structure at the same time. For the sample without scan rotation, a major texture component $\langle 011 \rangle$ and a minor texture component $\langle 001 \rangle$ along the BD is discovered along the BD, and it becomes $\langle 011 \rangle$ fiber texture along the BD as different level of scan rotation being applied. Scan rotation induces the randomness in the thermal field during the printing process, and it results in different level of anisotropic microstructure. The 67° scan rotation is one of the standard printing parameters in an EOS M290 equipment, and the purpose is to decrease the anisotropic mechanical behavior by tuning to a more isotropic microstructure. However, even with the 67° scan rotation, it is inevitable to observe elongated grain structure with specific texture along the BD. Therefore, the coming papers will investigate the influence of elongated grain structure on the mechanical behavior of LPBF materials.

- II. C.-H. Yu, R. L. Peng, J.-E. Lundgren, and J. Moverare, “Anisotropic mechanical properties of thin-walled Hastelloy X manufactured by laser powder bed fusion,” **European Congress and Exhibition on Powder Metallurgy, 2019.**

This paper is the first study on the thin-walled Hastelloy X. Tensile tests with the strain rate of 10^{-3} s^{-1} have been carried out on the samples with different nominal thicknesses, 1mm, 1.5mm, 2mm, 2.5mm and 4mm, at room temperature and elevated temperatures, from 400 °C to 700 °C. The anisotropic mechanical properties were verified by the vertical and horizontal tests shown in Figure 2.7.

The thickness-dependent mechanical properties are observed, where the thinner sample exhibits lower yield strength and UTS, but the ductility has no such tendency. The thickness-dependence is due to the overestimated cross-section that takes the load, and a cross-section calibration by reducing the surface roughness is proposed. The anisotropic mechanical properties are demonstrated by the higher yield strength and UTS in the horizontal tests and the better ductility in the vertical tests. At 700 °C, the horizontal ductility has a clear drop while the vertical ductility remains at the same level as the lower temperatures, and a transition from ductile to brittle behavior at 700 °C is proved by the fracture surface observation.

- III. C.-H. Yu, R. L. Peng, V. Luzin, M. Sprengel, M. Calmunger, J.-E. Lundgren, H. Brodin, A. Kromm, J. Moverare, “Thin-wall effect and anisotropic deformation mechanisms of an additively manufactured Ni-based superalloy,” **submitted manuscript.**

This paper is a continuous study on the thin-walled Hastelloy X to further investigate the cross-section calibration method and the different deformation mechanism between the vertical and horizontal tests.

To verify multiple thickness-dependent factors, a microstructure study, surface roughness measurement, texture analysis and residual stress measurement have been carried out. The statistic texture analysis by using neutron diffraction reveals a clear texture transition from the $\langle 011 \rangle$ fiber texture along the BD to the $\langle 001 \rangle$ fiber texture along the TD when the thickness decreases. By combining the study of microstructure, melt pool structure and grain orientation maps, the average texture of an entire component can be deduced from the angular relationship between the elongated grain structure and the BD. Besides, since the rough surface of as-built LPBF component is inevitable in nature, a cross-section calibration method is developed with an assumption that the thickness-dependent yield strength is mainly related to the Taylor’s factor. The Taylor’s factor is calculated from the pole figures obtained by neutron diffraction, and it corresponds well with the decreasing tendency of yield strength as the sample becomes thinner, hence, a more precise cross-section estimation can be made by comparing the yield strength between different samples. The concerns of residual stress, dislocation density and grain size effect are ruled out by the result from XRD analysis since

there is no thickness-dependence. Therefore, the cross-section calibration method by reducing roughness parameter R_p is proposed to obtain a more precise yield strength when the length of a dimension is close to minimum limit.

The elongated grain structure is responsible for the anisotropic mechanical properties. A further investigation of dislocation structure under TEM image also uncovers an elongated subgrain structure with dislocations piling up at grain boundaries, which is assumed to contribute to the anisotropy. Besides, the calculated Taylor's factors for the vertical and horizontal loading conditions show that the texture exhibits a great influence on the anisotropy. The deformation mechanism study is conducted by examining the microstructure and texture of the deformed sample. At room temperature and 700 °C, a clear lattice rotation is found in the vertical tests, and the trace of texture evolution is similar to single crystals deformation behavior. On the other hand, the microstructure of horizontally deformed sample does not undergo significant change, and the texture evolution is similar to polycrystals.

