

## **Secondes journées du GDR ALMA**

**INSA Rouen – GPM. Mardi 27 juin 2023 – Jeudi 29 juin 2023**

### **Résumés des interventions**





## Oral presentations – Invited contributions

**Mardi 27/06/2023**

### **1. Outstanding strain-hardening of new titanium-based alloys manufactured by laser power bed fusion**

Thierry Gloriant

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In this presentation, microstructural and mechanical properties of new NiTi alloy and Ti-22Zr-9Nb-2Sn metastable  $\beta$ -titanium alloy elaborated by laser power bed fusion process (L-PBF) will be presented. Both alloys are characterized by the fact that a stress-induced martensitic transformation occurs. By controlling the L-PBF process parameters, outstanding strain-hardening could be achieved, thanks the martensitic transformation occurring in the plastic domain.

**Mercredi 28/06/2023**

### **2. Large scale wire-based additive manufacture, latest and future developments**

Stewart Williams

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Wire based directed energy deposition has been the subject of much research over the last few years. This has been mostly done with standard off-the-shelf sources with limited control and deposition rate. Recently through the NEWAM project new processes have been designed featuring much higher levels of thermal and geometric control combined with higher build rates. These will be described, and their capabilities highlighted. This development has been supported with new developments in process modelling using CFD techniques. Future developments featuring the major challenge of more rapid and cheaper qualification will be described.



## Oral presentations

**Mardi 27/06/2023**

Première session. Mécanique, rupture

### 1. Mechanical Resistance assessment of 316L stainless steel additively-repaired structures

Baris Telmen<sup>1</sup>, Fabien Szymtka<sup>1</sup>, Anne-Lise Gloanec<sup>1</sup>, NicolasThurieau<sup>1</sup>, Gilles Rolland<sup>2</sup>, and David Haboussa<sup>2</sup>

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To quickly characterize the static and cyclic mechanical strength of a structure repaired by additive manufacturing, a specific specimen geometry and reparation protocol is developed. Repairs are performed using two processes (Cold Spray and Direct Energy Deposition) when specific role of adjustable parameters and post-treatment are analyzed. Tensile and self-heating tests are performed to assess the mechanical resistance of the repaired structures. The fundamental role of the microstructure in the vicinity of the repaired area in the initiation and propagation of cracks is highlighted and discussed.

### 2. On the fracture behavior of 316L stainless steel produced by laser powder bed fusion

Edouard de Sonis, Sylvain Dépinoy, Anne-Françoise Gourgues

Mines Paris Tech

The room temperature impact toughness and fracture toughness of 316L austenitic stainless steel manufactured by laser powder bed fusion has been studied in relationship with its microstructure. The effects of the grain size and morphology, of the dislocation structure and of the population of oxide nanoparticles were systematically investigated by using two different powders and heat treatments leading to stress-relieved, recovered, and recrystallized microstructures, respectively. A good correlation was found between impact toughness and fracture toughness, both during crack initiation and crack propagation. Both kinds of tests involved dislocation glide and mechanical twinning; ductile fracture originated from interfacial decohesion between the matrix and the nanoparticles. The anisotropy in both impact and fracture toughness originated from the grain shape and from some sensitivity to ductile intergranular fracture. Recrystallization dramatically decreased the resistance to crack propagation, due to the chemically unstable oxide particles that were now located at recrystallized grain boundaries.

### **3. Effect of scanning parameters on texture development and its consequences on the anisotropy of mechanical properties**

Denis Solas, Gabriella Tarantino, Thierry Baudin, Anne-Laure Helbert

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In additive manufacturing, texture development can be controlled by changing the scanning strategy of the laser. As the growth preference of the columnar cells is along the direction with the largest temperature gradient, the texture can also be modified by changing the shape of the melt-pool which mainly depend on the power and scanning speed of the laser. In this work, the influence of processing parameters on texture development in nickel samples is investigated. From texture measurements, an elasto-plastic self-consistent model is used to simulate the anisotropy of mechanical properties.

The resulting mechanical properties of homogeneous specimens with controlled textures are then measured experimentally under tensile loadings. The tensile deformation and damage evolution are quantified via clip-on gauge extensometers and the measured anisotropic properties are compared with predictions of the self-consistent model.

