



PONTIFICIA UNIVERSIDAD CATOLICA DE CHILE
ESCUELA DE INGENIERIA

EXPERIMENTAL RESEARCH OF LASER SURFACE MELTING EFFECTS ON ANNULAR DISCS OF STAINLESS STEEL 316L USING RADIAL AND CARTESIAN SCANS

MATÍAS FELIPE GONZÁLEZ AGUILERA

Thesis submitted to fulfill the requirement for the Degree of
Master of Science in Engineering

Advisor:

JORGE RAMOS GREZ

Santiago de Chile, June 2022

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To my family

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ABSTRACT

Laser is one of the most used tools in current manufacturing for various processes such as cutting, welding, additive manufacturing, marking, and surface texturing, among others. Its direct use on metals allows treatment of localized surfaces without affecting nearby material or the piece's body. Depending on the expected result, laser surface treatments are classified into hardening, texturing, alloying, coating, and melting. Using the latter is possible to modify the microstructure, dissolve precipitates, and increase hardness and wear resistance, among other applications depending on the material and the parameters used. In this work, it is proposed to empirically study the Laser Surface Melting (LSM) technique on 316L stainless steel annular discs to evaluate the influence of laser scanning patterns on the results of the laser treatment. The considered scanning patterns correspond to a Cartesian, the most used pattern in the LSM process, also known as “zigzag”, and a Radial pattern, previously proposed in Additive Manufacturing research, which moves the laser in a cylindrical way. The main contribution of this work is to introduce the variable of the scanning pattern in LSM studies, besides generating a complete characterization of laser treatment on 316L stainless steel annular discs. The results show that, under the same conditions, the variation of the scanning pattern produces a meaningful change in the stress state of the specimen and its deformation after treatment. The main results of LSM treatment were an increase in crystallite size and a decrease in dislocation density, a state of compression and slight hardening in the melted layer, and a decrease in toughness. This knowledge can be used to encourage new scanning patterns in this field. In addition, it allows a qualitative approach in similar laser applications such as additive manufacturing, layer remelting, and indirect laser brazing.

Keywords: Scanning strategies, Laser Surface Melting, radial pattern, annular discs, laser additive manufacturing.

RESUMEN

El láser es una de las herramientas más utilizadas en la manufactura actual, para procesos varios como corte, soldadura, manufactura aditiva, entre otros. Su utilización directa sobre metales permite un tratamiento de superficies localizado, sin afectar material cercano ni al cuerpo de la pieza. Según el resultado esperado, los tratamientos laser de superficie se clasifican en endurecimiento, texturizado, aleación, revestimiento y fundido. Este último permite modificar la microestructura, disolver precipitados, incrementar dureza y resistencia al desgaste, entre otras aplicaciones según material y los parámetros utilizados. En este trabajo, se propone estudiar empíricamente la técnica de Laser Surface Melting (LSM) sobre un acero inoxidable 316L, considerando el patrón de escaneo como factor influyente en los resultados del tratamiento laser. Los patrones de escaneo considerados corresponden a uno Cartesiano, típicamente utilizado en LSM, también conocido como zigzag, y un patrón Radial, propuesto previamente en investigaciones de Manufactura Aditiva, recorriendo la superficie en forma cilíndrica. La principal contribución del presente trabajo es introducir la variable del patrón de escaneo en los estudios de LSM, además generando una completa caracterización del tratamiento con láser sobre discos anulares de acero inoxidable 316L. Los resultados muestran que, en iguales condiciones, la variación del patrón de escaneo produce un cambio significativo en el estado de tensiones de la muestra y en su deformación luego del tratamiento. Los principales resultados del tratamiento fueron un aumento del tamaño de cristalito y disminución de la densidad de dislocaciones, un estado de compresión y leve endurecimiento en la capa fundida, y la disminución de tenacidad. Este conocimiento puede ser utilizado para incentivar el uso de distintos patrones de escaneo en el área. Además, permite un acercamiento cualitativo en aplicaciones laser similares como en manufactura aditiva, refundido de capas y soldadura laser indirecta.

Palabras Claves: Estrategias de escaneo, Fusión de Superficie con láser, patrón radial, discos anulares, manufactura aditiva con láser.

1 INTRODUCTION

AISI 316L type austenitic stainless steel (ASS) is a common engineering material widely used as a structural material. It shows high resistance to corrosion and oxidation and good formability (Sarnek et al., 2017). ASS has a wide range of applications in various industries, such as the chemical, nuclear, marine, petrochemical, and electronics industries. However, it possesses low low-yield strength and low wear resistance. 316L ASS has been constantly researched to reduce its weakness, for example, using advanced thermomechanical processes and surface mechanical attrition treatment to obtain ultrafine-grained and nanocrystalline structures (Fu et al., 2020).

On the other hand, tubular or pipe structures are widely utilized in various applications and industries, for example, in automotive for crash boxes or bumper bars, for energy absorption, and to reduce the weight of the structures (Bracq et al., 2021). Annular discs have been studied in mechanical and medical areas. Some applications were multi-disc clutch, artificial discs for prosthesis, transmission systems, and, in general, there are circular components and annular discs in many machining devices.

Concerning surface treatments, a laser heat source has been highly applied due to its ability to focus the laser on-demand, avoiding affecting a surrounding material and the bulk properties. Laser surface modification is the physical and chemical modification of the surface condition using diverse types of lasers (Jeyaprkash et al., 2020). Among the more popular are the high-power Neodymium Yttrium-Aluminum-Garnet (Nd: YAG) laser and carbon-di-oxide (CO₂) laser, which can be used in pulsed or continuous wave mode with different powers, velocity, and distance between laser paths. Currently, fiber lasers have replaced CO₂ lasers due to their higher absorptivity and power density, resulting in minimal distortion of the original sample.

The laser surface modification has the purpose of hardening and/or improving corrosion and wear properties of the surface. Currently, there exist five kinds of laser surface treatment: Laser surface hardening (LSH), Laser surface melting (LSM), Laser surface texturing (LST), Laser surface alloying (LSA), and laser cladding (LC).

LSM aims to melt the surface through a high-power laser beam, thereafter, solidifying quickly. Due to the formation of a melting zone, the surface microstructure is changed by increasing or decreasing the grain size, depending on the alloy composition, and/or dissolving precipitates. In other words, the purposes for which LSM is used are as diverse as the materials and applications can be. Vilchez et al. (2020) proposed using an LSM with continuous-wave Yb: YAG fiber laser ($\lambda = 1070 \text{ nm}$) as post-processing of a welded joint of SS 316L to relieve the residual stresses and dissolved carbides precipitate in the welded region (Vilchez et al., 2020). They used power of 200W and scan velocity of $10 \text{ mm}\cdot\text{s}^{-1}$, reaching a depth of melt of $120 \text{ }\mu\text{m}$. The purpose was to improve the surrounding area and the molten zone to avoid preferential corrosion. Moradiani et al. (2022) treated the surface of AISI D2 steel to improve its wear performance by using the LSM with an Nd: YAG pulsed laser with a mean power of 400W. The diameter of the laser was not reported. They achieved stabilization of retained Austenite inside the molten zone with a dendritic structure, increasing the wear resistance to the double compared to the conventional heat-treated samples. The latter was attributed to the retained austenite transformation to martensite during the wear test (Moradiani et al., 2022). It should be noted that the laser depth effect was between 25 to $50 \text{ }\mu\text{m}$. However, some authors have reported achieving a variable depth according to the laser parameters used. Chaurasia et al. (2021) using a fiber laser reached up to $450 \text{ }\mu\text{m}$ of depth, with 500W and $100 \text{ mm}\cdot\text{s}^{-1}$ in Inconel 625.

A frequently overlooked variable in LSM studies is the laser scan pattern. It refers to which paths on the object's surface will follow the laser beam to produce the treatment, and it has been strongly considered in other sectors with similar laser uses or energy source

movement, as in additive manufacturing (AM). Numerous studies of scanning patterns have been developed in Selective Laser Melting, one of the main techniques of AM (Hajnys et al., 2020; Jhabvala et al., 2010; Kruth et al., 2010). The research indicates that the orientation of the grains is highly dependent on the scanning speed, scanning pattern, and the local part geometry. Hence scanning strategy can be considered a powerful tool to control the microstructural texture (Kruth et al., 2010). Due to the similarity with the LSM process, it is suitable to consider that different scanning patterns could determine the quality results in LSM.

Another sector that uses LSM is Indirect laser brazing, where the modifications on the surface, such as increasing roughness and the formation of a thicker oxide layer, increase the total laser absorbed by the material, resulting in higher energy efficiency during the laser brazing process (Zaifuddin et al., 2020).

A recent study has used non-conventional laser paths in LSM (X. Wang et al., 2022). Wang et al. (2022) applied a laser heat source to induce thermal stress, shaping annular discs of 1.2 mm thickness into bowls with radial heating paths. It was found that deformation increases with laser power and decreases with heating lines longer than an optimum length of about halfway between the inner and outer radius. Other laser radial paths could be found in the work of Hauser et al. (2005). They proposed a technique for additive manufacturing based on spiral growth manufacturing, where the laser radial movement is continuous and in polar coordinates, which is thermally efficient when producing cylindrical parts since the laser would not move too far away from a point (Carter et al., 2020; Hauser et al., 2005).

This study provides quantitative evaluations of the influences in LSM of cartesian scan pattern compared to a radial pattern, presenting the modifications produced by these treatments on 316L stainless steel thin annular discs. The specific cartesian pattern used is the Meander scan, which is the most common pattern used in LSM, also called as

“zigzag”. The radial scan pattern is the same used in Hauser et al. (2005) and in the work of Zañartu & Ramos (2010) to move the binder fluid used. The effects of both scan patterns over the surface were characterized mechanically by residual stresses, tensile strength, microhardness, roughness, and studying their microstructure.

Knowing the different consequences of using radial and cartesian laser scans pattern, for example, in residual stresses or deformation, would not only allow improvement in LSM treatments through better predictions but it could be a complement to studies that propose cylindrical/spiral laser scan patterns and a continuous process in AM or be advisable about using specifics scanning patterns in the indirect laser brazing to improve the results.

Chapter 2 shows the background compilation and details of the setup for the experimentation. A preliminary study developed as the background is presented in Chapter 2. Chapter 3 corresponds to the main investigation. The article “Effects of laser surface modification on Stainless Steel 316L thin annular discs: radial vs. cartesian laser scan strategies”, submitted to an International Scientific Indexing (ISI) journal, is presented. That chapter details the methodologies, the microstructure and mechanical characterization, the results of the different properties evaluated, and the discussion. Finally, Chapter 4 provides the global conclusions of this study and recommendations for future work. Annexes with detailed research results are also included.

1.1 Hypothesis

- The laser scan pattern in the process of Laser Surface Melting plays a significant role in the mechanical properties of the samples, such as tensile strength, surface roughness, and microhardness.

- Under identical conditions, the radial scan pattern produces 25% less residual stress than the cartesian pattern, based on its constant and mostly smaller value of laser path length compared to cartesian scanning paths.
- The non-constant distance between two laser lines in a row of the radial scan pattern produces observable differences in microstructure compared to the cartesian scan pattern.

1.2 Objectives

This study has two main objectives: (i) To estimate the mechanical changes in 316L samples with laser surface modification with laser scans cartesian and radial, and (ii) to assess the principal differences between a laser surface melting produced by a cartesian and a radial scan pattern. Specific objectives and methods are the following:

- To establish the variables and optimal parameters for the investigation through the development of a preliminary study
- To determine the microstructural changes produced by the LSM and identify the differences using a cartesian vs. a radial scan pattern, through the characterization of the microstructural evolution of the samples studied using Optical microscopy, FESEM, and XRD analysis.
- To establish the difference in crystallite size and microstrain between annular discs treated with LSM, evaluating the effects of laser melting and the different pattern used, through XRD analysis.

- To quantify the response of annular discs treated in a modified tensile test adapted to rings.
- To identify the stress state in samples treated with LSM by measuring residual stresses using ultrasonic waves.

2 BACKGROUND

2.1 Study case

The research field of this work corresponds to Laser Surface Modification on metals, which includes different techniques according to the objective proposed. In specific terms, the study corresponds to the application of Laser Surface Melting on AISI 316L annular discs, considering two levels of power, and two different laser scan patterns shown in Figure 2-1; a Cartesian pattern also known as zigzag or meander, and a Radial pattern proposed by Hauser et al. (2005), to manufacture objects with axis-symmetric geometry.

The present study is expected to show how much the scanning pattern impacts surface modification of the annular specimens. This variable has been well-established in additive manufacturing processes, but it has not been included in LSM fundamentals. As it has been observed, the scanning pattern can affect the relative density, especially the distribution of pores (Pauly et al., 2017), but also the residual stress. J. Wang et al. (2022) studied manufacturing with different scan patterns using direct energy deposition (DED) of Ti-6Al-4V. They researched the relationship between the scan pattern with the residual stress and deformation of the samples, finding that a decrease in the scan pattern length (also known as island size) caused a reduction in the residual stress. Additionally, they demonstrated great differences in residual deformation caused by laser jump patterns between these islands, proposing that the heat flow from outside to inside could have the best result regarding residual stress, also proposed by Yan et al. (2018). Indeed, several studies have indicated that spiral patterns have reached favorable results due to their more uniform and consistent surface heating and cooling (Jhabvala et al., 2010). Considering this latter observation, it is expected that a rotating spiral pattern will help address

thermally induced problems due to non-uniform heating and cooling (In Kim & John Hart, 2021).

The laser scan treatments mentioned above along with the additive manufacturing processes, are currently equipped to produce laser scans of the Cartesian type, i.e., in the XYZ direction. This type of motion in additive manufacturing has some challenges (Carter et al., 2020), such as dimension adjustment and contour consolidation when these are not straight. On the other hand, the polar coordinate pattern has been investigated in 3D polymer additive fabrication and other studies at the algorithm level. The polar coordinate combined with a spiral movement of the printing platform reduces manufacturing time or delays by allowing continuous printing and powder feeding (Hauser et al., 2005). The use of polar coordinates is thermally efficient when producing cylindrical parts since the laser would not move too far away from a point. However, no empirical studies demonstrate the mechanical differences between the two scan patterns, both Cartesian and Radial, neither in 3D part manufacturing nor laser surface modifications, where frequently the cartesian scan pattern is used without question.

This research aims to study the modifications laser surface treatment has on 316L stainless steel thin annular discs by scan patterns, as shown in Figure 2-1. The distance between paths, defined as “hatch distance”, is a variable strongly affected by the scanning pattern used. The hatch value is constant in cartesian scan cases, while it varies in the radial direction in the radial scan pattern. Another distinctive feature of the hatch in the radial scan pattern is the corners located at the edges, where the laser path changes in direction and hatch spacing is almost zero.

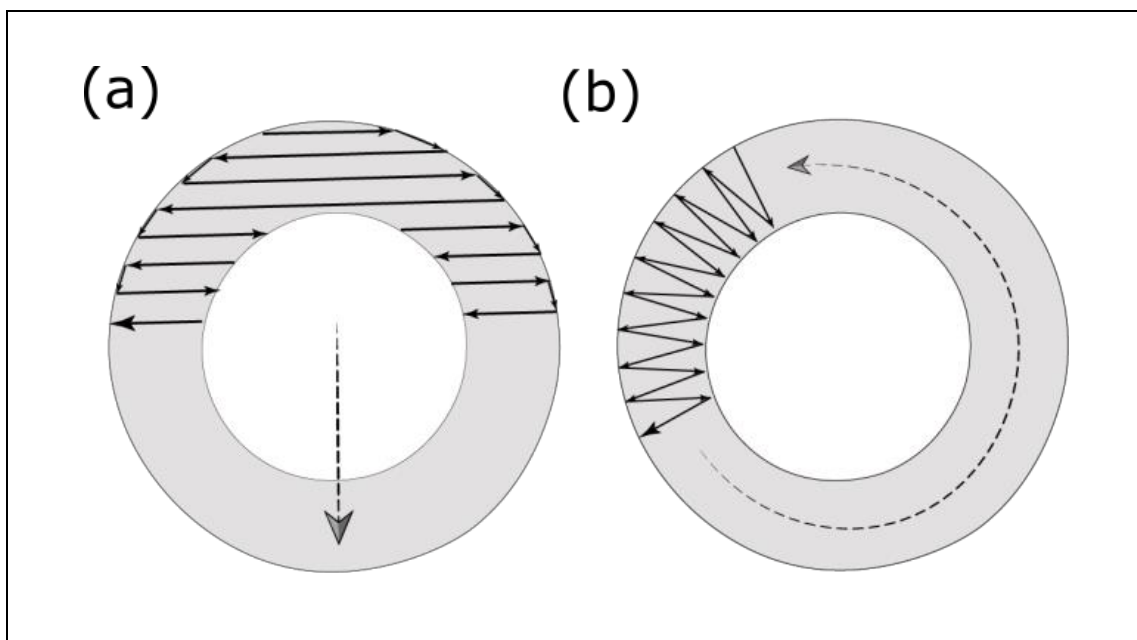


Figure 2-1: Representative scheme of the scan pattern applied to 316L annular discs. Solid lines show the laser paths (a) Cartesian scan (zigzag) and (b) Radial scan pattern.

A variable affected by the hatch is the energy density. The meaningful changes of hatch between paths for the radial pattern are expected to impact some properties in an LSM treatment significantly. Other variables that should produce differences are the thermal gradient and the path length, this latter is uniform in the radial scan pattern but not in the cartesian pattern.

