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PRELIMINARY EVALUATION OF SMA ADDITIVE MANUFACTURING PROCESS USING SELECTIVE LASER MELTING FOR APPLICATION IN SCRAMJET ENGINES

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Abstract. *Since the need for smart materials to be applied to the leading edge of scramjet engines, the present project analyzed the parameters in the NiTi matrix alloy, as they present characteristics of Shape Memory Effect (SME) and Superelasticity (SE). Following studies on additive manufacturing process in NiTi parts, using an unconventional process, the parts were manufactured by 3D printing using CAD software. It is a relatively quick process; therefore, metal parts are structured layer-by-layer using a laser beam to melt the material. This technique has advantages over the conventional technique of manufacturing parts with more complex geometries without the need for molds or prototypes. For the process to occur in a promising way in the market, the source of material deposition is made by Selective Laser Melting (SLM), which has the fusion of the powder layer and deposition of directed energy. In this work, the processes of Powder Bed Fusion (PBF) and Directed Energy Deposition (DED) from Additive Manufacturing (AM) were addressed. When analyzing the processing for the production of parts via SLM, it can be said that the energy density and power of the laser are relevant parameters for obtaining parts with good mechanical properties, low porosity and high density. The mechanical alloying method applied to handle the powder mixture showed better results in its microstructure compared to simple mixing.*
Keywords: *Additive Manufacturing, Selective Laser Melting (SLM), Directed Energy Deposition (DED), Powder Bed Fusion (PBF), Scramjet Engines*

1. INTRODUCTION

Currently, Additive Manufacturing (AM) has become a major focus for modern industry owing to its advanced techniques in manufacturing parts with complex geometries without the need for specific tools and optimizing the process time. Its manufacture consists of depositing the raw material, which can be in the form of wire or powder under a metallic part by fusion with the aid of a heat source such as laser or electric arc, building layer-by-layer from a three-dimensional digital model originating from computer software. AM has been applied to various types of materials such as glass, ceramics, metals, polymers, and composites (SUN et al, 2021). These techniques can be applied in multiple ways, for example, arc welding, laser cladding, selective laser melting, sintering of metal powders and by variations of these mentioned processes.

In the last 20 years, major technological advances in the processing of metal AM, including industrial lasers, computer hardware and software, have contributed to a reduction in costs and enabled a modern method. Techniques developed in research laboratories are now adhered to in industry. However, extensive knowledge of this technique is required to produce defect-free, reliable, integrated AM components. The production and design of high-performance components for aerospace applications, such as complex fuel injector nozzles used in aircraft, previously required assembly of multiple parts, have result in significant cost savings (Debroy et al, 2018).

In the mid-1960s, employees in the U.S. Naval Ordnance Laboratory discovered the memory effect of the equiatomic NiTi alloy, which became known as Nitinol, and was later applied in the aircraft industry. Its effect is associated with a change in structure when a solid-state phase transformation occurs, called martensitic transformation, which is induced by the application of stress or temperature variation, this phase is associated with reversible structural modifications inserted into the material during deformation.

According to (Martelluci and Harris, 1991), advances in propulsion, aerodynamics, and flight control design have allowed aerospace vehicles to operate at speeds above Mach 5 (hypersonic speed), however, many problems still exist owing to aerodynamic heating and shock wave interactions resulting in high airframe temperatures, causing high thermal stresses and rapid ablation

According to Costa (2014), a scramjet engine uses an oblique shock wave generated during hypersonic flight to promote the compression and deceleration of the undisturbed flow at the inlet of the scramjet engine, which is integrated into a hypersonic vehicle. To obtain proper conditions at supersonic speeds for fuel combustion in a combustion chamber, scramjet engines feature a critical component that has an extreme influence on engine operation and performance. As a vehicle accelerates, the shock layer generated during hypersonic flight tends to become thinner; thus, the shock wave tends to become more tightly bound to the vehicle body. Given this scenario, a variable-geometry system is required to allow the scramjet engine to adapt to the different operating environments faced in its flight, i.e., to make the leading edge of the cowl move back or forward on the leading edge optimizing the engine's performance. One proposal to meet these requirements is to employ intelligent materials, the so-called Shape Memory Alloys (SMA) at the leading edge of the scramjet engine.