### **4. Effect of chemical microsegregation on the hot cracking sensitivity of nickel-based superalloys manufactured by L-PBF**

Elisa Borges Mendonça<sup>1,2</sup>, Sylvain Dépinoy<sup>1</sup>, Christophe Colin<sup>1</sup>, Marie-Hélène Berger<sup>1</sup>, Nicolas Leriche<sup>2</sup>, Fabien Cuvilly<sup>3</sup>.

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Nickel-based superalloys, among which Inconel 738 and René 77, are used in the aeronautic engines due to their good mechanical properties at high temperatures. To reduce the weight of these components without reducing their performance, Safran aims at using additive manufacturing processes, such as laser powder bed fusion (L-PBF), to optimize their geometry. Nevertheless, one drawback of this process is the high cooling rate during solidification, leading to hot cracking in non-weldable nickel-based superalloys. In the literature, the segregation of boron, zirconium or silicon during solidification, as well as specific misorientations between solidified grains, are often reported as factors enhancing the hot cracking phenomenon. The aim of this study is to determine which factor is predominant based on comparisons between Inconel 738 and René 77 manufactured with different processing conditions. The resulting microstructures, corresponding to different solidification conditions, were characterized by TEM, SEM, APT and EBSD. Microsegregation profiles were determined by STEM-EDX analyses, based on a suitable reconstruction protocol. Other microstructural features of interest are the dendrite size, precipitation in the inter- and intra-dendritic areas, the crystallographic

texture and the grain boundary misorientations. The relationship between these microstructural features and the crack density in the as-built state is then discussed.

## **5. Influence of volume energy density in additive manufacturing and temperature on the strain hardening mechanisms of L-PBF Ni20Cr alloy**

Shubham Sanjay Joshi<sup>1</sup>, Clément Keller<sup>2</sup>, Williams Lefebvre<sup>1</sup>, Eric Hug<sup>3</sup>

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Additive Manufacturing (AM) brings about an array of modifications in microstructure with respect to conventional routes transforming mechanical performances. These new microstructure features depend on process parameters and especially on volume energy-density delivered by the laser on powder layer. Among the different alloys manufactured by AM, Ni-alloys exhibit high-strength and fatigue-resistance at elevated temperature opening the way of fabrication of gas turbines and jet-engine components. AM-process influences straining mechanisms and research has been done to connect these mechanisms to different microstructural-features like dendrites, precipitates, etc. Ni-superalloys experience precipitation hardening due to the formation of  $\gamma'$  and  $\gamma''$  phases. However high fraction of  $\gamma'$  makes the parts susceptible to hot-cracking during process which could prevent their application in industry. To better study the elementary microstructural mechanisms in Ni-alloys, a theoretically monophasic and binary Ni20Cr-alloy manufactured by laser powder-bed fusion was studied in this work. Samples were built with several volume energy-densities leading to specific microstructures. Kocks-Mecking modelling was employed to identify the most impactful microstructural features influencing enhanced Yield stress in AM and investigate strain hardening mechanisms for AM samples prepared by different volumetric energies. Dendrites were found to be the main contributing factor in hardening at room temperature. Similar trend was detected until 700 °C with lower flow stress values and contribution of dendrites simply owing to their thermal stability. For higher temperature levels (900 °C), the work-hardening of the alloys progressively vanishes, indicating the deletion of dendrites and the homogenization of the microstructure.

Deuxième session. Procédés, microstructures

## **1. Development of nickel based functionally graded material by Laser Metal Deposition**

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Functionally Graded Materials (FGMs) have recently gained significant attention due to their gradual properties and composition. With its multiple feeders and its ability to control the amount of delivered powder, the Laser Metal Deposition process is a promising technology for producing FGMs. This study emphasizes the importance of parametric analysis to produce tailored material properties and proposes simulation and experimental characterization techniques to develop specific heat treatments.