2.2 Setup for experimentation

Laser surface melting was carried out using a ytterbium fiber laser, model YLR-300-MM-AC-Y11 by IPG Photonics, with 300 W peak power and 1070 nm emission wavelength. The full setup consists of the laser and a collimator; a beam expander BEST-1064-2.5X, a Marking Head at 1064 nm model LSJC-1064-20-8220 (which include galvo-scanner motor and drivers), and an F-theta lens model STY-1064-244-350, all last three by Sintec Optronics and finally; support for the samples. Different efficiency measurements and marking tests on laser engraving films were developed to identify the focal length. The laser beam diameter was estimated at $70 \pm 10 \mu\text{m}$. The laser melting was carried out in an opened chamber at room temperature.

2.3 Preliminary study

A preliminary study was performed to evaluate the effectiveness of the chosen parameters such as velocity, time of laser treatment, and sample thickness, among others. During this approximation, samples of AISI 316 were used as coins, and the same laser and setup developed the LSM as the main investigation. The nominal chemical composition of coin samples given by the supplier was (wt.%), 0.08% C, 2.0% Mn, 1.0% Si, 16-18% Cr, 10-14% Ni, 2.0-3.0% Mo, 0.045% P, and 0.03% S. The samples were obtained after cutting them from a rolled bar of 38.1 mm outer diameter, using wire electrical discharge machining (WEDM).

After cutting, samples were polished with P180 and P240 grit sandpaper to eliminate the superficial layer affected by WEDM, reducing their thickness by about 0.1 mm on one side. The coins thickness tested was between 0.5 and 2.0 mm.

These coins served as a preliminary study to determine the thickness ranges for annular disc samples studied in the main reseach and the laser velocity. The microstructural characterization method was developed in this preliminary study and replied then with 316L annular discs. The scan patterns used in the coin samples are shown in Figure 2-2. It is important to note that the LSM was applied on a central zone of 30 mm diameter, i.e., the laser did not melt the whole coin face, which allowed to observe the melting pool edge and differentiate it from the surface not treated in the external contour.

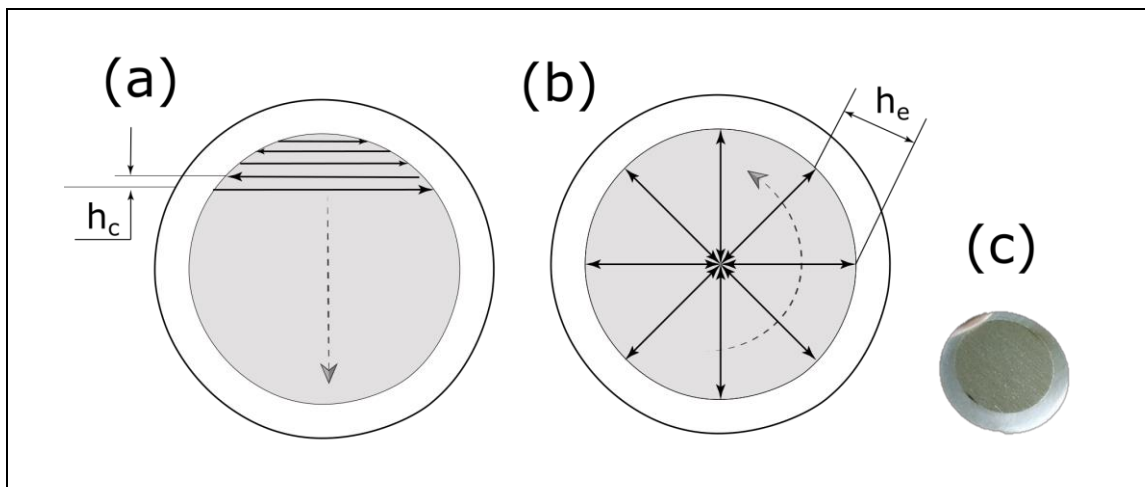


Figure 2-2: Representative scheme of the scan pattern applied to 316 samples (a) Cartesian scan and (b) Radial scan pattern (c) Picture of a coin treated.

Thickness less than 1 mm was tested during 1 minute of treatment, $0.6 \text{ m}\cdot\text{s}^{-1}$ laser velocity, and 250W. These coins show deformation, and the laser affected the reverse face. In both scan patterns, the deformation in coins visually disappears after a 1.6 mm thickness.

Then, two laser velocities (0.6 and $1 \text{ m}\cdot\text{s}^{-1}$) were proved with 280W. The thickness selected was 1.8mm, and those samples did not show deformation. The samples treated with 280W and $0.6 \text{ m}\cdot\text{s}^{-1}$ laser velocity was mounted in Bakelite, leaving the cross-section free for viewing. The cut before mounting the sample was developed using WEDM. To disappear the effects produced during this cutting process and get able to reveal the microstructure, the samples mounted in Bakelite were successively grinded with 400, 600, and 1200 grit sandpaper, then polished with a $6 \mu\text{m}$ Al_2O_3 suspension. The solution used to reveal the microstructure was Aqua regia (30 ml of distilled water, 20 ml of HCl, and 15 ml of HNO_3). Figure 2-3 shows the optical microscopy images obtained from these samples.

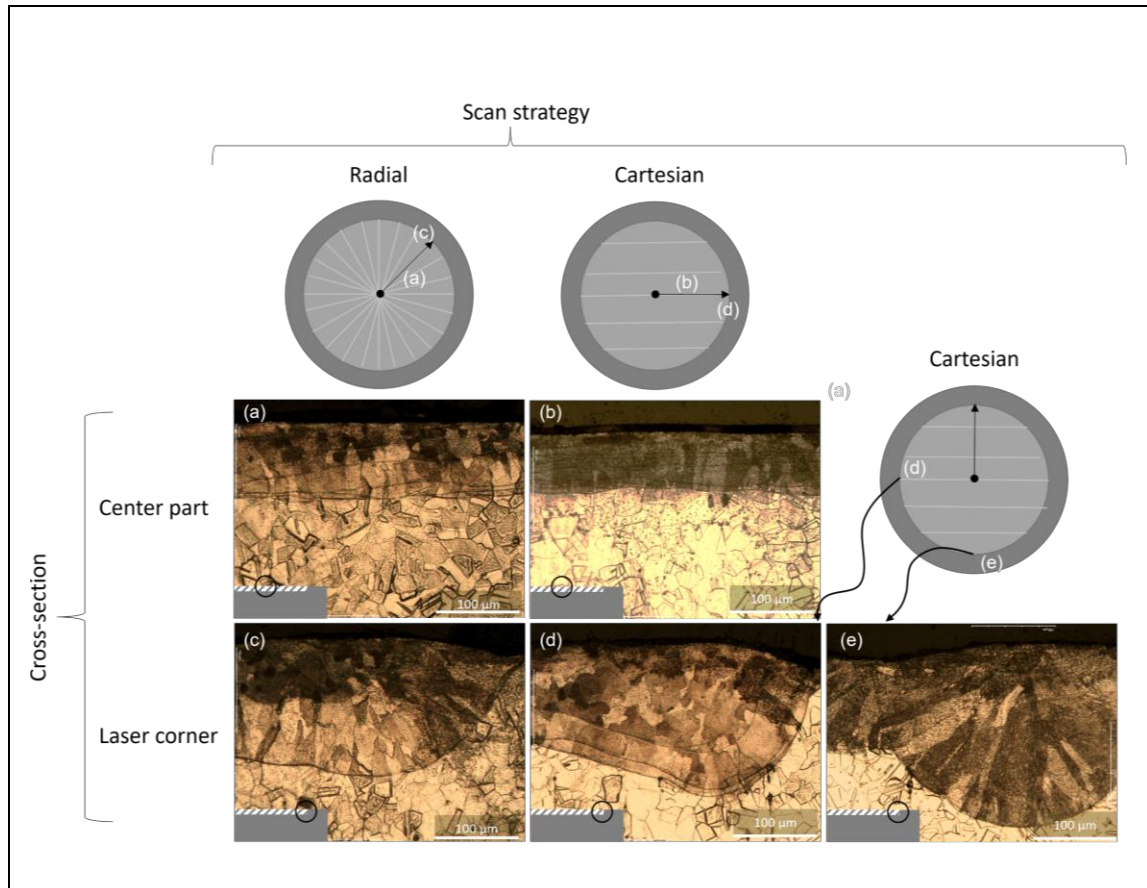


Figure 2-3: Optical microscopy images of the cross-section showing the microstructure after laser application (a, c) radial scan pattern and (b, d) cartesian pattern.

In the corner of each image of Figure 2-3 is a coin representation to identify where each is from. Figure 2-3 (a,c) correspond to radial scan pattern. The rest correspond to the cartesian scan pattern. Figure 2-3e was obtained from a perpendicular cut to the laser path, whereas the other images were obtained from a parallel cut to the laser path. Figure 2-3 (a, b) shows ripples, associated with the overlapping of the following paths. Figure 2-3 (c,

d, e) shows a pool on the edge of the laser treatment, where the laser reduces its velocity to produce the direction change.

After the mounting, microhardness testing Vickers HV was performed using a Leco M-400-H with a test load set to 2 N. The measurements were conducted on a cross-section in-depth, from the surface up to 1050 μm , approximately. The microhardness evolution is shown in Figure 2-4.

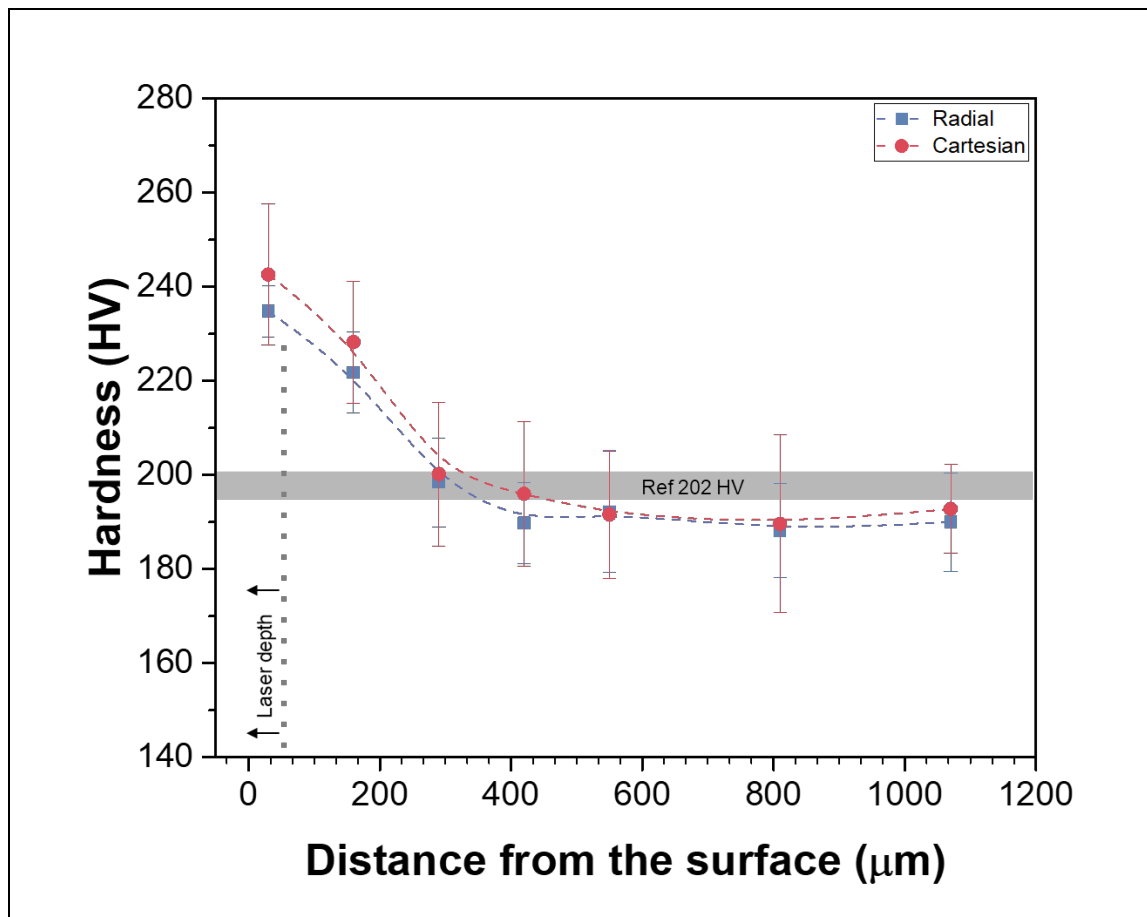


Figure 2-4: Measurement of microhardness evolution in the depth of coins cross-section.

As shown in Figure 2-4, the laser, independent of the pattern type used, modifies the surface properties, raising the hardness of the material to approximately 300 microns from the base. The hardness increases are due to the microstructural changes produced in the material after laser treatment, where there was a newly solidified microstructure. The formation of precipitates at the surface is expected, possibly associated with carbides.

The increased microhardness observed on the coins treated with laser and the formation of new phases motivated the development of an XRD analysis to identify those new phases in the melted zone. Due to economic and time limitations, this analysis was applied only to annular discs (Chapter 3).

Table 2-1 summarizes the measurements obtained from laser-treated coin samples. Each Hardness value corresponds to the measured point closest to the surface. The greatest depth for the radial scan pattern could be due to its lower hatch value, especially at the center. The greatest depth on edge in both scanning patterns, which can be observed in Figure 2-3 (c,d,e), may be related to the reduction of the laser velocity due to the change in laser direction. These depth values are exposed in Table 2-1 as Edge Pool Melted Region,

Table 2-1: Results in terms of hardness and roughness of coin samples after laser surface modification with laser velocity of $0.6 \text{ m}\cdot\text{s}^{-1}$ and 280W power.

Sample	Melted Region (μm)		Hardness, $\text{HV}_{0.2}$	Surface Roughness (μm)	
	General	Edge Pool		X	Y
Control	-	-	197 ± 6	0.10 ± 0.01	0.10 ± 0.03
Cartesian	82 ± 5	193 ± 23	248 ± 16	5.08 ± 0.68	1.24 ± 0.20
Radial	66 ± 17	159 ± 2	233 ± 6	3.53 ± 1.22	2.50 ± 0.86

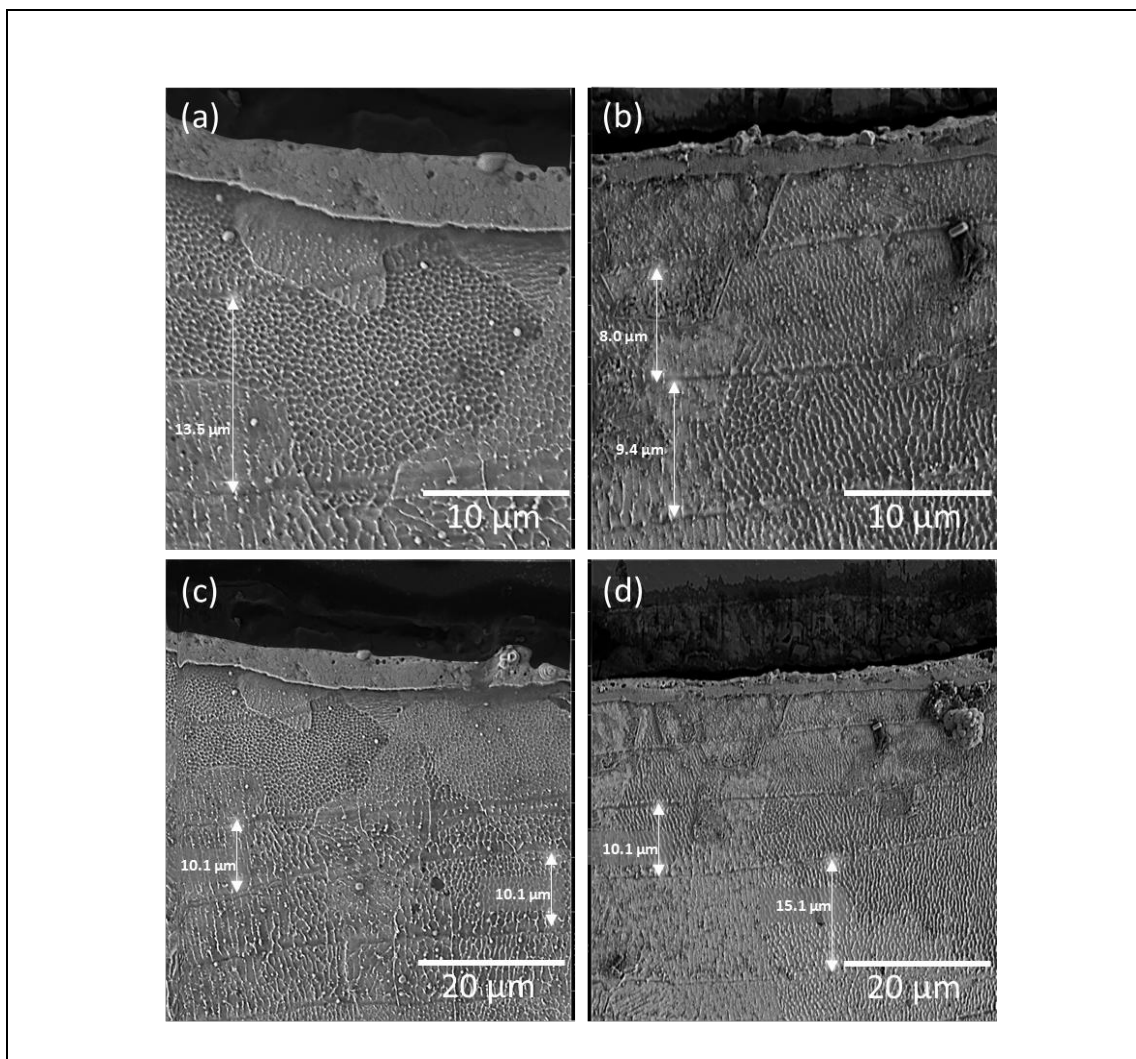


Figure 2-5: FESEM images of the cross-section of laser affected zone with (a, c) radial scan pattern and (b, d) cartesian pattern

The center part of the cross-section localized about 15 mm from the coin's center to the edge, where observed with FESEM. The images obtained showed the texture produced by the laser, the ripples, and a thin layer of iron nitrides in the outermost layer.