Among the alloys present in the manufacturing process, SMA demonstrate the ability to return the shape or size previously defined when exposed to a thermo-mechanical action, presenting important characteristics: Shape Memory Effect (SME) and Superelasticity (SE). The SME is the ability to recover from inelastic deformation after heating to a certain temperature, which is observed in the microstructure of the material and the SE is the property of the material that allows the initial recovery when removing the load that is being exposed (Brito et al, 2020). As shown in Table 1, we can analyze the most commonly used alloys in additive manufacturing and their applications.

Table 1. Table related the most commonly used AM alloys according to applications (Milewski, 2017).

Alloys ⇨ Applications ⇩	Aluminum	Maraging steel	Stainless steel	Titanium	Cobalt chrome	Nickel super alloys	Precious metals
Aerospace	X		X	X	X	X	
Medical			X	X	X		X
Energy, oil and gas			X				
Automotive	X		X	X			
Marine			X	X		X	
Machinability and weldability	X		X	X		X	
Corrosion resistance			X	X	X	X	
High temperature			X	X		X	
Tools and molds		X	X				
Consumer products	X		X				X

To choose the method to be used, it is necessary to analyze the geometry of the part and the alloy to be deposited (Souza, 2022). Metallic alloys can be manufactured by various processing techniques, such as conventional processes, in the case of casting, followed by thermo-mechanical processes or powder metallurgy, however, these types of processes are more expensive than non-conventional processes. The non-conventional process, characterized by the principles of additive manufacturing, provides advantages over the aforementioned techniques, however, manufacturing time of large batches and the volume of porosity remain the main disadvantages (Marques et al., 2014). Thus, the overall objective of this study is to analyze the microstructural effects of alloys with shape memory effect, address advantages and disadvantages after additive manufacturing processing via selective laser melting.

2. MATERIALS AND METHODS

2.1 Additive Manufacturing

Additive manufacturing (AM) is a layer-by-layer manufacturing process, similar to 3D printing, in which materials are added according to a CAD project. Its manufacturing process is ideal for components of great complexity and manufacturing small batches of products without the need for molds or prototypes. According to (Berman, 2012), the constructive process starts with the principle of modeling the geometry chosen from specific CAD software. Then, the program has the function of "slicing" the geometry in numerous layers and sets the coordinates in which the printer should follow. In construction, pre-deposited powders are used, which are melted and built up in overlapping layers on a metallic surface. The selected geometry is printed until completion, as seen in Fig. 2.

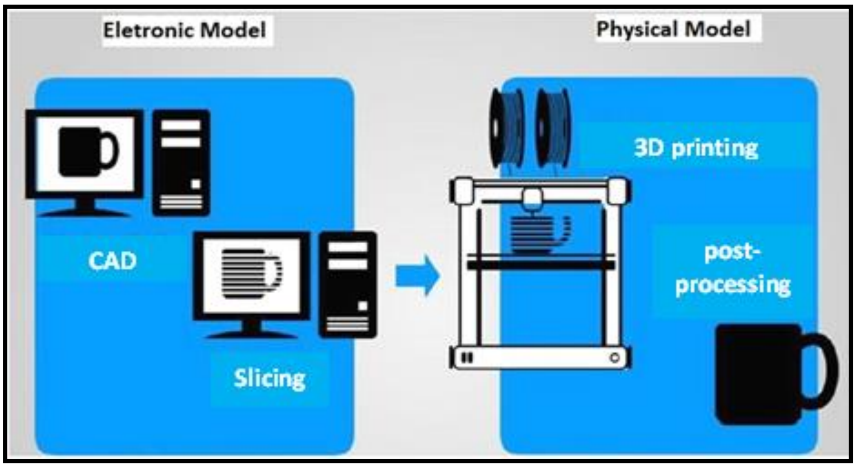

Figure 2. Schematic representation of the additive manufacturing process. (Adapted from Oliveira, 2017)