This paper also makes a comparison to conventionally manufactured materials, a wrought HX, with consideration of the tensile mechanical behavior, grain structure and dislocation structure. The wrought HX exhibits lower yield strength but better work hardening and ductility, and the reason for the difference is due to the high dislocation density of LPBF HX at the as-built state. During the LPBF process, the high thermal gradient deforms and strengthens the material, hence, the yield strength is higher than the wrought HX but the work hardening capacity is consumed at the same time.

IV. C.-H. Yu, R. L. Peng, H. Brodin, and J. Moverare, "Anisotropic deformation and fracture mechanisms of an additively manufactured Ni-based superalloy," in TMS Superalloys 2020.

This paper is a continuous study on the mechanical response of LPBF Hastelloy X to the elevated temperatures. Since a ductile to brittle transition is traced at 700 °C, this work further investigates the mechanical behavior under different slow strain rates tensile testing at 700 °C. The comparison is drawn among the three different strain rates, 10^{-3} s^{-1} , 10^{-5} s^{-1} and 10^{-6} s^{-1} , and it is also compared with the mechanical behavior of a wrought HX. The strain rates of 10^{-5} s^{-1} and 10^{-6} s^{-1} are defined as slow strain rate.

At the strain rate of 10^{-3} s^{-1} and in the intermediate temperature region, a degradation of strength at higher temperature is shown, but the mechanical behaviors from 400 °C to 600 °C are similar. In addition, dynamic strain aging (DSA) is discovered, and a transition from type A to type C is traced from 400 °C to 600 °C. The DSA behavior is slightly different between the vertical and horizontal tests, and it is assumed to be related to the elongated subgrain structure and the dislocations piling up at subgrain boundaries. Compared to conventionally manufactured HX, the temperature region of the type A to type C DSA transition in LPBF HX is 50 °C-100°C lower. At 700 °C, the level of work hardening decreases and the DSA does not occur, moreover,

the horizontal ductility drop is found while the vertical ductility remains at the same level.

At 700 °C with the strain rates of 10^{-5} s^{-1} and 10^{-6} s^{-1} , a degradation of strength with slower strain rate is shown, and softening right after the yield point occurs in both the vertical and horizontal tests. The horizontal ductility of both slow strain rates has a further drop with the same level while the vertical ductility still remains at the same level. The degradation of strength and the softening behavior are caused by creep damage. Creep voids are observed at grain boundaries, and they are responsible for the decrease of horizontal ductility. From the fracture surface study, the grain boundary embrittlement is more profound in the horizontal tests, and it also occurs in the vertical tests but depends on the angular relationship between the grain boundary and the LD.

The most interesting discovery is that deformation twins are observed and only in the vertical tests. Compared to other Ni-based superalloy, the tendency for deformation twinning to occur in HX is low due to the high stacking fault energy, and deformation twinning in conventionally manufactured HX has not been reported in the literature. The deformation twins are mostly found in the $\langle 111 \rangle // \text{LD}$ oriented grains, which share similarity with the tensile behavior in TWIP steels and the creep behavior in single crystal Ni-based superalloys. It is assumed that the original $\langle 011 \rangle // \text{LD}$ oriented grains undergo lattice rotation toward $\langle 111 \rangle$ crystallographic direction, and the deformation twinning takes place in the twinning favorable $\langle 111 \rangle // \text{LD}$ oriented grains with the assistance of stress concentration at grain boundaries. The larger level of lattice rotation and deformation twinning enable the vertical ductility to remain at high level even with creep damage.

This Licentiate thesis has systematically investigated the microstructure and tensile mechanical behavior of Hastelloy X manufactured by laser powder bed fusion. The following conclusions are drawn into four themes to answer the aims and research questions of this study.

1. As-built microstructure

- The directional laser energy input leads to elongated grain and subgrain structure, and the angular relation between the grain growth direction and the building direction leads to different average texture results.
- Scan rotation induces randomness in the thermal field during the printing process. As a result, a major $\langle 011 \rangle // \text{BD}$ with a minor $\langle 001 \rangle // \text{BD}$ texture component is shown in the sample without scan rotation, and it transfers to $\langle 011 \rangle // \text{BD}$ fiber texture with applied scan rotation.