Two main observations of the preliminary study were the solidification ripples shown in Figure 2-5, produced by the overlapping and the high laser speed, and the melted pool formed in the external contour. The latter observation was associated with the deceleration of the laser movement to allow the change of direction. Considering the depth of the melted pool shown in Table 2-1, the radial scan pattern seems to minimize the affected zone in the edge, which could be useful in LSM treatment where the treatment does not cover the entire surface.

That pool effect was tested using a Concept Laser (CL) MLAB 200R unit by General Electric, designed for Additive Manufacturing processes. A Cartesian scan pattern was used, with the same laser velocity of samples ($0.6 \text{ m}\cdot\text{s}^{-1}$), but different drivers to control the laser movement, corresponding to the default machine drivers. Other different conditions consisted of a controlled atmosphere and 200W of power, the maximum value allowed by the machine. Finally, a similar melted pool was obtained, demonstrating that the edge of laser scans is a zone of power concentration on surface scanning. The melted depth of the edge pool was about $120 \text{ }\mu\text{m}$, while the rest of the melted zones did not reach $45 \text{ }\mu\text{m}$.

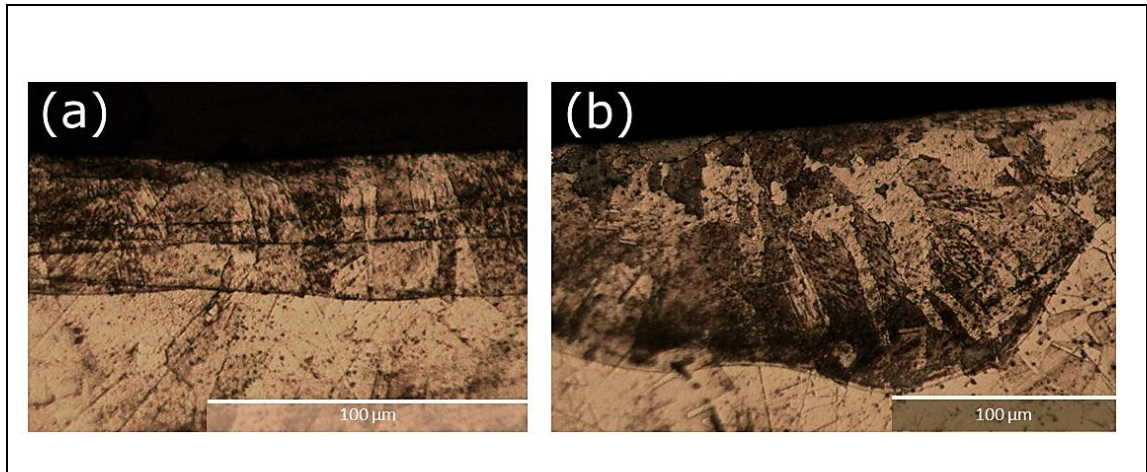


Figure 2-6. Optical microscopy images of the cross-section showing the microstructure after LSM using the cartesian scan pattern in CL machine (a) Center of the melted area
(b) Edge of the melted area

3 EFFECTS OF LASER SURFACE MELTING ON STAINLESS STEEL 316L THIN ANNULAR DISCS: RADIAL VS. CARTESIAN LASER SCAN STRATEGIES

3.1 Experimental Procedure

3.1.1 Materials

Stainless steel AISI 316L samples as annular discs were modified superficially by different laser patterns. The nominal chemical composition of the AISI 316L discs (wt.%), given by the supplier is 0.035% C, 2.0% Mn (max.), 1.0% Si (max.), 16-18% Cr, 10-15% Ni, 2.0-3.0% Mo, 0.04% P (max.) and 0.03% S (max.). Using wire electrical discharge machining (WEDM), the annular discs were obtained from a 56-25 mm outer-inner diameters rolled bar. After cutting, samples were polished on both sides with sandpaper of P180 and P240 grit to eliminate the superficial layer affected by WEDM, which does not exceed a depth of 0.1 mm in cutting faces (Sidhom et al., 2013). Hence, the thickness was reduced by about 0.2 mm with polishing discs. The final sample thickness corresponded to 1.75 mm.

3.1.2 Laser surface treatment

Laser surface melting was performed using a Ytterbium fiber laser, model YLR-300-MM-AC-Y11 by IPG Photonics, with up to 300 W peak power and 1070 nm emission wavelength. The full setup consisted of the laser and a collimator; a beam expander BEST-1064-2.5X, a Marking Head at 1064 nm model LSJC-1064-20-8220 (which include two

galvo-scanner motor and drivers), and an F-theta lens model STY-1064-244-350, all last three from Sintec Optronics. Finally, to support the samples, a holder was designed and built. The laser melting was carried out in an opened chamber and under ambient conditions.

As already mentioned, annular discs were surface-treated using two laser scanning patterns, cartesian and radial, both with a scan velocity of $0.6 \text{ m}\cdot\text{s}^{-1}$ and two laser power levels of 250 W and 280 W. The energy supply was applied for 1 minute. Therefore, the total energy supplied is the same for samples with 250W and samples treated with 280W.

The Marking Head was controlled with EzCad2 Software, which allows defining the movement of the laser during the melting. The cartesian pattern is a standard that can be selected in the software under the Hatch item. The radial pattern trajectory was created using Python and imported into EzCad2 as a .svg file.

The hatch space (h), defined as the distance between lines in a row or passes of the laser beam, for cartesian scans (h_{re}) was set at $55 \text{ }\mu\text{m}$, ensuring the presence of overlapping between laser paths in a row. This hatch value is constant for the cartesian pattern, while for radial scans, the hatch value varied between $33 (h_{ri})$ to about 0 and between $75(h_{re})$ to about $0 \text{ }\mu\text{m}$. These differences are shown in Figure 3-1. An average between the internal and external hatch in the radial pattern could lead to considering the mean for radial scans to be about $55 \text{ }\mu\text{m}$. However, the significant changes in the hatch in each path must be considered.

A variable affected by the hatch is the energy density. Due to the hatch changes in the radial scan, the energy density per unit area of this pattern is continually changing, while in the cartesian scan, this value is constant. Energy density supplied per area, considering a total efficiency, could be calculated in J/mm^2 by,

$$E_a = P / v h \quad (3.1)$$

Where 'P' is the power supplied in W, 'v' is the velocity of the laser in mm·s⁻¹ and 'h' is the hatch in mm. This expression loses its applicability in the corners, where the hatch space approaches 0 or at times where the velocity slows down to change its direction.

Considering the symmetry of the samples, the LSM was carried out on the two faces of the annular discs, waiting for the cooling of discs at room temperature between laser treatments on each face. Figure 3-1 shows the scan patterns applied to the annular discs. For discs treated with cartesian scan, the laser was applied to both faces with a rotation of 90° between sides. The straight arrows represent the upper treatment, whereas the discontinuous arrows show the scan pattern applied to the bottom part. For annular discs treated with radial scan, the pattern shown in Figure 3-1b was applied on both sides, but in this case, any rotation does not generate a major difference between faces.

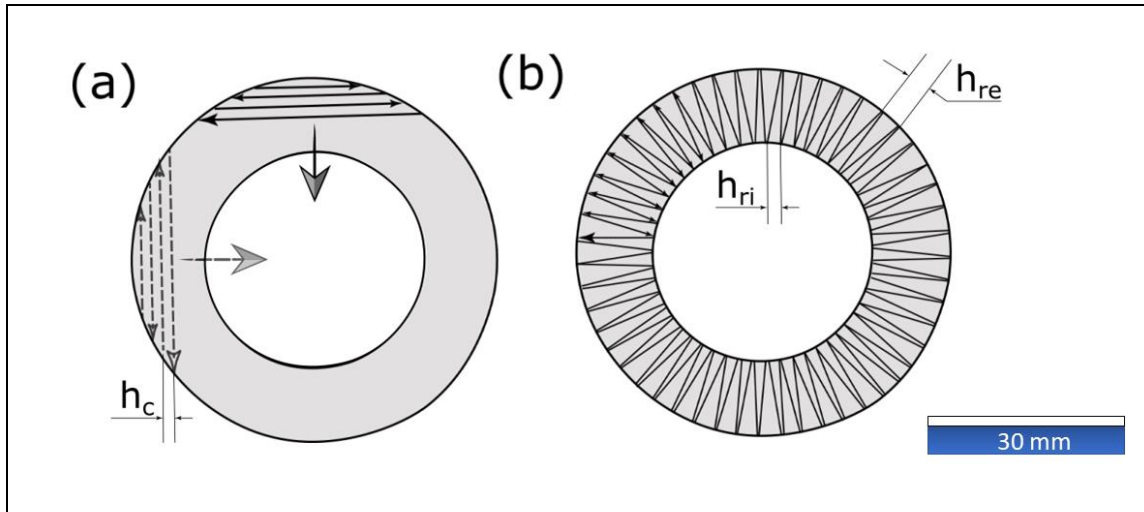


Figure 3-1: Representative scheme of the scan pattern applied to 316L samples. (a) Cartesian scan and (b) Radial scan pattern.

The parameters used in each experiment are summarized in Table 3-1, using a sample ID for each configuration. The configurations were performed in triplicate, which allowed statistical analysis of the significance of the resulting mechanical properties, considering power and scan patterns as independent factors.

Table 3-1: Summary of laser parameters used over discs samples at $0.6 \text{ m}\cdot\text{s}^{-1}$ laser velocity for 1 minute, keeping the energy given to samples constant.

Config	Power Laser (W)		Scan Pattern (-)	
	250	280	Cartesian	Radial
S1		✓	✓	
S2		✓		✓
S3	✓		✓	
S4	✓			✓

Figure 3-2a shows an annular disc treated with an S1 configuration. Figure 3-2b an annular disc treated with S2 configuration, i.e., using both scanning patterns presented in Figure 3-1, with the power laser of 280 W.

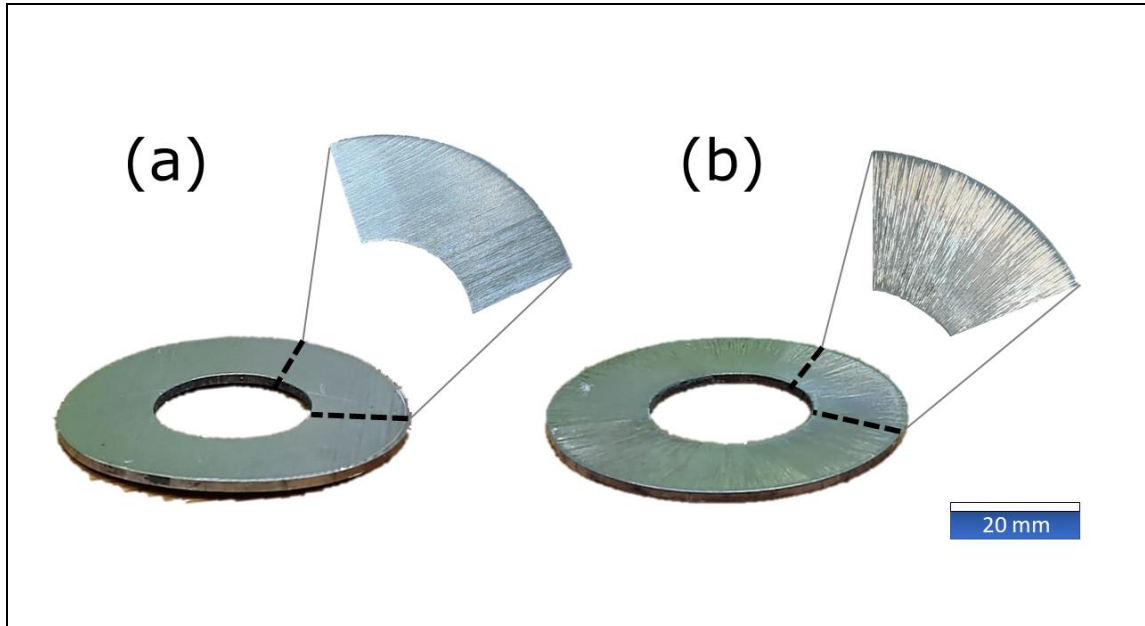


Figure 3-2: Pictures of annular discs treated with different scan patterns
(a) Cartesian scan and (b) Radial scan pattern.

3.1.3 Microstructure characterization

After laser treatment, the AISI 316L annular discs were cut lengthwise using WEDM and then mounted in bakelite, leaving the cross-section free for viewing. The affected zone produced by the cutting with WEDM is eliminated through a polished process. The samples were successively grinded with 400, 600, and 1200 grit sandpaper, then polished with a 6 μm Al_2O_3 suspension. Aqua regia solution (30 ml of distilled water, 20 ml of HCl , and 15 ml of HNO_3) was applied for 7 seconds to reveal the microstructure. Optical microscopy of the Department of Mechanical Engineering and Metallurgy (Nikon OPTIHOT-100 microscope) was used to capture the microstructure, and field emission-

scanning electron microscope (FESEM) images were also obtained (Quanta FEG 250, Thermo Fisher Scientific, Waltham, MA, USA) using secondary electrons. The latter was developed in the Physics Department of the Pontifical Catholic University of Chile.

3.1.4 Mechanical characterization

3.1.4.1 Roughness and hardness

Before and after laser surface treatment, surface roughness measurements were carried out on the samples using a roughness tester TIME TR110 model device (Time Group Inc., Beijing, China). The cut-off wavelength was between 0.8-2.5 mm and a speed of 1 mm·s⁻¹, as indicated by the International Standard ISO 4288. Five measurements were taken per configuration.

Microhardness testing Vickers HV was performed using a Leco M-400-H with a test load set to 2 N, which included an objective lens to see and measure the indentation dimensions. The measurements were carried out on discs cross-section in-depth, from the surface up to 550 µm approximately.

3.1.4.2 Tensile strength

A tensile test on the annular disc samples was performed with a uniaxial tensile test Instron machine, model 4206, 15 kN, and deformation rate 2 mm·s⁻¹. The tensile system was

adapted to use half discs of 25 mm in diameter as grippers, capable of distributing the load along the inside diameter of the specimen (Figure 3-3b). The state stress produced by the device allows a fracture to occur in the traction zone, as indicated in Figure 3-3a as FZ (fracture zone).

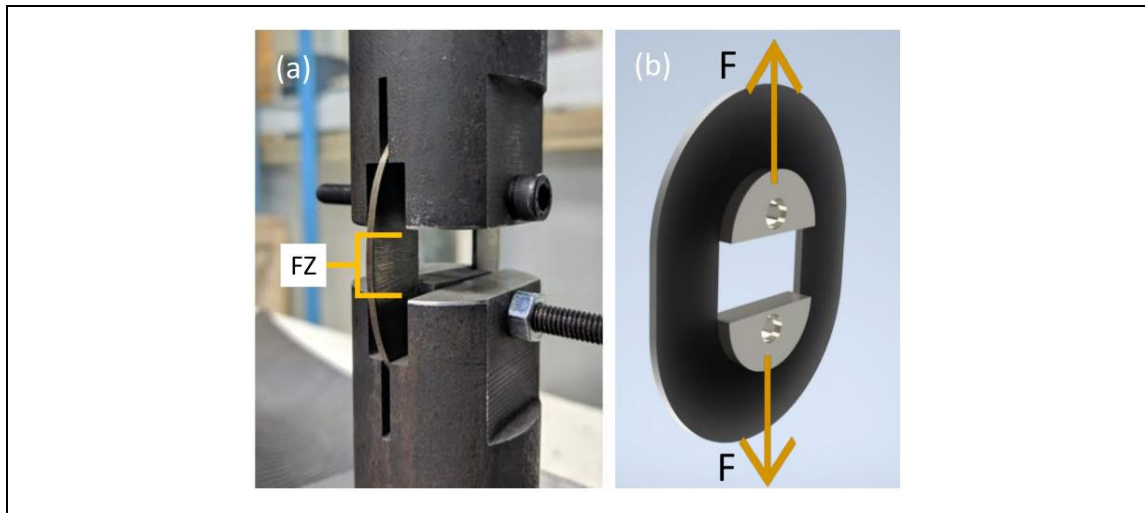


Figure 3-3: Real and schematic images of the tensile system adapted to the Instron machine. (a) disc during the tensile test with the fracture zone (FZ) indicated and (b) a schematic representation of the half coins coupled to the disc.

3.1.4.3 Residual stresses with Ultrasonic technique

The time of flight of longitudinal waves and ultrasonic shear waves was measured in five 316L stainless steel discs. One under a stress-free condition, corresponding to the control sample without laser treatment, two under a Cartesian treatment pattern (S1, S3), and two

under a Radial treatment pattern (S2, S4). Measurements were made at three positions on each disc.

Once the flight times were measured, the Poisson's ratio was obtained at each of the measurement points. After the Taylor series expansion analysis, the residual stresses in the material were obtained (Carvajal, 2016).

The method presented by Carvajal L. (2016), is based on the equation that relates Poisson's ratio to the ratio between ultrasonic longitudinal and shear wave velocities and the first-order treatment of the acoustoelastic effect. The directly additive effect of stress components in Poisson's ratio allows the determination of absolute values in a material subjected to a state of plane stress only by measuring the flight time of longitudinal and shear waves with normal incidence.

A Panametrics Model 5077PR ultrasonic pulse generator and a TiePie model Handyscope HS6-1000 oscilloscope to generate the waves and measure the time of flight, with a sampling frequency of up to 1 GS/s, were used. The pulse-echo method was selected along with the thickness in all points used to characterize the specimens. A 5 MHz Panametrics model V406 transducer with a diameter of 12 mm was used for the longitudinal waves. In the case of shear waves, a Panametrics transducer model V155 of 5 MHz, with a diameter of 12 mm, was used.