Among the routes for the production of materials from Ni-Ti alloy, many researchers specializing in the area, consider to mention the importance of preparation of powders to obtain the desired properties, i.e., it is possible to proportion different characteristics, such as increased super-elasticity effect in alloys rich in Ni and shape memory effect in alloys rich in Ti. Consequently, it can directly affect the particle size, distribution, and degree of impurity. However, the search for optimal processing in manufacturing allows the production of products with high density and low impurity (Elahinia, et al., 2016) and (Oliveira, 2019).

In this study, rapid prototyping techniques are presented. According to (Debroy et al., 2018), the AM process can be divided into two subgroups according to ASTM Standard F2792: Directed Energy Deposition (DED) and Powder Bed Fusion (PBF). It is possible to group them as the heat source, in the case of Laser (L), Electron Beam (EB), Plasma Arc (Plasm Arc - PA) or Gas Metal Arc (GMA) (Souza, 2022).

Figure 3 shows a schematic of the main additive manufacturing technique using metal powder. Powder Bed Fusion (PBF) method, Fig. 3(a), uses an electron beam or laser to fuse a layer of powder. Typically, the thickness of the fused layer on the platform surface measures between 30 and 100 µm. After one layer is fused, the underlying part is covered with an additional layer of powder, which continuously surrounds the entire part. This technique requires that the powder be of an alloy composition, atomized as a single alloy or mixture. In addition, a sufficient amount of powder is required to remain until the end of part construction. The Directed Energy Deposition (DED) method (3(b)) feeds metal powder coaxially into the laser or electron beam through a set of nozzles, building a path of molten material that uses inert gas to direct the powder or wire into the melt pool where the focal point of the laser or electron beam is pointed. The head of the machine containing the powder stream moved in different directions and is above the top of the platform. It is recommended that the material used be an alloy or spherical powder mixture with a diameter between 50 and 150 µm. (Bandyopadhyay et al., 2022), (Zhang and Bandyopadhyay, 2019) and (Zheng et al., 2018).

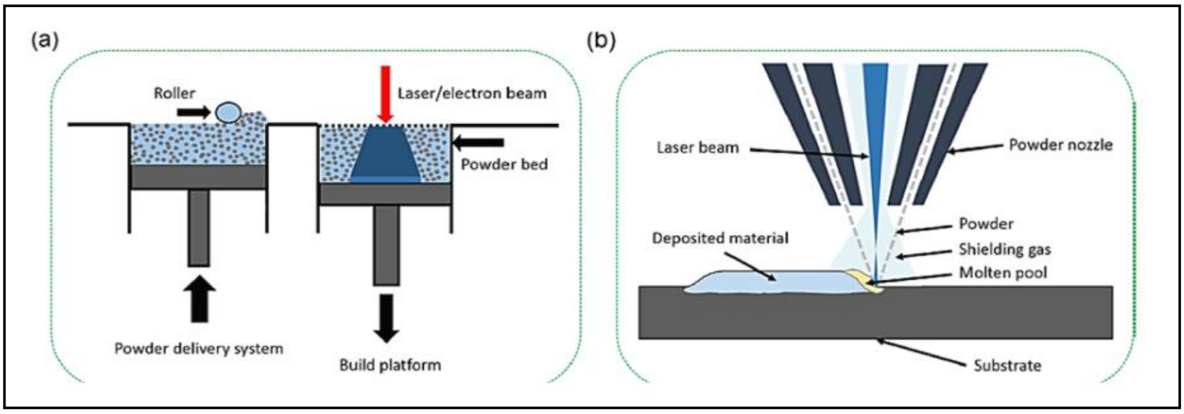

Figure 3: Principles of the methods: (a) powder bed fusion (PBF) and (b) directed energy deposition (DED) (Adapted from Yang et al., 2021).