## **2. Processing of CoCrMo alloy samples with bimodal grain size distribution by Composite Extrusion Modeling**

Lucía García de la Cruz<sup>a</sup>, Paula Alvaredo<sup>a</sup>, José Manuel Torralba<sup>a,b</sup>, Mónica Campos<sup>a</sup>

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CoCrMo alloy is commonly used in biomedical applications for its biocompatibility, its resistance to corrosion and its good mechanical properties. The manufacturing methods of the implants processed with such material require the direct production of the desired geometries without the need for subsequent machining. With Composite Extrusion Modeling (CEM) complex geometries can be modelled and printed from optimized feedstock.

In a previous work, commercial CoCrMo powder was used, combined with paraffin and high-density polyethylene as binders, to successfully process samples with a relative density,  $\rho_{rel} > 0.96$ . The current work, investigates the possibility of combining the commercial powder with a nanostructured one (obtained by ball-milling) to produce a bimodal microstructure, thus improving the mechanical properties of the processed samples.



### **3. Material Extrusion (MEX) Additive Manufacturing using MIM feedstocks: a versatile technique for the Additive Manufacturing of various metallic parts.**

Gurminder Singh<sup>1,2</sup>, Swathi Manchili<sup>1</sup>, Luis Olmos<sup>3</sup>, Jean-Michel Missiaen<sup>1</sup>, Didier Bouvard<sup>1</sup>, Jean-Marc Chaix<sup>1</sup>

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Sinter-based additive manufacturing techniques, such as binder jetting or stereolithography have gained increasing interest in recent years. In these techniques, the 3D part is printed in the green state and must further be sintered by a high temperature treatment. Despite the 2-step process, these techniques can be attractive, because they generally use cheaper printing machines and also because they are more in-line with the controlled industrial processes in Powder Metallurgy. Among these processes, Material Extrusion (MEX) Additive Manufacturing using Metal Injection Molding (MIM) feedstocks holds a special place. In this technique, MIM granules are extruded through the nozzle of a printer to build the 3D part. The process benefits from the passed development of the MIM industry and industrial companies in the sector can easily make the conversion to diversify their applications. Generally speaking, the use of powder-polymer granules which are optimized for powder forming makes it possible to efficiently crush the filament during printing, thus reaching a green density of the metallic material in the printed part as high as 60%. The further debinding and sintering steps are basically the ones specified for MIM parts and density higher than 95% can typically be achieved for the sintered material. This presentation will show a few applications of this technique for processing of copper, steel, aluminium or titanium alloy parts.

#### Troisième session. Microstructures

##### **1. Atom probe tomography investigations of as-built and heat-treated microstructures of Al-based alloys processed by laser powder bed fusion**

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Micro and nanostructures inherited from laser powder bed fusion (LPBF), since they strongly differ from classical microstructures produced by conventional casting process, require detailed investigations at various scales to clarify the process-microstructure-property relationships of Al-based alloys processed by LPBF. In the framework of a multiscale characterization approach, Atom Probe Tomography enables to access 3D fields of composition at the nanometre scale. It is hence possible to first quantify the degree of supersaturation of Al solid solution in the as-built condition and to later follow the kinetics of decomposition of this

solution as heat treatments are applied. On this basis, various heat treatment strategies can be foreseen.

## **2. Three-dimensional modelling of solidification grain structures generated by laser powder bed fusion - Application to Inconel 718 Nickel-based superalloy**

Théophile Camus<sup>(1,2)</sup>, Gildas Guillemot<sup>(1)</sup>, Charles-André Gandin<sup>(1)</sup>, Oriane Senninger<sup>(1)</sup>, Daniel Maisonnette<sup>(2)</sup>, Oriane Baulin<sup>(2)</sup>

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Inconel alloys are of clear interest for a wide range of industrial applications regarding their tensile strength and creep resistance at high temperature. Nevertheless, due to their properties, these alloys are also difficult to shape by casting or machining processes, notably for complex geometries. An interest has emerged to apply L-PBF additive manufacturing forming methods on these materials to develop near net shape components and particularly on the Inconel 718 alloy. However, microstructures developed during forming processes have also large influence on final properties. Consequently, optimized scanning strategies have to be identified to control and master grain evolution in L-PBF during solidification stage. In this objective, a model based on a Cellular Automaton (CA) approach has been applied to follow microstructural development during L-PBF forming process. In addition, a new hybrid methodology is proposed with the benefit of a full thermohydraulic simulations predicting both track shape and temperature field in steady state regime. The main advantage of this innovative approach is to reach unprecedented sizes of simulation domains while still preserving a full numerical solution at the melt pool scale and fine microstructure description. The other advantage is the possibility to easily vary process and material parameters to approach industrial practices with limited computing time. Various scanning strategy have been thereafter analyzed and discussed regarding their effect on microstructure characteristics. This strategy should pave the way to future investigation of process parameter effects and associated prediction of metallurgical properties.