Figure 3-4 shows the normal incidence of ultrasonic waves in an isotropic, homogeneous, semi-infinite, and stress-free medium of thickness d .

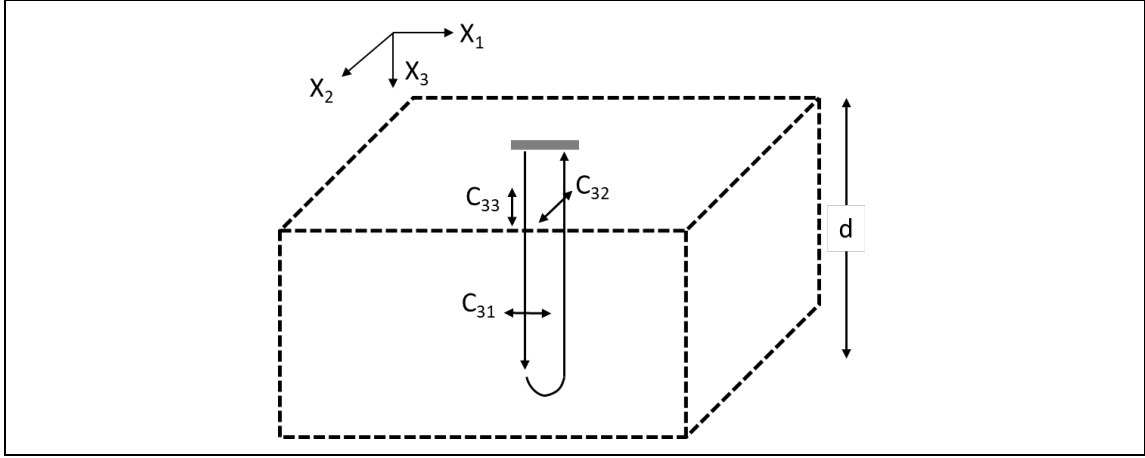


Figure 3-4: The normal incidence of ultrasonic waves in an isotropic medium.

The main expression to calculate the Poisson's ratio (ν) is shown in equation 3.2, where $c_L = c_{33}$ is the speed of the longitudinal wave and $c_T = c_{31} = c_{32}$ is the speed of the polarized shear wave along x_1 or x_2 .

$$\nu = \frac{(c_L/c_T)^2 - 2}{2[(c_L/c_T)^2 - 1]} \quad (3.2)$$

If t_L y t_s are the times of flight of the longitudinal and shear waves, respectively, traveling the same distance d , the Poisson's ratio can be obtained only by measuring times of flight, having:

$$\nu = \frac{(t_s/t_L)^2 - 2}{2[(t_s/t_L)^2 - 1]} \quad (3.3)$$

If ν is determined in a medium under stress σ_1 along x_1 , and depending on the polarization of the shear wave, the following equations are obtained:

$$\nu_{31}(\sigma_1) = \frac{(c_{33}/c_{31})^2 - 2}{2[(c_{33}/c_{31})^2 - 1]} = \frac{(t_{31}/t_{33})^2 - 2}{2[(t_{31}/t_{33})^2 - 1]} \quad (3.4)$$

and

$$\nu_{32}(\sigma_1) = \frac{(c_{33}/c_{32})^2 - 2}{2[(c_{33}/c_{32})^2 - 1]} = \frac{(t_{32}/t_{33})^2 - 2}{2[(t_{32}/t_{33})^2 - 1]} \quad (3.5)$$

The acoustoelastic constants K_{ij} , relate the components of tension σ_i, σ_j and σ_k to the change in speed that they produce and, therefore, to the time of flight. Assuming known acoustoelastic constants, the two previous equations allow the determination of $\nu_{31}(\sigma_1)$ and $\nu_{32}(\sigma_1)$ for a known value of stress σ_1 along the x_1 axis, having:

$$\nu_{31}(\sigma_1) = \frac{\left(\frac{(1 + K_{22}\sigma_1)}{(1 + K_{21}\sigma_1)} \right)^2 \left(\frac{t_S}{t_L} \right)^2 - 2}{2 \left[\left(\frac{(1 + K_{22}\sigma_1)}{(1 + K_{21}\sigma_1)} \right)^2 \left(\frac{t_S}{t_L} \right)^2 - 1 \right]} \quad (3.6)$$

And,

$$\nu_{32}(\sigma_1) = \frac{\left(\frac{(1 + K_{22}\sigma_1)}{(1 + K_{23}\sigma_1)} \right)^2 \left(\frac{t_S}{t_L} \right)^2 - 2}{2 \left[\left(\frac{(1 + K_{22}\sigma_1)}{(1 + K_{23}\sigma_1)} \right)^2 \left(\frac{t_S}{t_L} \right)^2 - 1 \right]} \quad (3.7)$$

Now, if ν it is determined in the same medium subjected to a stress state, two different values should be expected, depending on the polarization of the shear wave.

It can be shown that these equations represent a linear relationship between stress and Poisson's ratio, so to solve the inverse problem of evaluating stress from measured Poisson's ratio values, we apply Taylor series expansions of $\nu_{3j}(\sigma_i)$ about $\sigma_i = 0$, having:

$$\nu_{31}(\sigma_1, \sigma_2, \sigma_3) = \nu + P_{11}\sigma_1 + P_{12}\sigma_2 + P_{13}\sigma_3 \quad (3.8)$$

And,

$$\nu_{32}(\sigma_1, \sigma_2, \sigma_3) = \nu + P_{21}\sigma_1 + P_{22}\sigma_2 + P_{23}\sigma_3 \quad (3.9)$$

Each P_{ji} is the acoustoelastic coefficient that relates σ_i to $\nu_{3j}(\sigma_i)$ and is obtained by propagating longitudinal and shear waves perpendicularly to the uniaxially applied stress σ_i . The P_{ji} value depends on the acoustoelastic constants K_{ij} , the specific expressions are exposed in Annex 1.1.

For a plane stress state ($\sigma_3 = 0$) equations 3.8 and 3.9 represent a linearly independent system of equations so that if the P_{ji} and ν of the stress-free material are known, the stress can be determined by direct measurement of the times flight of waves in the stressed material.

The K_{ij} values used were calculated from a hot-rolled structural steel ASTM A-36 during the validation of the method (Solis, 2010). The acoustoelastic constants are shown in Table 3-2. The ν used was calculated with equation 3.3 and times measured from control annular discs. It was considered a planar state ($\sigma_3 = 0$), hence the P_{13} , K_{11} and K_{12} are unnecessary, and a slightly orthotropic medium, which means that is expected different values for ν_{31} and ν_{32} .

Table 3-2: The values of acoustoelastic constants used to calculate residual stress with the ultrasound method.

	Mean (GPa ⁻¹) x 10 ⁻³	Error (GPa ⁻¹) x 10 ⁻³
K_{22}	-1.56	0.05
K_{21}	-23.0	3.0
K_{23}	-1,48	0,07

3.1.4.4 X-ray diffraction analysis

X-ray diffraction (XRD) is a versatile non-destructive technique used to characterize the crystallographic structure of materials. It can provide detailed information about lattice parameters of crystals, phases, texture, or stresses. Ganesh et al. (2015) showed that full width at half-maximum (FWHM) and intensity variation of the diffraction peak are two sensitive parameters for the characterization of surface microstructure. Besides, the FWHM of diffraction peaks is modified when stress-strain accumulation varies in the material (Vashista & Paul, 2012).

The XRD analysis was done on control, cartesian, and radial annular discs after LSM under 250W. The XRD machine was a Miniflex 600 by Rigaku with a copper anode. Thus the wavelength for the X-rays was 1.54059 (Å) for K α 1 emission and 1.54441 (Å) for K α 2. The angle sweep (2θ) was performed from 15° to 140°.

For the XRD analysis, Rietveld refinement was performed with Match! Software version 3, fitting the peaks with a pseudo-Voigt function. With the same software, the K α 2

emission lines of the X-ray source were removed to avoid the doublet structure on peaks, which hampered the XRD analysis (de Rooi et al., 2014). Before the refinement, also the background of data was removed.

Using Bragg's law and identifying the Miller plane related to the peaks, it is possible to calculate the lattice parameter. For AISI 316L, the specific ordering form is Austenite, also known as gamma iron (γ). The crystalline structure of austenite is of the face-centered cubic type, which means the lattice parameter has a unique value. This value was obtained after Rietveld refinement, using the Match! 3 software.

Two quasi quantitative methods were used to compare crystallite size and microstrain between the samples. The first used the Scherrer formula (volume-weighted mean column length method), assuming the FWHM(w) is all related to the crystallite size (β_D equals w). The second was the Williamson-Hall (W-H) method, which allows identifying the component of strain (β_S) of the total FWHM, then $\beta_D + \beta_S$ equals w . In both methods, it was assumed that the size-broadened and strain-broadened profiles are pseudo-Voigt functions. The fitting was developed in Match! 3 software, applying a Rietveld refinement. From the refinement, the FWHM(w), the angle center of the peak (x_c), and the amplitude of the curve (A) were identified.

Knowing the FWHM, it is possible to calculate the Crystallite Size (D) using the Scherrer equation, and it is possible to estimate the number of defects in a crystal. In the Scherrer equation, K is a shape factor usually taken as about 0.94 (Akl et al., 2021; Khan et al., 2010),

$$D = \frac{K \lambda}{\beta_D(2\theta) \cos\theta} \quad (3.10)$$

The dislocation density (d), defined as the length of dislocation lines per unit volume, has been estimated using the following equation (Khan et al., 2010)

$$d = \frac{1}{D^2} \quad (3.11)$$

Quantitative information concerning the preferential crystal orientation can be obtained from the texture coefficient, TC , defined as (Bindu & Thomas, 2014)

$$TC_{hkl} = \frac{I_{hkl}}{I_{0(hkl)}} \left(\frac{1}{N} \sum_i \frac{I_{i(hkl)}}{I_{0(hkl)}} \right)^{-1} \quad (3.12)$$

Where I_0 is the XRD intensity reference of the randomly oriented grains. If the nanoparticles are randomly oriented in all the $\langle hkl \rangle$ planes considered, the TC value should be about 1 for each peak. While values higher than 1 indicate the abundance of grains in a given $\langle hkl \rangle$ direction, smaller values indicate the lack of grains oriented in that direction.

The W-H method is a simplified method that differentiates between size-induced and strain-induced peak broadening (Bindu & Thomas, 2014). The strain-induced broadening due to crystal imperfection and distortion was calculated using the formula,

$$\varepsilon = \frac{\beta_s(2\theta)}{4 \tan \theta} \quad (3.13)$$

Now, assuming that the particle size and strain contributions to line broadening are independent of each other, the total peak broadening (w) can be represented by the sum of the contributions of crystallite size (β_D) and strain present in the material (β_s). From both expressions, Equations 3.10 and 3.13, the contributions can be isolated to give,

$$w = \frac{K\lambda}{D} + 4\varepsilon \tan\theta \quad (3.14)$$

By rearranging Equation 3.14, the result is the W-H equation for the uniform deformation model (UDM),

$$w \cos\theta = \frac{K\lambda}{D} + 4\varepsilon \sin\theta \quad (3.15)$$

Considering $4\varepsilon \sin\theta$ as the x-axis and $w \cos\theta$ as the y-axis, Equation 3.15 has the form of a linear equation where crystallite size (D) is estimated from y-intercept and the strain (ε) from the slope of the linear fit.

In the UDM model, the crystal is considered isotropic, and it is assumed that the properties of the material are independent of the direction along which it is measured. To incorporate an anisotropic approach, the W-H equation is modified by an anisotropic strain expressed from Hooke's law $\varepsilon = \sigma/E_{hkl}$, where σ is the stress of the crystal. That equation is just an approximation and is valid for a significantly small strain; when strain is increased, the particles deviate from this linear proportionality (Bindu & Thomas, 2014),

$$w \cos\theta = \frac{K\lambda}{D} + 4\sigma \frac{\sin\theta}{E_{hkl}} \quad (3.16)$$

For a cubic crystal as austenite, Young's modulus is given by (Stinville et al., 2011),

$$\frac{1}{E_{hkl}} = S_{11} + (2S_{12} - 2S_{11} + S_{44})A_{hkl} \quad (3.17)$$

Where S_{11} , S_{12} and S_{44} are the compliances of the 316L, which values are 10.7×10^{-12} , -4.25×10^{-12} and $8.6 \times 10^{-12} \text{ m}^2 \text{ N}^{-1}$, respectively.

The A_{hkl} factor is used to determine the anisotropy values; it is defined as $A_{hkl} = (h^2k^2 + k^2l^2 + l^2h^2)/(h^2 + k^2 + l^2)^2$, with limiting values of 1/3 for (hhh) orientation type and 0 for (h00) orientation type.

3.2 Results and discussion

3.2.1 Microstructural evolution

Figure 3-5 shows the optical microscopy images of microstructure after laser melting the surface with a 0.6 m·s⁻¹ laser scan velocity and a laser power of 280W. Cartesian and radial scanning patterns affected the samples superficially (~80 μm), where new grain growth is observed along the cooling direction during resolidification. Therefore, in the molten zone, the grains have a columnar appearance, while unaffected grains exhibit grains with an equiaxed morphology with some twins and grain size of $59.1 \pm 31.8 \mu\text{m}$. The morphology of the melted zone resembles that obtained after welding (Fan et al., 2021; Ma et al., 2018) or samples fabricated by additive manufacturing based on fusion such as Laser powder bed fusion and SLM (Ahmed et al., 2022; Huang et al., 2020; Racot et al., 2022). Figures 3-5 (a, b) correspond to the longitudinal cut, showing the melted surface and bulk interface. It is possible to observe the columnar structure produced by the thermal conduction from bulk, which produced a higher thermal gradient in the vertical direction. The rest of the figures correspond to the transversal cut, which means the laser path is toward the image. It shows the melted waves and overlapping laser paths. The main difference between the microstructure observed was the less uniform distances in the radial scan pattern, as shown in Figures 3-5.

The results concerning the depth reached by the laser melt front for the different scanning patterns and the surface roughness are summarized in Table 3-3. The laser depth of the melted region was similar in both patterns. However, it was slightly larger for the cartesian scan at 250W and 280W, with higher variability in the data. The latter could be attributed to its smaller hatch spacing ($55\text{ }\mu\text{m}$), producing a longer melted zone. Additionally, the cartesian scan showed more solidification ripples than the radial. This latter should be related to the hatch spacing. The nature of the radial scan produces different hatch depending on the radial distance, which changes from 33 to $75\text{ }\mu\text{m}$, while it remains constant in the cartesian scan pattern. However, the main difference between cartesian and radial scans is that the radial scan pattern maintains the path length, allowing a lower cooling rate in most treatments.

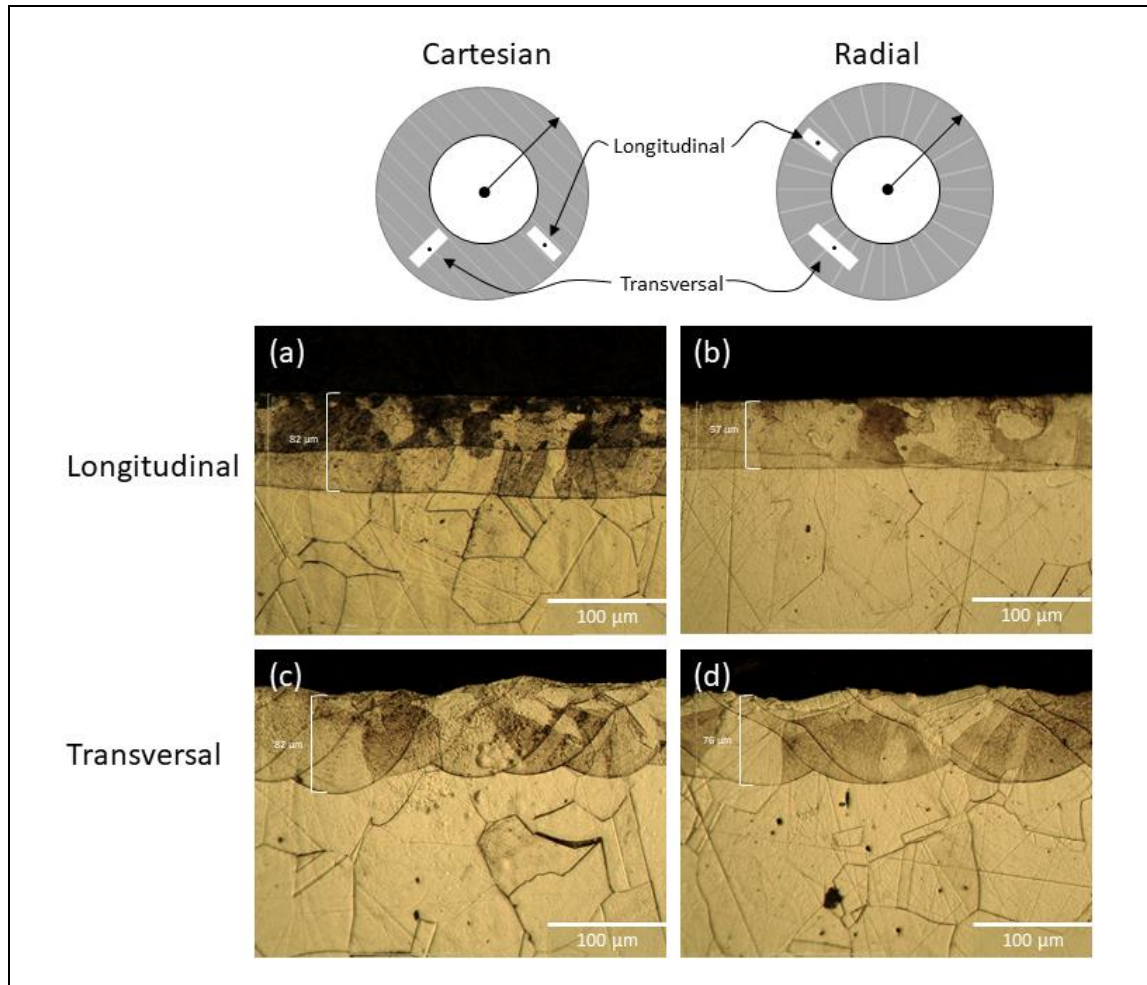


Figure 3-5: Optical microscopy images of a cross-section showing the resolidified microstructure after laser application with 280W (a, c) cartesian and (b, d) radial pattern.