By comparing the parameters of the DED and PBF processes, we observed differences and limitations. For example, for higher power and shorter production time, we used DED, however, if the industry demands a higher cooling rate and better dimensional accuracy, we recommend PBF. Thus, there is no single method that covers all the needs of the industry, such as complexity and size of the desired geometry, the alloy that will be deposited, resources and time available and skills to properly select the technique that will be used in the preparation of the piece in order to meet the expected criteria (Alberti et al., 2015).

Each specific application generates a need for a new alloy with improved properties so that the cost of the manufactured product is also a significant factor. Industrial processes for these new alloys can be designed using computational approaches, experimental data or specific knowledge (Mitra et al., 2021). The chemical properties of the material, its thermomechanical properties, and production volume directly influence the performance of the material used in additive manufacturing. For example, by adding elemental powder Ti to SMA, the melting point can be raised above 450°C for aerospace applications or manage the use of Ni in metal alloys in biomedical applications by causing sensitivity in patients. These increases in the chemical composition of alloys used in AM will improve the future of manufacturing in the coming decades (Bandyopadhyay et al., 2022).

Among the techniques currently available for AM, SLM is the most widespread and used worldwide, extending to various industries such as automotive, aerospace, medical technology and among others. It allows the production of components that are difficult to manufacture using conventional processes and parts that require minute details and free internal cavities (Montuori et al., 2020) and (Ozsoy et al.,2021).

2.2 Selective Laser Melting (SLM)

From the evolution of selective laser sintering, which uses a laser beam or other sources to melt the raw material in its powder state (Ribeiro, 2018), a new technology called Selective Laser Melting (SLM) was created. Similar to rapid prototyping techniques, the SLM process starts from the prototype of the 3D CAD model of the product to be manufactured, that is, a specific software slices the model in cross sections in which directs the command to the equipment responsible for the selective fusion of the metal powder concomitant with the laser.

The process started by fusing the powder bed on the surface using a laser with an average power of 50W and a peak power of 3kW. The laser beam was conducted using fiber optics and focused on the substrate. Powder deposition was applied to the fixed piston base. The laser beam scanned the powder bed according to the data preset in the CAD software and scanned the chamber filled with inactive gas to prevent oxidation. The head of the equipment was attached to an x-y table, and it was possible to control the height of the axis. When aligned with the platform, the powder was dragged through the construction platform and a three-dimensional model is built on the base plate. After the first layer of molten powder is deposited, the material quickly solidified, then the piston is lowered to the next layer until the proposed model is fully completed. The scanning strategy consists of building a 3D model by scanning the outer area of the geometry and hatching the inner area in X and Y-directions, as shown in Fig. 4. (Abe et al., 2017), (Oliveira, 2019) and (Marques, 2014).