## **3. Laser powder bed fusion of small struts. Influence of the scanning conditions and the part size on the microstructures and mechanical resistance.**

Julien Rodrigues Da Silva<sup>1,2</sup>, Anne-Laure Helbert<sup>2</sup>, Zehoua Hamouche<sup>1</sup>, Corinne Dupuy<sup>1</sup>, Thierry Baudin<sup>2</sup>, Patrice Peyre<sup>1</sup>

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Laser powder bed fusion (L-PBF) has recently achieved a stage of industrial maturity for dense intermediate-sized parts (10 mm<sup>3</sup> to 40 cm<sup>3</sup>). However, studies on smaller parts remain poorly

documented, especially for struts with small diameters<sup>1</sup> (<2 mm) which are a basic component of any lattice structure.

The aim of this study is to understand the size-effects alongside the L-PBF process parameters on the microstructural properties of struts made of Inconel 625, with the combined use of an instrumented L-PBF bench and an industrial L-PBF machine.

The work consists of evaluating possible correlations between process parameters (scan strategy), strut's diameter (between 0.2 mm and 2 mm), the melt-pool size, the local heating-cooling cycles and resulting microstructures and mechanical resistance. For this purpose, a large number of struts were manufactured with L-PBF and analysed to provide a realistic estimation of microstructures and tensile strengths. In a second step, using in-situ high-speed imaging, melted zones were investigated and compared with a simplified numerical model in order to provide a deeper understanding on post-mortem results.

Initial results suggest a strong link between the size of melt zone and the morphological and crystallographic texture of objects, especially for small struts. Moreover, the model reveals a correlation between the melt zone shape and microstructures such as grains or solidification cells size, particularly in relation to scanning strategies. These results have important implications for optimizing the L-PBF process applied to structure lattices, and achieving desired properties in the final product. Altogether, the combined approach offers a powerful tool for improving the quality and efficiency of L-PBF manufacturing of little objects.

### **Mercredi 28/06/2023**

#### Quatrième session. Aspects numériques

#### **1. Speeding up mesoscale thermo-mechanical simulations of powder bed fusion: focus on temporal discretization and spatial adaptivity**

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Geometric distortion of AM metallic parts is a major concern in the industry. It may stop the manufacturing, damage the AM machine and cause parts to fall out of dimensional tolerances. Therefore, a costly trial-and-error procedure is performed before finding the optimal set of manufacturing parameters, such as laser power, laser speed or hatch distance. Reliable simulations of AM processes are thus essential to reduce the time required to print the first part correctly.

Part-scale distortions are routinely computed with macroscopic simulation strategies. They provide reasonable results with elementary structures (i.e., beam-like or plate-like) but can fail as soon as geometries become more complex. Therefore, the use of the so-called mesoscale conduction-based strategies seems mandatory for accurate results. These models employ realistic heat sources and scanning paths but neglect fluid dynamics to lower computational

costs. Yet, they are considered too expensive to be applied at the part-scale. We will show how proper temporal discretizations and spatial adaptivity schemes can reduce their cost.

## **2. Two-scale modeling of the WAAM process. Link between thermo-hydrodynamics and mechanics**

Clément Le Falher (1,2,3), Simon Morville (1), Stephen Cadiou (3), Mickael Courtois (3), Philippe Le Masson (3), Pascal Paillard (2)

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Simulation of arc-wire additive manufacturing is under development in many academic and industrial sectors and at different scales. The study at the melt pool scale is often interesting to study deposition shapes and defects but not very relevant to calculate the stresses and strains at the part scale. On the opposite, large scale mechanical modeling often relies on strong assumptions about the heat input which often generates either errors or costly and time consuming source calibration campaigns. In this paper, we will present a multiscale approach based on a thermo-hydrodynamic calculation allowing an accurate knowledge of the temperature field. This temperature field is then directly transferred to a larger mechanical model at the scale of the part, thus avoiding the determination of an equivalent heat source. This is solved in the finite element code Comsol Multiphysics. The principle and methodology will be discussed on a case of manufacturing a stainless steel part.