Twins have been transformed into independent grains in the melted zone, changing the crystal orientation, as can be observed in Figure 3-5a. Besides, in this area the grain size decreased, implying the increase in yield strength according to the Hall-Petch relationship, and it can explain the slight increase in surface hardness as shown in Figure 3-8. The quantitative measurement of grain size within the melted layer was not possible to obtain, but the optical microscopy images show that melted layers do not have the largest grain

presented in bulk. It is also possible to note two solidification ripples within the microstructure in Figures 3-5a and 3-5b, almost parallel to the surface.

Figure 3-6 shows FESEM images of the cross-section of the laser-affected zone processed with different scanning strategies. The affected area shows a microstructure with a completely austenitic structure having equiaxial and columnar grains, which typically appear in laser welds (Fan et al., 2021; Ma et al., 2018). Figures 3-6a and 3-6d show the melted surface and the bulk interface. It is possible to observe the columnar structure produced by the thermal conduction from bulk, which produced a higher thermal gradient in the vertical direction. More towards the surface, those columnar structures were oriented with different angles, even horizontally, as shown in Figure 3-6e. The FESEM shows the texture of melted waves and the overlapping between laser paths. It is possible to observe that a certain surface undulation overlaps with the subsequent wave due to the small hatch space used, producing a widening in the upper area, which is easily recognizable in Figures 3-6 (b, e). In general, the texture of Figures 3-6(c, f) was present in the melted layers of two laser scan patterns. Those grains are completed columnar near the interface. Then towards the surface, there is a mixture of columnar and circular textures.

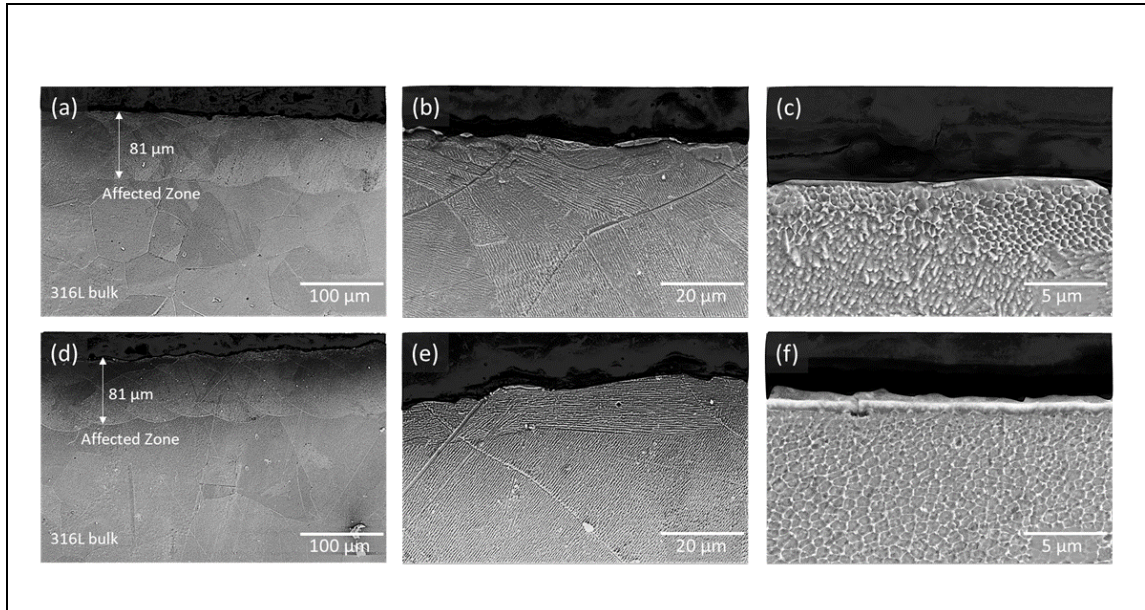


Figure 3-6: FESEM images of the cross-section of laser affected zone with (a, b, c) cartesian scan pattern and (d, e, f) radial scan pattern

3.2.2 Mechanical performance

Figure 3-7 shows the deformation of discs treated with 280W. It is possible to observe a clear deformation in annular discs treated with the cartesian pattern, whereas the deformation using the radial scan pattern was negligible. The main deformation factor could be the longer paths of the laser in the cartesian pattern, producing higher cooling and temperature gradients. The radial scan pattern presented a more constant cooling rate during solidification than the cartesian. The cooling rate in radial scans is lower than the cartesian cooling rate in almost the entire surface, except at the beginning and the end of the cartesian scan pattern.

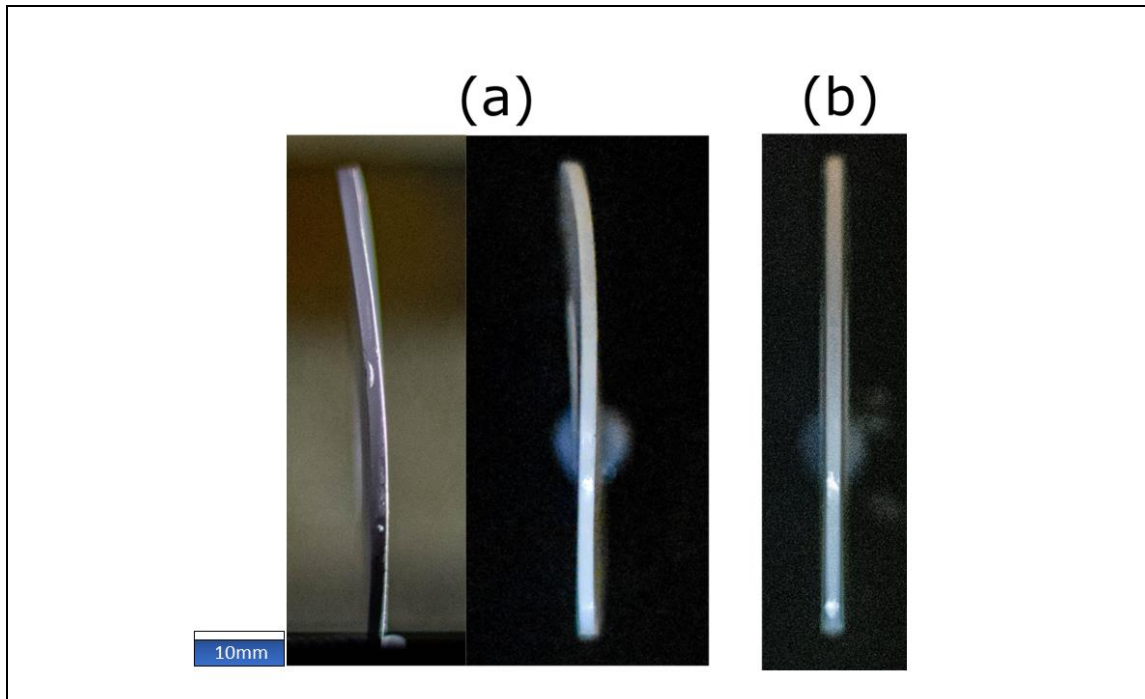


Figure 3-7: Deformation of annular discs with LSM using 280 W laser power (a) discs treated with cartesian pattern (b) discs treated with the radial pattern

The results of roughness are presented in Table 3-3. The LSM with the cartesian scan pattern produced a smaller increase in roughness values than treatment with the radial pattern, especially under the higher power configuration. The main reason for a greater roughness in radial treated samples is the non-constant hatch space. Besides, due to the constant direction of laser movement, in this case, maintaining the x-direction, there is a bigger difference between roughness measurement in the x and y direction than the difference on samples with the radial pattern.

Table 3-3: Results of melted region depth and roughness Ra measurements of samples, including the control and annular discs after LSM.

Sample	250 (W)			280 (W)		
	Melted region (μm)	Roughness (μm)		Melted region (μm)	Roughness (μm)	
		X	Y		X	Y
Control	-	0.22 ± 0.10	0.31 ± 0.12	-	0.22 ± 0.10	0.31 ± 0.12
Radial	69.0 ± 3.9	2.88 ± 0.70	3.01 ± 0.60	78.8 ± 1.9	4.32 ± 1.45	5.00 ± 1.20
Cartesian	72.3 ± 7.6	1.87 ± 0.84	2.45 ± 0.43	89.5 ± 9.9	2.16 ± 1.20	3.44 ± 0.83

Figure 3-8 shows the microhardness depth profile results of the different scan patterns. According to the scan pattern, it is possible to distinguish the values of the metal base hardness (average 151 HV) compared to the hardness values in the laser affected zone (187 HV). As it can be seen, independent of the scan pattern, the laser modifies the surface properties, raising the hardness of the material until approximately 300 μm from the top of the molten pool downward into the material. The hardness increases are due to the microstructural changes produced in the material after laser melting, where a newly resolidified microstructure with internal defects due to the quick solidification appears. The hardening increase with radial scan near 150 μm is associated with the residual stresses on the melted zone interface with the base metal (Kwok et al., 1998; Sriba et al., 2018), whose values are similar to those of the cartesian process. However, the lower hardness at near 30 μm is attributed to a compact defect-free zone during the solidification compared to the cartesian scan.

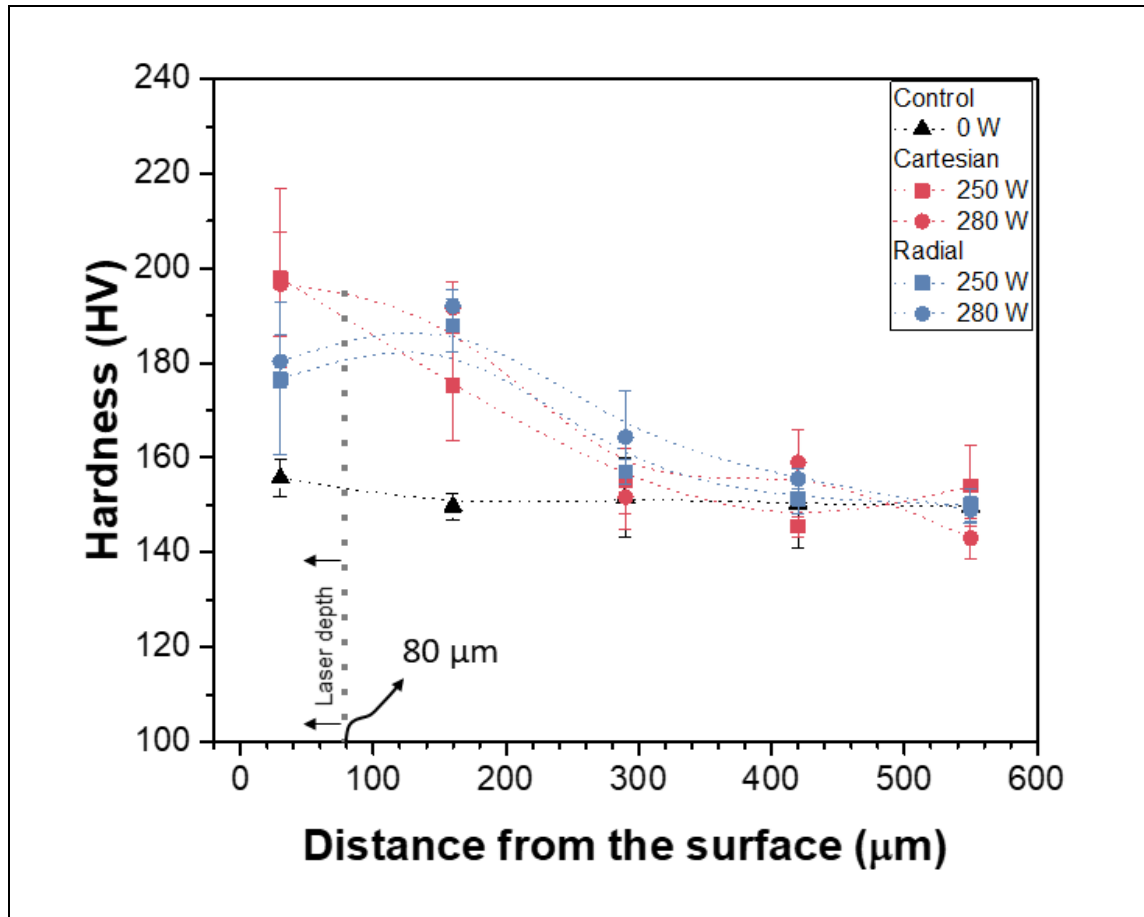


Figure 3-8: Measurement of Vickers microhardness evolution in the depth of annular discs samples.

Despite the laser melting of the surface reaching approximately 100 μm, the heat from the molten zone affected the adjacent microstructure, modifying the residual stresses between both zones, but without sufficient energy to increase the grain size up to 300 μm.

Figure 3-9 shows the results of the tensile tests of the annular disc samples at a deformation rate of $2 \text{ mm} \cdot \text{s}^{-1}$. Each curve represents the mean and its confidence interval from the testing results of each disc for a specific configuration. All samples show a

similar elastic modulus (as expected), ultimate strength, and yield strength but different elongation. The ultimate strength, in this case, represented by the maximum load, was reached by the control and sample with the cartesian scan pattern and 250 W. The lowest load supported was achieved by the radial scan pattern and 250 W. However, these values have no rotund differences since they vary from 27.9 kN to 25.8 kN.

The maximum elongation obtained from the tensile strength test was the property more affected by the laser surface modification. It is possible to observe in Figure 3-8 that the maximum value was achieved for the control discs (approx. 30 mm) and the lowest value for the discs treated with the radial scan pattern, which only reached an elongation of 15 mm.

It is important to point out that the internal area of the annular discs was not laser-treated. That could be a reason for the loss of strength and tenacity in samples with LSM. The mixture between 2 melted layers and the bulk showed a worse performance in longitudinal traction. The melted layer produced an increased fragility.

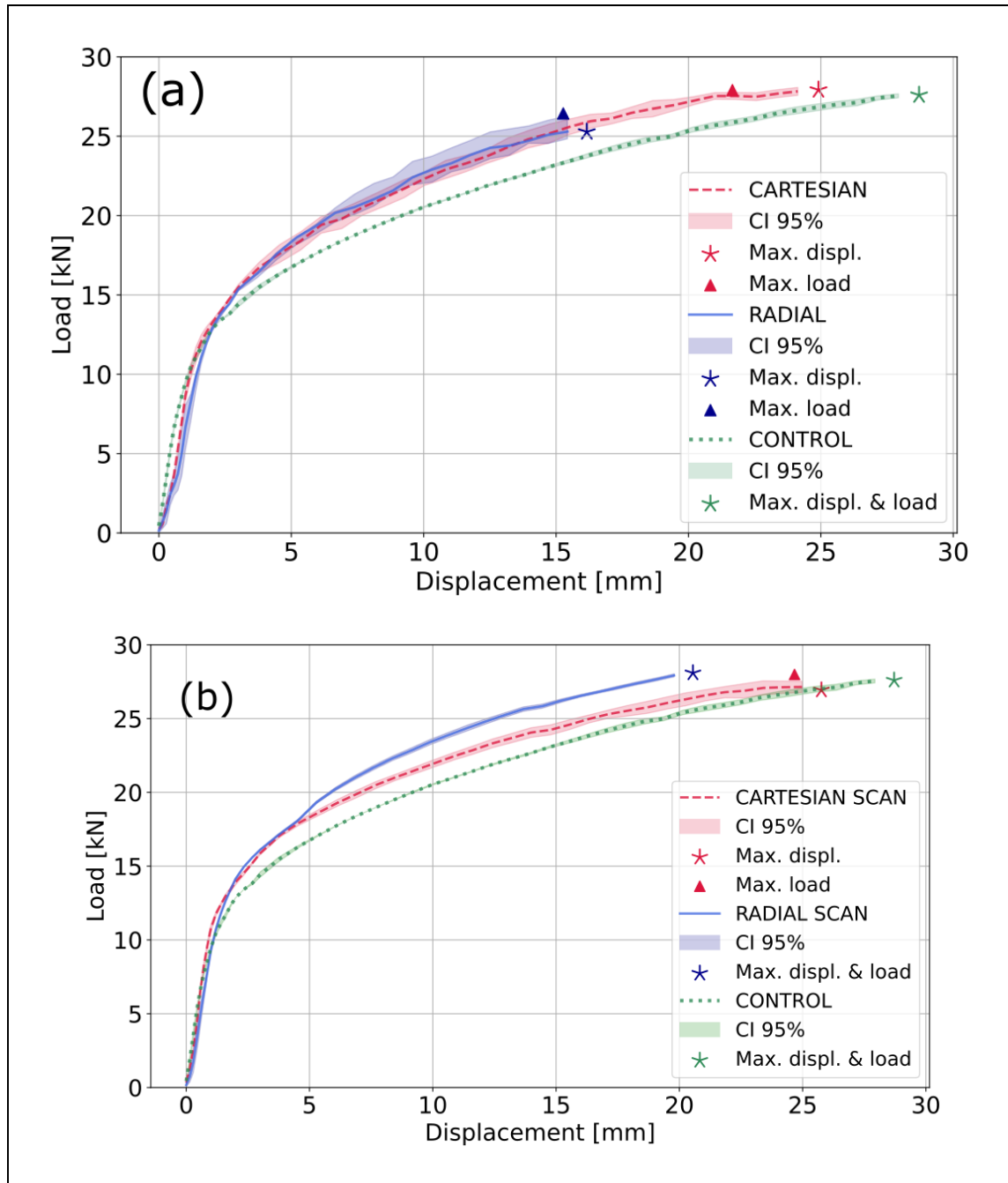


Figure 3-9: Tensile test results for discs modified with different laser scan patterns and control discs without laser modification. Annular discs modified with laser with (a) 250 W and (b) 280 W of power.