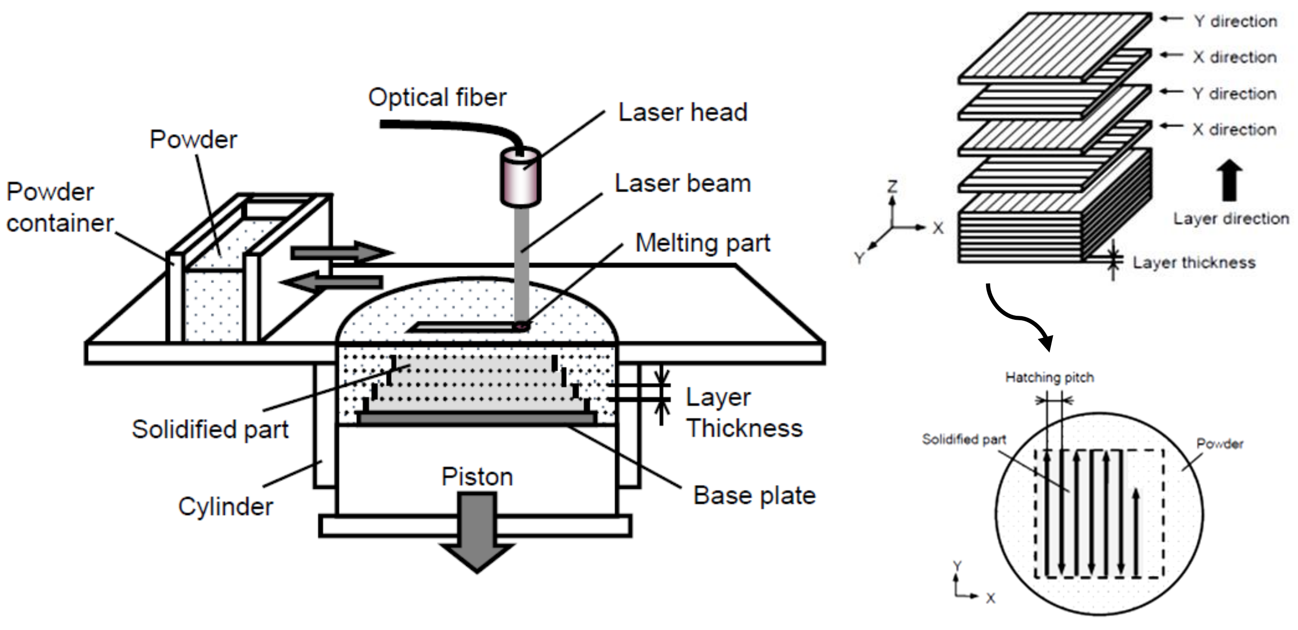

Figure 4: Schematic diagram of the facility used in the selective laser fusion process and its scanning method (Adapted, from Abe et al., 2017)

To avoid wasting raw materials not used during the process, the powder was removed from the equipment and reused. One of the advantages of SLM is the ability to manufacture parts with full density, mechanical properties comparable to materials produced on a large scale using conventional forging or casting methods, cryptic and custom geometry, parts with significant details, and better surface finish (Yadroitsev and Bertrand., 2010), (Ozsoy et al., 2021) and (Yap et al., 2015). However, they have disadvantage regarding low productivity and high cost of equipment/lasers.

3. A CRITICAL REVIEW

In this study, we analyze the properties of the NiTi alloys. It is an alloy with a shape-memory effect, which is widespread and easily accessible. We can find the parameters of the alloy in more detail in the experimental tests performed by (Oliveira.,2019) through additive manufacturing via selective laser melting in her dissertation.

According to (Oliveira.,2019), when analyzing images obtained using a Scanning Electron Microscope (SEM) one finds two results by using two different techniques. In Figure 5, the simple mixing of Ni powder and Ti powder occurred in a planetary mill without the use of balls, in which it can be observed that some Ni particles were in contact with the surface layer of the Ti particle, but did not enter owing to the having occurred a mechanical mixing with high energy. We also found isolated nickel and titanium particles, due to the absence of stainless-steel balls in the mixing process, which would cause more effective shocks and the breaking of particles, and as an effect, the interaction of nickel inside the titanium particle.

a) Magnification 150x b) Magnification 1000x c) Magnification 5000x d) Magnification 20000x