2. Thin-walled effects

- With a variation of thickness from 4mm down to 1mm of plate-like samples, a clear texture transition from $\langle 011 \rangle // \text{BD}$ fiber texture to $\langle 001 \rangle // \text{TD}$ fiber texture and a declination of calculated Taylor's factor are discovered as the thickness decreases. A reference group of rod-like sample shows a texture similar to Goss texture, but the mechanism for its formation remains unclear.
- The declination of calculated Taylor's factor corresponds well with the decreasing tendency of yield strength. A load bearing cross-section calibration method is proposed by reducing a roughness parameter R_p .
- The different crystallographic texture in the as-built state leads to different texture evolution.

- The residual stress of 4mm and 1mm sample is at the same level. A tensile residual stress around 90 MPa along the BD is traced and it is nearly stress free along the TD.

3. Tensile behavior at elevated temperatures and different slow strain rates

- With the strain rate of 10^{-3} s^{-1} , the work hardening behavior and ductility remain similar at different intermediate temperatures, from 400 °C to 600 °C, but a degradation of yield strength and UTS is revealed with the elevated temperature. At 700 °C, a ductile to brittle transition of fracture behavior is found, and it is more significant in the horizontal tests due to the grain boundary detachment.
- Dynamic strain aging is found between 400 °C and 600 °C. A transition from type A to type C is shown but with lower temperature range compared to conventionally manufactured HX, and the absence of DSA at 700 °C is assumed to be related to the ductile to brittle transition.
- At 700 °C with the slow strain rates of 10^{-5} s^{-1} and 10^{-6} s^{-1} , creep damage is found to cause the degradation of strength, the softening behavior right after the yield point and the grain boundaries embrittlement by forming creep voids. Deformation twinning is discovered only in the vertical tests, which occurs mostly in the twinning favorable $\langle 111 \rangle // \text{LD}$ oriented grains, and it is beneficial for the vertical ductility to remain at high level even under creep damage.
- With a comparison between LPBF and wrought HX, the rapid cooling in the LPBF process induces high dislocation density and strengthens the material, but it also sacrifices the capacity for work hardening at the same time.

4. Anisotropic mechanical behaviors

- In general, the horizontal strength is higher while the vertical ductility is better. The strength difference is highly related to the as-built crystallographic texture since different critical shear stress is required depending on the loading condition. A large lattice rotation to support high ductility in the vertical test is found in each of the testing variations mentioned above. The vertical texture evolution is similar to the deformation mechanism of single crystals, yet the horizontal lattice rotation behavior is close to polycrystals.
- In the slow strain rates testing, deformation twinning is only discovered in the vertical tests, which occurs mostly in the twinning favorable $\langle 111 \rangle // \text{LD}$ oriented grains, and it is beneficial for the vertical ductility to remain at high level even under creep damage.

CHAPTER 7

Future work

The licentiate thesis is a halfway examination of the entire PhD research, and the plans for the next stage are proposed as follows.

- **In-situ neutron diffraction study**

It will be a continuous study on the anisotropic deformation mechanism at 700 °C under the slow strain rate conditions, 10^{-5} s^{-1} and 10^{-6} s^{-1} . In-situ neutron diffraction is planned to be carried out on the sample during tensile testing, and the continuously recorded diffraction at evolving strain level is expected to give a better understanding of lattice rotation and deformation twinning process. A TEM work on the deformed sample is planned to investigate the dislocation movement, hence, a study with the combination of different scales will give a clearer picture of deformation mechanisms. Furthermore, it can be a good reference study for low cycle fatigue study and mechanical behavior simulation with the respect to crystal plasticity in the future.

- **Fatigue behavior of LPBF stainless steel 316L with tubular structure**

This is an on-going research on the low cycle fatigue behavior of LPBF stainless steel 316L. To reveal the thin-walled effect on the fatigue behavior, tubular fatigue specimens with different thicknesses of 1mm and 2mm and rough as-built surface are made. Two reference groups will be carried out, one is tubular fatigue specimen with polished surface, and another one is regular cylindrical fatigue bar. The fatigue deformation mechanism of LPBF stainless steel 316L will be studied with a comparison to conventionally manufactured stainless steel 316L.

From the point of view of functionality, the primary concern of engineering materials is the mechanical properties, especially the fatigue and creep properties since they determine the life time of a component and further influence the life cycle of an entire application. These two properties have been studied for a long time, but the knowledge in LPBF materials is very limited. Therefore, it motivates this PhD research to dig in the fatigue and creep behavior of LPBF materials based on the knowledge of monotonic tensile behavior in this Licentiate thesis.

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Part II

Publications

Papers

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