Chen et al. (2020) obtained similar results when studying high entropy alloys (HEA) with and without LSM. They attribute the samples' lower elongation and tensile strength with LSM to the residual stress and microcracks caused by the rapid solidification. The latter is possible because that could be regarded as the factor for the early initiation of the failure. Meng et al. (2017) treated a magnesium alloy with LSM and tested their samples by tensile stress at room and elevated temperatures. Magnesium alloy suffered a reduction in ductility after LSM processing. The author explains the phenomenon through the “amount of necking” of samples with LSM, which is less than the control samples, which means a reduction in ductility. Telasang et al. (2014) treated the AISI H13 tool steel surface using a high-power continuous-wave diode laser. The surface melted layer depth increased with incident laser energy density. After LSM, its re-melted surface was an inhomogeneous microstructure comprising non-uniform distribution of retained austenite, carbides, and martensite varying with depth.

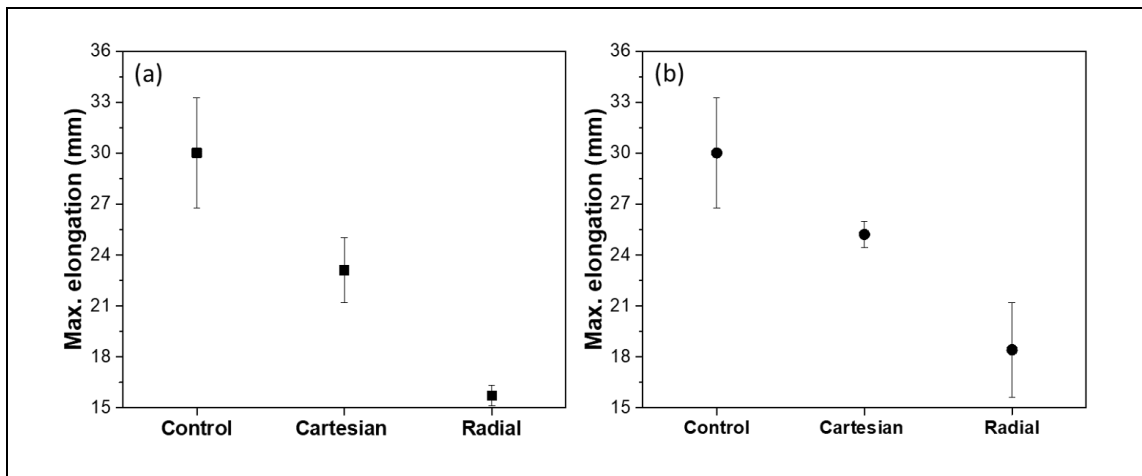


Figure 3-10: Max. elongation reached by annular discs in the tensile test, with an error with a confidence interval of 95 %. Annular discs modified with laser with (a) 250 W and (b) 280W of power.

In the present case, the reduction of elongation is due to the increase of internal defects during the solidification of the molten zone, which makes the material more brittle and marks the onset of failure, and the residual stresses between the molten and the non-thermally affected area.

Figure 3-11 shows the results of the residual stresses at each of the measured points in MPa using the ultrasound technique. It is important to consider that the depth of the laser melting zone (LMZ) was around 8% to 10% of the total thickness of the disc. The ultrasonic measurements of residual stress represent the average stress state along with the thickness, which includes the LMZ, the zone affected by heating without melting any material at all, and a bulk zone with the original stress state of the annular discs.

According to the results obtained, the stresses found in cartesian laser-treated annular discs present a greater dispersion than the discs laser-treated under radial scans. Besides, the radial discs show a more tensile state of residual stresses. In contrast, cartesian annular discs values oscillate between compression and tensile residual stress, with a greater tendency to compression state.

The residual stress produced by the laser depends on the amount of melted surface, the cooling rate, and the thermal gradient. The high changes in cooling rate that occur during the LSM with cartesian scan pattern, due to the change in the laser path length, produced a residual stress alternation.

The residual stress produced by the laser is added to the original state of samples, which is expected to have residual stresses by its manufacturing process. In the case of the annular discs studied, the compression state presented by the cartesian sample produced a higher deformation, which could be a consequence of a bigger tensile relaxation compared to the generated by the radial scan pattern.

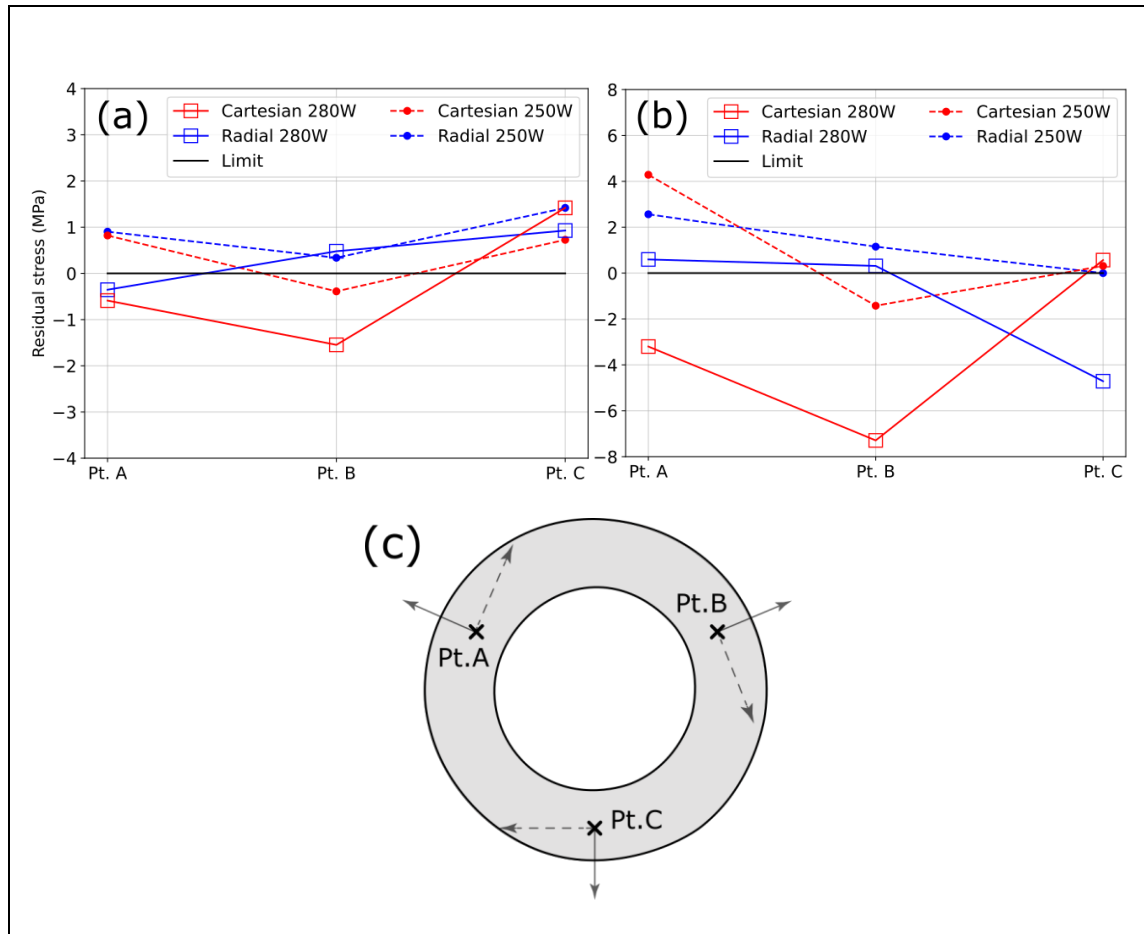


Figure 3-11: Residual stresses measured with ultrasonic waves technique related to the control disc. (a) Measured in the radial direction (b) Measured in the tangential direction (c) Scheme of points and directions of measurements; Continuous lines indicate the radial direction of measurements, while segmented lines indicate the tangential.

Finally, the compression state in the radial direction could contribute to a better performance in the tensile test of annular discs treated with the cartesian scan pattern, compared to annular discs treated with the radial pattern.

Because of the progressive reduction in peak temperature towards the boundary of the laser track, the residual stress is reduced in its magnitude along with the transverse and depth directions (Ganesh et al., 2013). It is expected that the point with the smaller residual stress is in the last area melted for samples treated due to the reduction of temperature variation produced. In the radial scan pattern, the last area melted is closer to the beginning area, which could modify the residual stress produced in the beginning area and is expected to have an impressive result in the last area. In Figure 3-11b, the C point of the sample treated with the radial scan pattern and 280W laser power was located between the beginning and the end of the laser treatment path, resulting in the highest residual stress measured for radial scans. Considering this previous result, it is possible to propose that a closed laser track as a radial scan pattern produces a compression state in the tangential direction of the laser movement, in the area that connects the beginning and the end of the track.

Table 3-4 shows the results of the XRD analysis in which each plane was refined and fitted with a pseudo-Voigt distribution. A represents the peak intensity, w the peak width, the xc the peak center, d dislocation density, and TC the crystal texture.

The lattice parameter for each specimen measured in Angstrom corresponded to Control 3.5959, Cartesian 3.5925, and Radial 3.5930. LSM produces a slight decrease. The results are the opposite of the microstructural evolution obtained by Balla et al. (2018). In their investigation, lattice parameter and grain sizes increased. However, the melted depth was around 500 μm , using velocities of 1 and 5 $\text{mm}\cdot\text{s}^{-1}$ and a continuous wave Nd: YAG laser with a beam diameter of 1.5 mm. This proves that the LSM could produce different microstructures in the same material, varying the laser velocity and power parameters.

It is possible to observe that all peaks of laser-treated discs in Table 3-4 show a slight positive displacement compared to the control discs, suggesting compressive residual

stress in the melted layer. The displacement of both laser scan movements is similar in all planes.

It has been shown by further experiments and analysis that generally, when the overlapping width at the surface is more than half of the surface width of the melting pool, there will be significant compressive stress in the surface layer; when the overlapping zone is too small, usually there is tensile stress in the surface layer which should be avoided due to being unfavorable to the fatigue resistance (Weiping, 1993). Related to the samples studied by XRD, the ratio between overlapping width and half of the surface width of the melting pool is about 2.6 in the cartesian scan and about 2.5 in the radial scan, which is a significant overlapping to expect compression stress in the melted layer. It is important to mention that the hatch changes in the radial scan pattern can produce different overlapping widths, which was observed in Figure 3-5d. The greatest disparity occurs in the external contour, where there exists the corner that almost overlaps the inward and outward paths and the bigger value of the hatch (75 μm between corners). The latter scenario could produce tensile states that decrease the tensile performance, as was seen in tensile testing.

An increase in stacking faults and structural disorder widens the XRD peaks (Kim & Chung, 2003). In the present XRD analysis, it is possible to observe that a thinning of peaks occurred in laser-treated annular discs. Studies have pointed out that the broadening of the peak indicates an accumulation of plastic damage, such as caused by dislocation generation during the deformation of the workpiece surface (Vashista & Paul, 2012). That could explain why XRD of laser-treated discs show thinner peaks because the rolled bar, which is the source where the discs were cut from, had an accumulation of plastic damage, and the laser acted as annealing the original state.

The calculated dislocation density (d) decreases in annular discs treated with the laser should be related to the tensile stress relaxation. That decrease in dislocation density could increase the fragility, especially in the interface between the layer melted and the bulk.

The lower deformation capacity showed by the laser-treated samples in the tensile test is mainly by the interface zone.

Different values were obtained for texture coefficient (TC), which indicates preferential planes of grain orientation. For control discs, grains are abundant in a (111) direction and a minimum quantity in a (220) plane direction. For annular discs treated with LSM, the results of TC are similar. The preferential planes were (111) and (200), but the significant trend for the (111) plane direction is softer. The least preferred plane direction for laser-treated discs was (311).

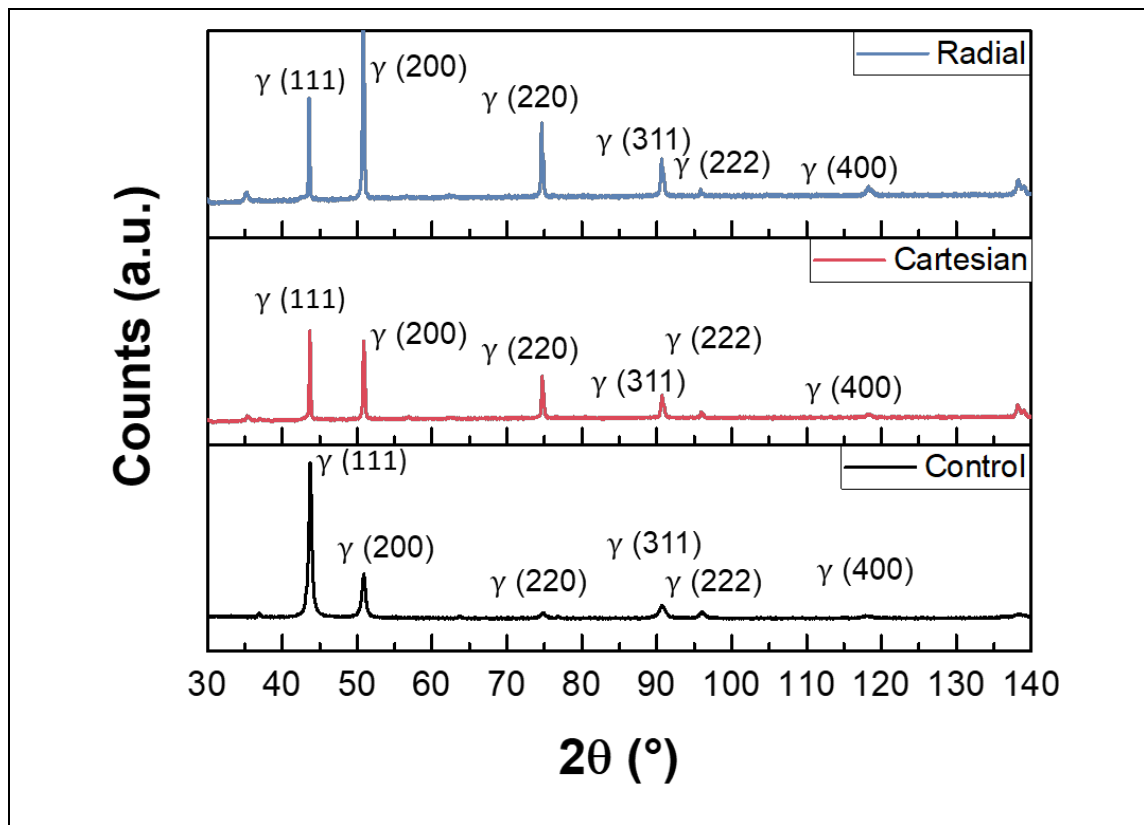


Figure 3-12: Raw results of XRD pattern performed on disc surface to the control, cartesian and radial samples before Rietveld refinement.

Considering the raw XRD pattern shown in Figure 3-12, i.e., before the refinement, it is possible to observe a different texture, with a preferential orientation along the a-axis in annular discs with radial laser treatment, due to enhanced intensity for the peak corresponding to (200) plane. That considerable difference could be produced by the surface roughness (Belassel et al., 2012), produced by a repositioning of atoms or Debye-Waller factors (Balzar, 1993). Additionally, it has been seen that XRD intensities vary significantly with an oxidation treatment (Kalyva et al., 2020). Suppose it is considered that the refinement overfitted the intensity peaks; in that case, the change of preferential planes shown in Figure 3-11 is clearly explained by the laser melting, the layers of solidifications, and the waves produced shown in Figure 3-6.

Table 3-4: The diffraction patterns per plane analyzed by fitting the peaks with a pseudo-Voigt distribution after Rietveld refinement. Changes in FWHM (w), intensity, the center of peaks (xc), dislocation density (d), and texture coefficient (TC) of the four first peaks (111, 200, 220, 311).

Peak		Control	Cartesian	Radial
1 1 1	$A (a.u)$	6239.10	1485.80	1487.95
	$w (^\circ)$	0.44	0.21	0.21
	$xc (^\circ)$	43.56	43.58	43.58
	$d (lines \cdot mm^{-2}) \times 10^8$	24.56	5.50	5.46
	TC	2.40	1.54	1.55
2 0 0	$A (a.u)$	2367.45	1062.05	1061.85
	$w (^\circ)$	0.440	0.21	0.21
	$xc (^\circ)$	50.73	50.76	50.76
	$d (lines \cdot mm^{-2}) \times 10^8$	23.38	5.40	5.34
	TC	0.91	1.10	1.10
2 2 0	$A (a.u)$	849.15	705.05	699.00
	$w (^\circ)$	0.45	0.23	0.23
	$xc (^\circ)$	74.58	74.63	74.3
	$d (lines \cdot mm^{-2}) \times 10^8$	18.63	4.99	4.87
	TC	0.33	0.73	0.73
3 1 1	$A (a.u)$	955.80	609.20	600.65
	$w (^\circ)$	0.46	0.26	0.25
	$xc (^\circ)$	90.54	90.60	90.60
	$d (lines \cdot mm^{-2}) \times 10^8$	15.08	4.68	4.52
	TC	0.37	0.63	0.62

The XRD results show that the laser does not increase the hardness and density of point defects, which should cause a linear increase in the FWHM of the XRD peak (Vashista & Paul, 2012). Besides, the decrease of FWHM seen in laser-treated annular discs implies a relaxation of tensile stress on the surface. Considering that all peaks of annular discs treated with laser are displaced positively compared to the control discs, which involve compressive residual stress, it is possible to confirm that the laser treatment produced a

relaxation of tensile stress on the surface. These results are also related to the decrease in the lattice parameters.