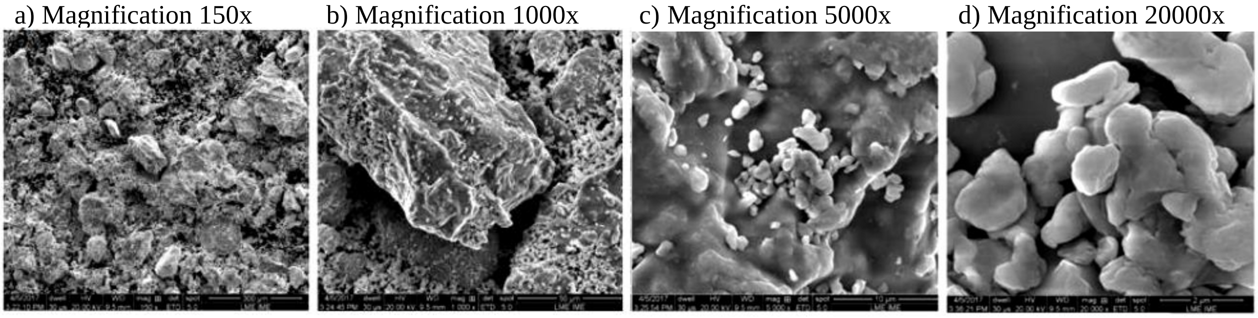

Figure 5: SEM of the NiTi alloy (Adapted, from Oliveira, 2019).

In Figure 6, the mechanical alloying was performed using a planetary mill and stainless-steel balls. It was verified the formation of agglomerated particles, without the presence of nickel on the surface of titanium particles. This fact could be associated to a more effective delamination due to the presence of the balls during the mixing process, thus causing plastic deformation and bonding of nickel and titanium powders (Oliveira, 2019).

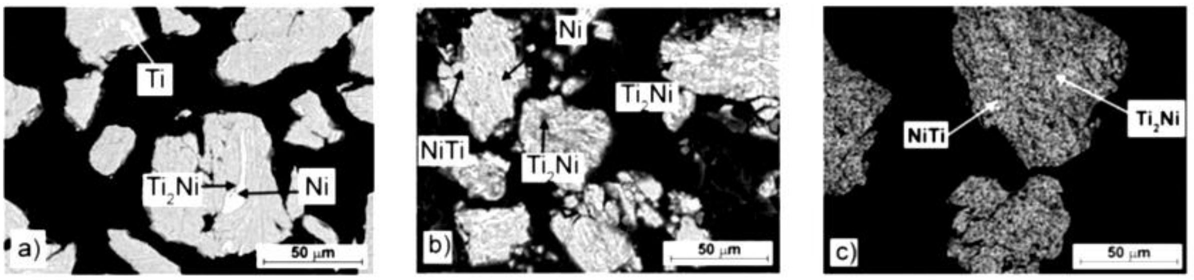

Figure 6: SEM of NiTi alloy using the mechanical alloying technique at 3 different times. a) 15 min b) 45 min c) 120 min (Novák et al., 2017).

During the irradiation process, part of the laser energy was absorbed by the powder and the other part was reflected. The absorbed energy melted the material generating a pool, and as the pool solidifies quickly, the substrate was ready to receive another layer. As this process repeats, the layers go through long cooling and heating cycles which directly affects the main characteristics of alloy transformations and functional properties of the part (Wang et al., 2018) and (Oliveira, 2019).

During the SLM process, the optical energy delivered needs to be high enough to generate dense parts, but enough to avoid common metallurgical problems such as pores and element losses. Therefore, it is important to understand the parameters related to the laser speed and beam diameter because it directly influences the quality of the built volume. At low speeds the beam remains longer in a given surface generating regions with molten pool material and irregular surfaces, damaging the subsequent layer, as can be seen in Fig. 7. According to (Haberland et al.,2014) the effects of the parameters can be delimited by energy density and scanning speed. This analysis becomes a processing chart, since high energy can be observed in the weld beads and tracks with low energy can be partially interrupted.

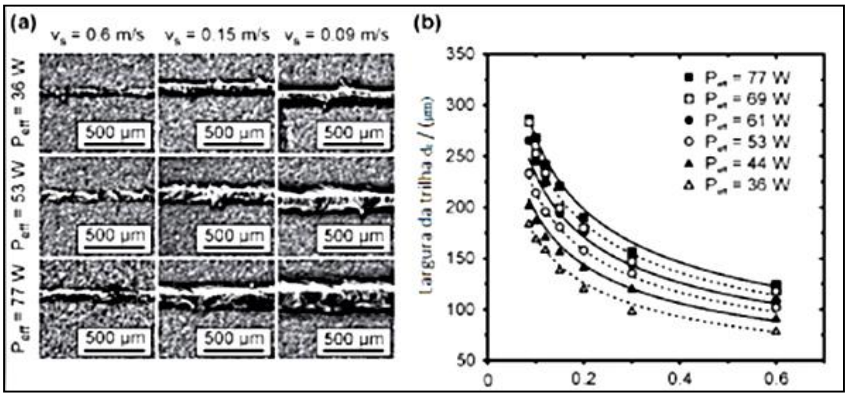

Figure 7: Micrograph and graph of NiTi trails by SLM (a) laser power (P_{eff}) vs scanning speed (V_s) and (b) trail width versus P_{eff} and V_s. (Haberland et al., 2014)

Another factor that causes cracking in the samples is the thermal gradient during the SLM process. One way to alleviate this problem is to heat the powder, but due to the oxidation of the NiTi alloy at high temperatures, an increase in cracking may occur; therefore, it is not a good alternative. Another relevant aspect is the type of substrate on which the sample was built influences the porosity and cracks in the samples (Oliveira., 2019). Figure 8, shows a cylindrical part printed using SLM from a simple mixture of nickel and titanium HDH powders. The laser power (P_{eff}) was 95W and energy density (ε) 21.32 J/mm³. Based on the external aspects evaluated by naked eye, it was possible to identify the presence of cracking and fragility points at the top and bottom of the cylinder. For more detail, we explore the work of (Oliveira.,2019).

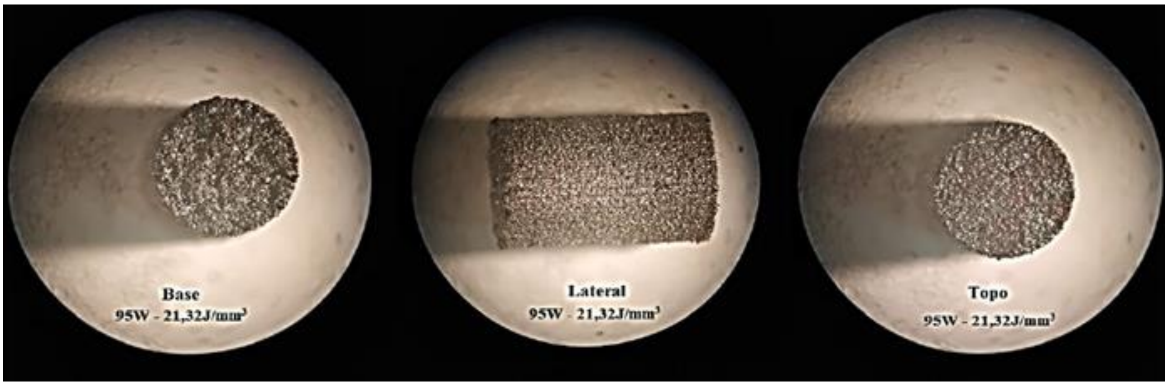

Figure 8: Optical images of a cylindrical part built via SLM of the base, side and top views with P_{eff} = 95W and ε = 21 J/mm³ (Oliveira, 2019). When evaluating the porosity of the cylinder via SLM, regions of the circular cross section were randomly collected for micrography testing at 50x magnification in the optical microscope, according to Fig. 9. In the analysis of the cylinder mentioned in the previous section, no relevant differences were identified in relation to the base region (1), central (2), and top regions (3). The pores were well distributed throughout the cylinder and no material was also found (Oliveira, 2019).