The results obtained by the Scherrer equation and Williamson-Hall (W-H) method are shown in Table 3-4. The crystallite size values obtained from the four first peaks were averaged for the Scherrer Method. On the other hand, the W-H used the seven more appreciable peaks between 15-140°.

The crystallite size for the control annular discs was calculated with the Scherrer method between 19-25 nm, for cartesian annular discs between 42-46 nm, and radial annular discs between 43-47 nm. As observed, the Scherrer Method's crystallite size is slightly greater than the size estimated by W-H. However, both methods show that the D in control samples is smaller than cartesian and radial, the two latter with D remarkably similar. Table 3-5 shows that the crystallites at least doubled their size in the melted layer.

It should be noted that these crystallite sizes are three orders of magnitude smaller compared to the grain size estimated by metallography images, and the laser-produced an opposite change in sizes of grain and crystallite for laser-treated samples, decreasing and increasing the sizes, respectively, compared to control discs.

The LSM effects in microstructure, such as the crystallite size increases in the melted zone, have also been observed by comparing the crystallite size and particle size of metal powders, both XRD and SEM images (Gubicza, 2014; Hankare et al., 2012). The grain size could be equal to or greater than crystallite size. The first directly relates to the tensile strength and properties such as ductility and high-temperature creep. Grain boundaries act as an impediment to dislocation motion, thus, smaller grains improve the strength, which has been largely used in polycrystal treatments based on the Hall-Petch Equation. The second is measured through XRD, calculated from the broadening of X-ray profiles, and is related to dislocations, planar defects, and chemical heterogeneities (Gubicza, 2014).

The crystallite size depends on the microstructure texture and the defects density. A bigger crystallite size will produce a thinner peak on the XRD results, associated with fewer dislocations. In severely deformed metals and alloys, the crystallite size obtained corresponds to the subgrain size. The austenite grains of 316L studied are composed of several grains, solidifying with a smaller size after both laser patterns. However, these grains developed a more uniform texture and decreased dislocation density through the relaxation produced by the laser treatment.

Table 3-5: Results of Scherrer and Williamson-Hall method for crystallite size (D) and microstrain (ϵ).

	Scherrer Method	Williamson-Hall Method				
		UDM		USDM		
		D (nm)	$\epsilon \times 10^{-3}$	D(nm)	σ (MPa)	$\epsilon \times 10^{-3}$
Control	22 ± 3	14 ± 1	-1.59 ± 0.2	19 ± 2	0.07211 ± 0.03	-0.5 ± 0.23
Cartesian	44 ± 2	37 ± 1	-0.24 ± 0.02	40 ± 1	0.01109 ± 0.005	-0.076 ± 0.04
Radial	45 ± 2	36 ± 1	-0.28 ± 0.02	41 ± 2	0.01289 ± 0.006	-0.089 ± 0.04

A relevant result for W-H methods is that all curves obtained had a negative slope, indicating that micro strains cannot be a dominant source of broadening. From these results, it can be inferred that the influence in the domain on the crystallite size of micro strains is negligible (Langford & Cernik, 1991). Despite that, the negative slope has been associated with the presence of compressive strain (Murugesan & Chandrasekaran, 2015), and due to the ineffectiveness of the W-H method to characterize faulting in very small crystallite sizes (Beyerlein et al., 2010), considering that broadening due to faulting has an hkl dependence that tends to increase with scattering angle, which should result in a W-H plot with a positive slope.

Among W-H methods, UDM considers the homogeneous isotropic nature of the crystal, whereas the USDM model considers the anisotropic nature of the crystallites (Bindu &

Thomas, 2014). The results for crystallite size were similar between methods. Considering the anisotropy of the austenite crystal structure, measurements of stresses were higher in control annular discs and similar for the laser-treated annular discs.

Even though integral breadth analysis based on the W-H method and subsequent modifications provides a simple means for a preliminary assessment of the main cause(s) of the broadening of diffraction lines (Scardi et al., 2004), the methods are useful to follow trends in series of analogously treated specimens. It is important to point out that the nature of line broadening caused by crystallite size, dislocations, and planar faults are essentially different. Therefore, the Williamson-Hall plot can be used for a qualitative assessment of the main origins of line broadening (Gubicza, 2014). More quantitative methods are necessary for future work, also testing more complex scanning patterns.

3.3 Conclusions

In this study, 316L stainless steel annular discs were used to assess the impact of applying a laser melting process, with radial and cartesian scans of the energy supply. Both scan patterns used the same laser velocity of $0.6 \text{ m} \cdot \text{s}^{-1}$ and the energy supply for 1 minute. Therefore, the total energy supplied only depends on the laser power used.

Two laser power were used for both scan patterns: 250 and 280W. The energy density per area was constant in the cartesian scan, while radially varies in the radial scans. The non-constant hatch values in the radial scan pattern, the non-constant path lengths in the cartesian pattern, and the different laser tracks between both scan patterns affected the results of the LSM.

The relationship between the scan pattern, energy density, microstructure, residual stresses, and tensile strength of parts processed was effectively established. The results are summarized as follows:

1. The main advantage in the LSM process of radial scan pattern compared to cartesian scan is the negligible deformation, which is expected to replicate in revolution parts. The lower geometric distortion is mainly due to the constant path length of the radial pattern.
2. The surface roughness R_a of samples treated was on average $1.3\text{ }\mu\text{m}$ higher for radial scan pattern than the produced by cartesian pattern.
3. Compared with the cartesian pattern, the main disadvantage of the radial scan pattern was the loss of ductility, which was observed in the lower maximum elongation in the tensile test of annular discs treated with the radial pattern. The high hatch variation of these samples in the external contour could be a factor of variable surface residual stresses, i.e., including tensile stresses in the melted layers.
4. The main factor in reducing ductility observed in laser treatment samples should be the interface between the melted layer and the bulk. That zone of drastic textures changes, and that work as a horizontal grain boundary produces a limitation to the mobility of the dislocations.
5. The laser treatment produced relaxation of tensile stress on the surface. The deformation produced on discs with LSM was negligible using the radial scan pattern. In contrast, the cartesian pattern presented a visible deformation associated with the longer paths of the laser, producing higher cooling and temperature gradients.

6. The changes in crystallite size and microstrain in annular discs with LSM were similar, regardless of the laser scan pattern used. Studying thicker samples using a higher energy density to prove if the laser scan pattern produces noticeable differences in XRD analysis is necessary.
7. The XRD analysis shows that LSM increases the crystallite size based on the thinner peaks obtained for samples treated. This implies the decrease of dislocation density by around 1/4 of the density in control samples.
8. Due to the negative slope in W-H methods, the width associated with microstrain is negligible and cannot be separated from the width of crystallite size. That result may be due to a compressive state, the limitation of the methods evaluating small crystallite sizes, or an inadequate function fitting used in the refinement.
9. Different mechanical properties and deformations were obtained after LSM using the cartesian and radial scan patterns. The results show that scan pattern should be considered a control variable in LSM, especially when thin pieces are treated or a high-power density is used.
10. The negligible deformation in annular discs treated with radial scan pattern promotes its use in indirect laser brazing. The applicability of the radial scan pattern in the Powder Bed Laser Fusion process cannot be certain due to the changes in the energy density, which could produce porosity by a lack or excess of fusion. In any case, the radial scan pattern could be beneficial in remelting applications.

Future work could be focused on increasing the power supplied and the size of samples, allowing a higher application of the results obtained in this study. It is important to explore

different scanner patterns considering the local part geometry. Taking into account these variables in the LSM process could be essential to achieving a fully controlled process.

4 GLOBAL CONCLUSIONS AND FUTURE WORK

This work aimed to characterize Laser Surface Melting on annular discs of stainless steel AISI 316L, considering two power levels and two scan patterns, Cartesian (zigzag) and Radial. The final goal is to achieve qualitative and quantitative assessments of the process consequences, comparing the microstructure evolution and mechanical changes between the two scan patterns.

Different measurements were developed, and instruments were used, such as optical microscopy and FESEM. Properties were successfully obtained, such as microhardness, roughness, residual stresses, uniaxial elongation and maximum tensile load, crystallite size, and microstrain. The integrated analysis of this information allowed us to identify the main differences caused by the laser scan pattern in LSM.

In the first stage, as a preliminary study, coins of AISI 316 SS were used to determine the optimal parameters according to the hereafter study objectives. The parameters defined using these coins were the samples' thickness and the laser velocity. On the other hand, this preliminary study provided interesting future comparisons with the AISI 316L annular discs due to the material and hatch space changes. It indicated that scan patterns could improve the contour in different laser-melting applications by studying the edge and its melting pool.

The main study on 316L SS annular discs was successfully developed in the second stage. The investigation allowed us to identify the properties more affected by the LSM and the properties that differentiate both scan patterns studied. The global conclusion regarding the measurements obtained could be:

- For better results in XRD analysis, the samples do not have to be deformed, which could introduce noise in the lecture on diffraction.

- By including the W-H methods, the crystallite size values decrease around 4 nm compared to the Scherrer method in each sample, which could be related to the negative slope that indicates the domain on crystallite size of micro strains is negligible. The main reasons for the negative slope could be overfitting of XRD data during the refinement, errors during the XRD test, or the presence of compressive strain. Despite this, micro strains did not be a dominant source of broadening.
- The increment of crystallite size in the melted surface is accompanied by an apparent decrease of grain size in both laser scan pattern cases.
- It cannot be assured that Rietveld's refinement increased the precision or overfitted the data because an important number of variables affect the XRD lecture. Nonetheless, since the material studied was polycrystal, refinement was considered necessary.
- The hardness increment in the layer melted is due to the decrease in grain sizes. The residual compression produced in this layer also increases the hardness of the next micrometers of the bulk to about 250 μm in-depth.

The hypotheses of this work were corroborated since some notable changes were obtained between samples treated with cartesian and radial scan patterns in qualitative and quantitative estimation. Besides, the radial scan pattern produces less residual stress and is more homogenous, with a tensile tendency. The texture coefficient could support the last hypothesis, related to the notorious differences in microstructure and overlapping observed. However, the other measurements as crystallite size, melted surface depth, and peak displacements, were mostly similar to cartesian and radial patterns.

Recommendations for future works include:

- To perform simulations and compare with the experimental results. The use of software favors studying different shapes, more variables, or more levels of power and velocity.
- To include the study of new scanning patterns in the process of LSM, also considering the measurement of oxidation and wear resistance.
- Including a more sophisticated method for XRD analysis to obtain more quantitative results in this characterization. Besides, future works could be expected to improve the Rietveld refinement or study its usefulness for similar cases of melted surfaces.
- Studying new scanning patterns and determining the main results in samples to generate data that could be used with Artificial Intelligence models. Robust data could predict the specific results and guide the treatment, for example, using a specific scanning pattern in certain areas.
- To extend the application of results to related areas mentioned during the present manuscript. The morphology of the melted zone obtained is like that obtained after welding or in samples fabricated by additive manufacturing based on fusion as Selective Laser Melting. Future work could evaluate the radial scan pattern in SLM, paying attention to residual stresses, especially in the contour finishes, due to expected differences in final roughness and porosity on the surface of samples.
- To go deeper into the effects and control of solidification ripples and edge melted pool.

Finally, the results obtained in this study suggest that the scan pattern should be considered in LSM treatment, especially for thinner pieces, and that the main difference between Radial and Cartesian patterns were macro, i.e., deformation after laser treatment and maximum deformation in the tensile test, while the microstructure and other properties such as crystallite size and depth of melted surface show similar values between both scan patterns.

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ANNEXES

Annex 1: Fieldwork

This Annex describes the methods and analyses carried out in this study. General criteria used methodologies and results are described.

Annex 1.1: Ultrasonic waves method to calculate Residual Stress

The study of residual stresses using the thickness of samples induces error because it is difficult to achieve a high precision measurement. The main advantage of Poisson's ratio is that it can be determined by measuring flight times, so there is no need to measure the sample thickness to calculate the residual stresses.

In the thesis of Solis (2010), the velocities on a Structural Steel were carefully measured, considering the elastic range and taking measurements in different tensile states during a tensile test. The purpose of this study was to validate the method.

With the velocities and the applied stress values, it is possible to obtain the acoustoelastic constants (K_{ij}). Using the values of these acoustoelastic constants, the residual stress in steels is obtained just by measuring the time of ultrasonic waves, using the equation (3.7 – 3.8) as a linearly independent system of equations for σ_1 and σ_2 .

To determine the P_{ij} values of the Taylor series for $v_{3j}(\sigma_i)$, the following expression was used (Solis, 2010):

$$P_{11} = \frac{(t_S/t_L)^2}{(t_S/t_L)^2 - 1} (K_{22} - K_{21}) [1 - 2\nu] = P_{22} \quad (\text{A.1})$$

$$P_{12} = \frac{(t_S/t_L)^2}{(t_S/t_L)^2 - 1} (K_{22} - K_{23})[1 - 2\nu] = P_{21} \quad (\text{A.2})$$

$$P_{13} = \frac{(t_S/t_L)^2}{(t_S/t_L)^2 - 1} (K_{11} - K_{12})[1 - 2\nu] = P_{23} \quad (\text{A.3})$$

A detailed description of this method to calculate residual stresses using the Poisson's ratio is available in the Carvajal L. patent published in 2016. Its validation and the estimation of constants for the steel are available in Solis R. thesis (2010).

Annex 1.2: Rietveld refinement of XRD data

In a polycrystalline sample, much information will inevitably be lost due to the random orientation of the microcrystals. In practice, the loss of information is due to the overlapping of independent diffraction peaks. The Rietveld refinement allows extracting more information from the XRD diagram by using the intensity profile data of each reflection instead of its integrated area in the refinement process.

The method consists of refining the crystalline and magnetic structure of a certain material, minimizing the weighted squared differences between the observed intensities and those calculated.

The quality of the adjustment can be analyzed with discrepancy values or R-factors, which correspond to one criterion for judging Rietveld fits (Toby, 2006):

- R_{bragg} , observed and calculated differences in intensities.

- R_f , observed and calculated differences in crystallographic structure.
- R_{wp} , “weighted-profile” R-factor, is the most direct measure for monitoring refinement convergence.
- R_{exp} , “expected” R-factor, essentially is a measure of data quality, it corresponds to the ‘best possible R_{wp} ’.

The ratio of the R_{wp} and R_{exp} R-factors gives the chi-squared value for the fit. Lower R-factor values generally imply better adjustment between theoretical and experimental results. The factors obtained in each refinement are shown in Table A-1. The factors obtained are higher than expected because of the notable differences in the amplitude produced by laser treatments. The reasons for these differences are commented on in chapter 4. Removing background, which was done to data to level the background and allow comparisons, is another reason that increases the R_w and χ^2 (Mccusker et al., 1999; Toby, 2006). The refinement method was applied with Match! 3 software and converged successfully in all cases. The “Modified March’s function” was used for preferred orientation due to the use by default of the “Exponential function” produced higher R-factors.

Table A-1: Factors values obtained from Rietveld refinement developed.

R-Factor	Control	Cartesian	Radial
R_{bragg}	22.5	34.5	35.1
R_f	21.9	25.6	26.0
R_{wp}	48.5	55.8	56.1
R_{exp}	6.1	7.7	7.6
χ^2	63.4	53.0	54.0

The final decision about the refinement quality should be made considering all available information: R-factors, χ^2 , and visual inspection of the difference curve. Finally, the decision is not only about the quality of the fit but also about the meaningfulness of the refined parameters and the quality of the experiment itself. The same refinement was

applied to the XRD data in the present study, i.e., using the same configurations. In the refinement was used the same phase to fit the data, and in each case, the background was removed. Although the R-factors were higher than expected, the refinement converged successfully. The refinement effects on each XRD data allow the comparison among the configurations (Control, Cartesian, and Radial).

Annex 2: W-H method graphs

Annex 2 presents the plots and linear fitting of the refined data using Match 3! Software, which was used to develop the W-H method.