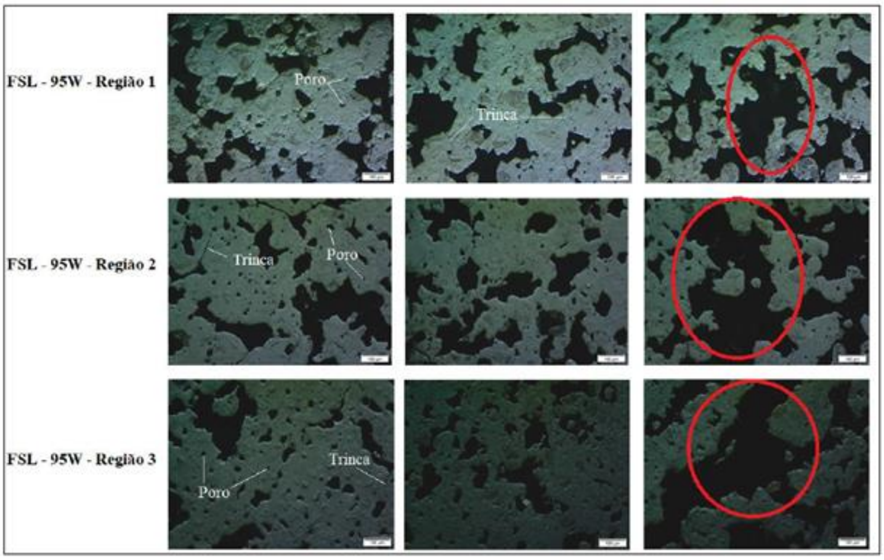

Figure 9: Optical microscopy images of the cylinder via SLM 95W-21.32J/mm3 highlighting cracks, pores and regions with absence of material (Oliveira, 2019). Through the superposition of the diffractogram results in selected conditions, it was possible to observe the presence of peaks of the elemental powders Ni and Ti, for more details refer to (Oliveira, 2019). The energy and interaction time were not sufficient for the formation of NiTi matrix phase, austenite B2, martensite B19' and neither phase R. The peaks evidenced the formation of the intermetallic expected in the Ni-Ti system which are: $NiTi_2$, Ni_3Ti_2, Ni_3Ti and Ni_4Ti_3 and formation of TiO_2 due to the presence of oxygen in the system (Oliveira, 2019).

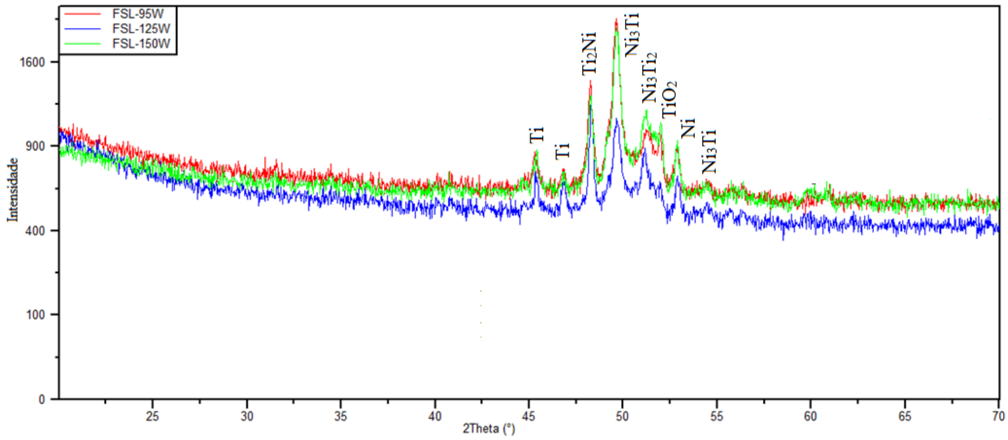

Figure 10: XRD of the cylinder via SLM at the selected conditions. P_{eff}: 95W, 125W, 150W (Oliveira, 2019).

4. CONCLUSIONS

After the tests performed, one can observe that some aspects of the single blended powder contributed to the absence of formation of the NiTi matrix alloy (as observed in the XRD test). The Ni particles were not adhered to the Ti particles and remaining isolated in their original state and Ni (Ni_3Ti and Ni_3Ti_2) and Ti (Ti_2Ni) rich precipitates appeared. For the selective laser melting technique, it was found that by increasing the energy density presented good results in reducing the pores, generating less cracks, increased hardness and with microstructure rich in Ni and Ti compared to other manufacturing routes.

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