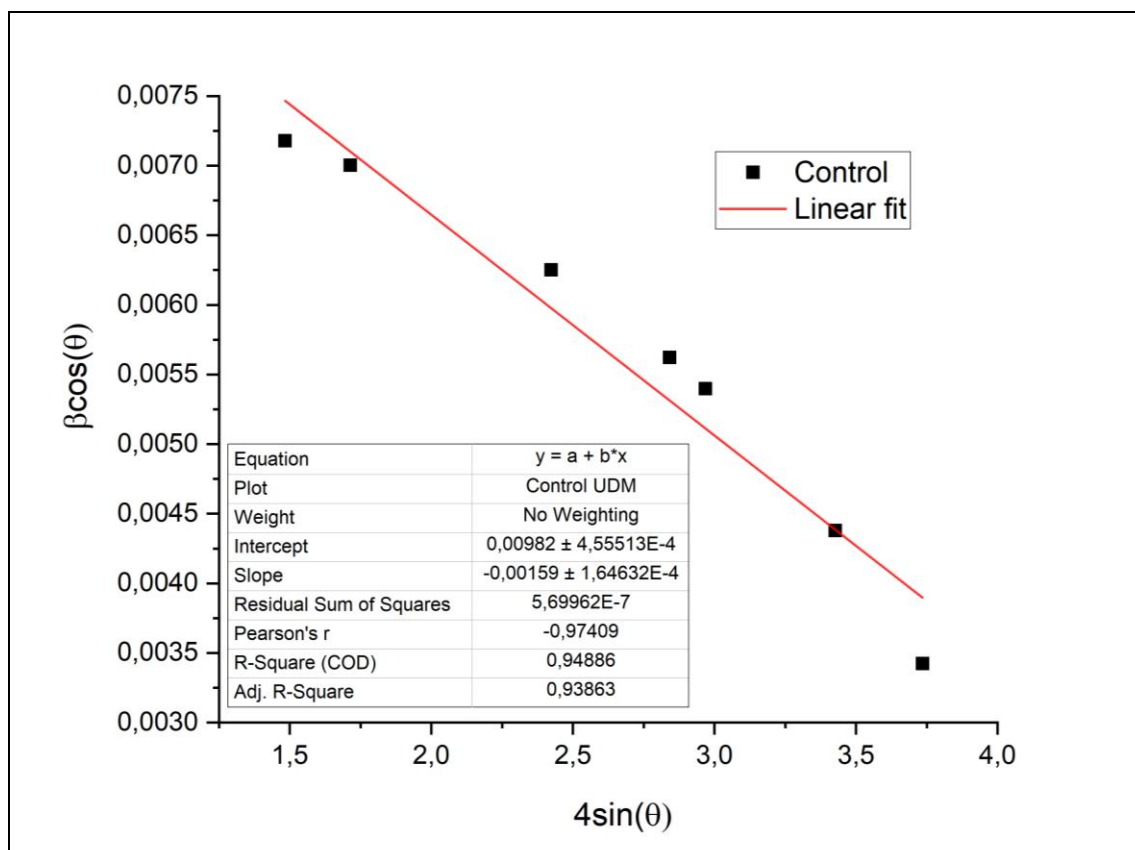


Figure A-1: W-H plot of Control sample. Linear fit following the UDM method

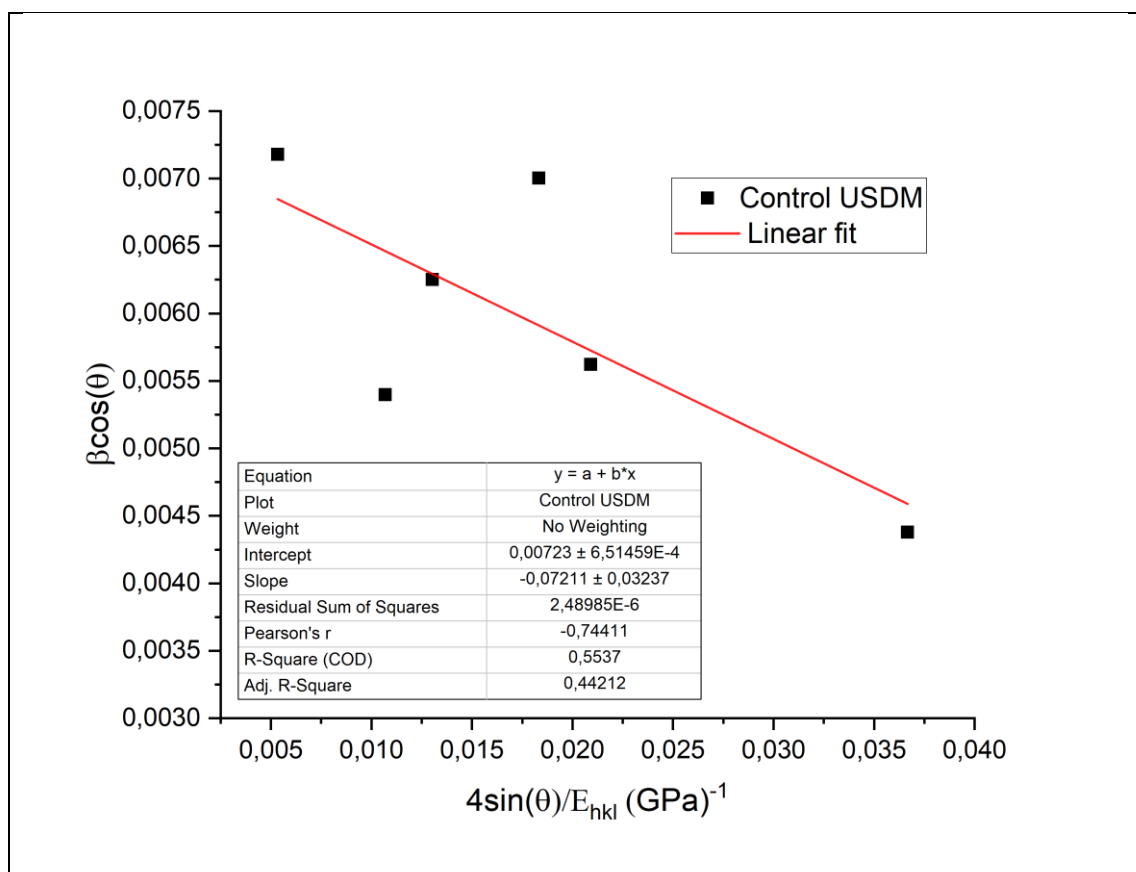


Figure A-2: W-H plot of Control sample. Linear fit following the USDM method.

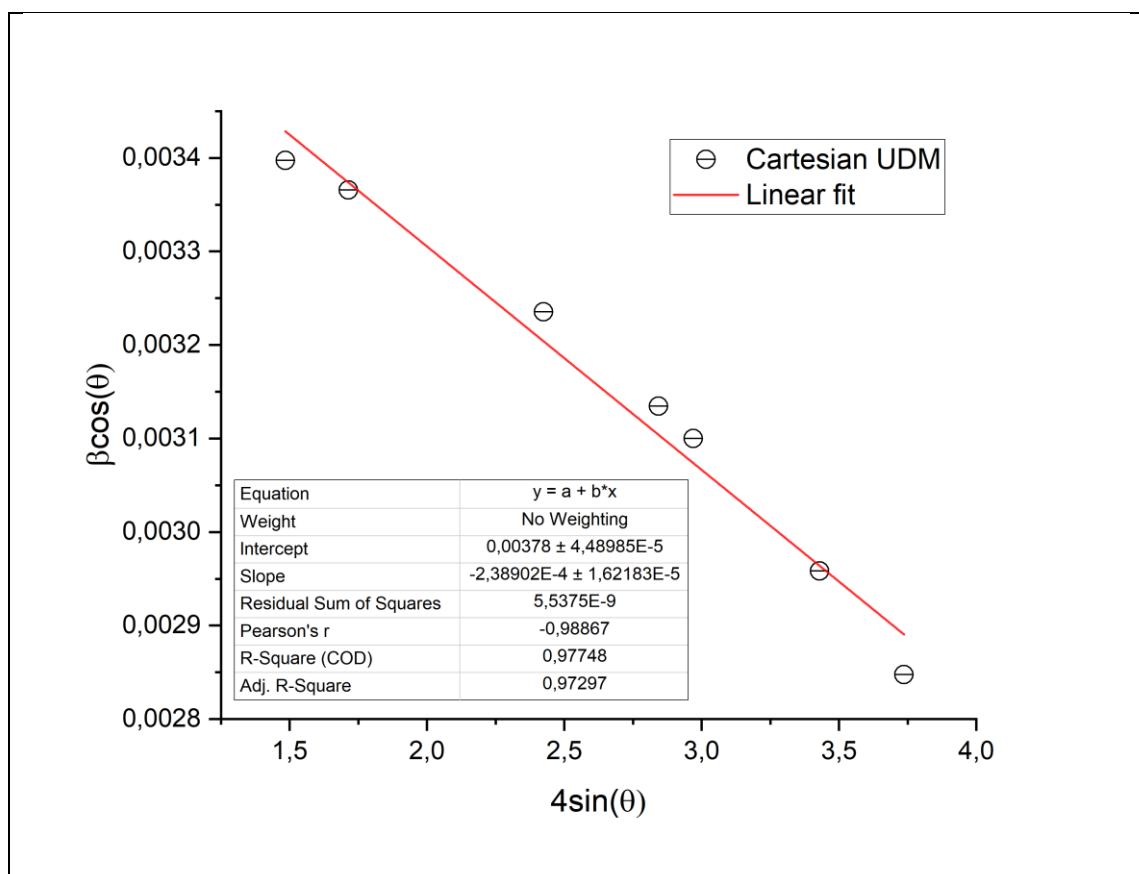


Figure A-3: W-H plot of Cartesian sample. Linear fit following the UDM method.

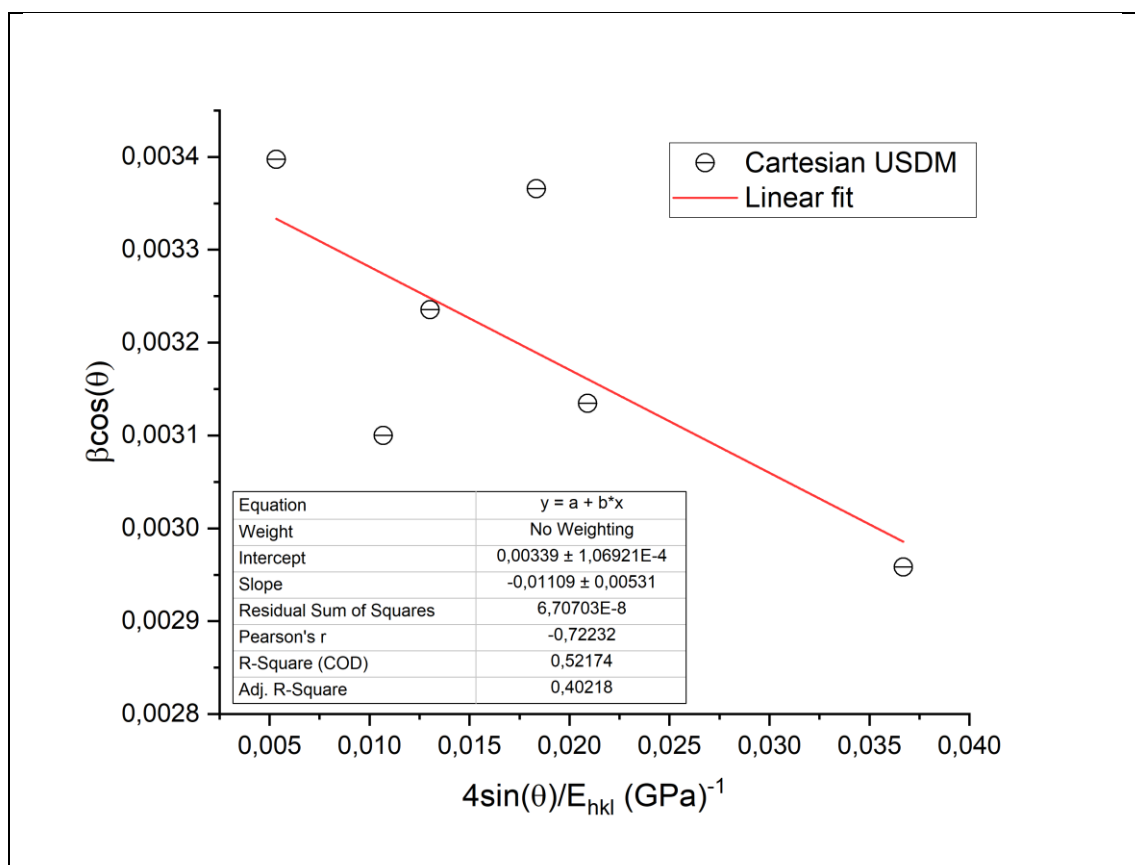


Figure A-4: W-H plot of Cartesian sample. Linear fit following the USDM method.

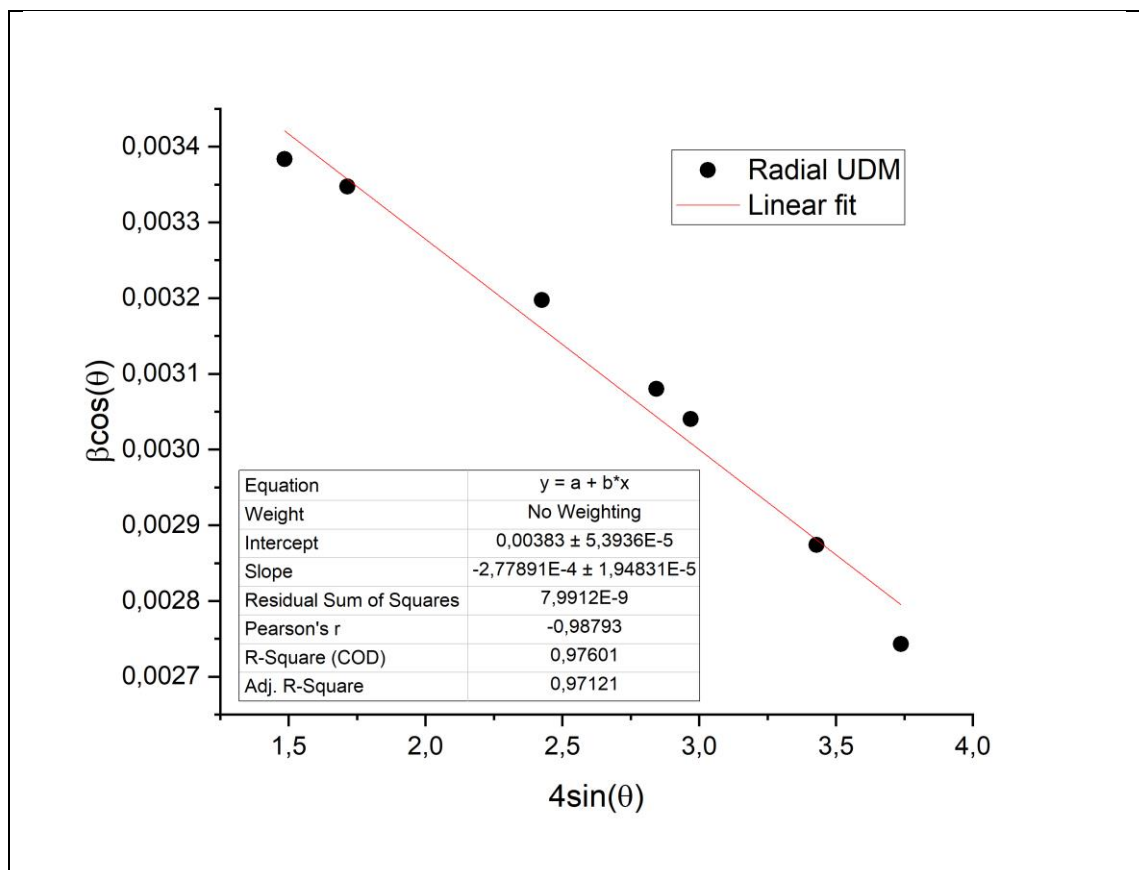


Figure A-5: W-H plot of Radial sample. Linear fit following the UDM method.

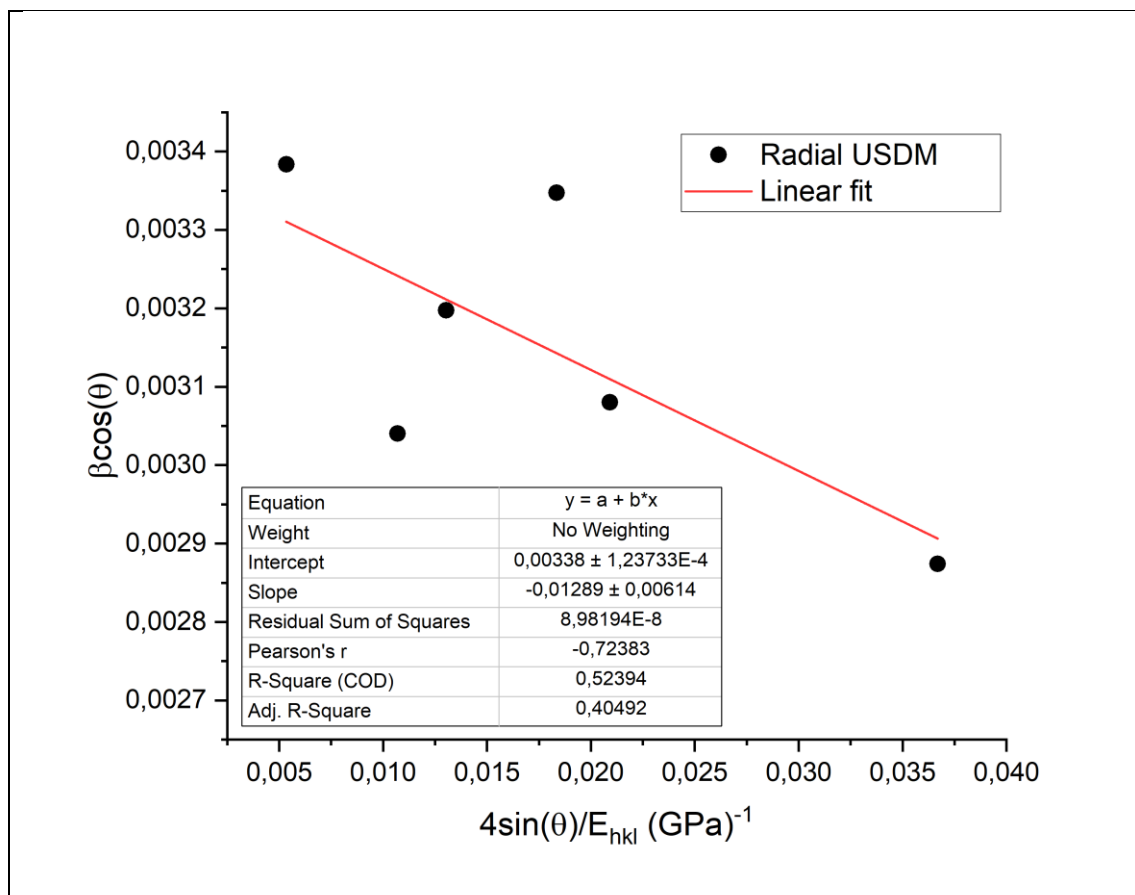


Figure A-6: W-H plot of Radial sample. Linear fit following the USDM method.