## **3. Development of a new multi-scale adaptive remeshing approach for the description of strong local gradients in order to predict defects in the whole part during LPBF Additive Manufacturing**

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The main challenge for numerical simulation of the whole part in additive manufacturing is to describe very local phenomena ( $<0.1$  ms) over a very long physical time ( $>48$ h). Multiscale numerical models or those reducing physical equations (e.g., purely mechanical models based on inherent strain) have been used to predict defects in the whole part such as porosities which are related to supercooling/subcooling, and distortion of parts during their manufacture. These models are still expensive in terms of computation time and are not very accurate, especially because of an almost constant mesh density in the meshed part. The model developed in this work uses a new multi-scale adaptive remeshing approach and the finite volume method to predict local gradients in LPBF. The building is done by layer-by-layer activation of maximal

refined voxel elements. As the construction progresses, some areas are redefined or refined according to the criteria allowing to better describe the local gradients while reducing the computing time. The problem is solved at several scales corresponding to the different levels of refinement of the mesh. The approach is used to simulate the distortion of additive manufactured parts by the inherent strain but it could be used for thermal or even thermomechanical problems.

#### **4. An efficient FEM approach for simulating metal deposition geometry achieved by LMD-p process**

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An original formulation is proposed in order to simulate molten metal deposition geometry with a very low CPU time. The FEM simulation strategy is composed of a 2-step resolution algorithm at each time step of the deposition process. At first, the 2D mesh of the surface inflates according to the volume flux of material added. The balance of stress is then treated on the same 2D mesh by considering the interaction between the curvature effect of the surface tension and the pressure difference between the molten zone and the atmosphere which is assumed to be nearly constant. Applications are proposed to show the interest of the numerical method developed in terms of computation efficiency and quality of geometry evolutions.

#### Cinquième session. Mécanique, fatigue

##### **1. Role of defects and microstructure on the fatigue durability of Inconel 718 obtained by Additive Layer Manufacturing route**

Cesar-Moises Sanchez-Camargo<sup>1</sup>, Yves Nadot<sup>1</sup>, Jonathan Cormier<sup>1</sup>, Fabien Lefebvre<sup>2</sup>, Wen Hao Kan<sup>3</sup>, Louis Ngai Sum Chiu<sup>3</sup>, Chen Li<sup>3</sup> et Aijun Huang<sup>3</sup>

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A Laser Powder Bed Fusion Inconel 718 produced with intentionally non optimized parameters is investigated to elucidate the role of defects and microstructure on the High Cycle Fatigue life. Samples coming from three build orientations and three heat treatments are tested at room temperature and at 650 °C under R = 0.1 load ratio. Results show that the printing orientation has a minor effect on fatigue life at both room and high temperatures. Most samples failed from

Lack of Fusion type defects (ranging from 200  $\mu\text{m}$  to 600  $\mu\text{m}$ ) and each defect size is carefully analyzed by using SEM observations. It can be observed that surface defect leads to transgranular fracture surface when sub-surface initiation leads to rather crystallographic type fracture surface. By producing S-N curve on Hot Isostatic Pressure condition, it is shown that the relative influence of defect on the S-N curve is much higher at 20°C compared to 650°C. Kitagawa diagram are plotted for both temperatures in order to quantify the influence of defect size on the fatigue limit. Natural cracks are observed at the surface in order to discuss the relative part of initiation life in the whole fatigue life.

## **2. Role of surface roughness, defects, residual stresses, and local microstructure on the fatigue strength of Laser Powder Bed Fusion (L-PBF) 316L stainless steel**

Marion Auffray<sup>1,2</sup>, Pierre Merot<sup>1,2</sup>, Franck Morel<sup>1</sup>, Etienne Pessard<sup>1</sup>, Linamaria Gallegos Mayorga<sup>1</sup>, Paul Buttin<sup>2</sup>, et Thierry Baffie<sup>3</sup>

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- Fatigue tests are carried out on 8 different batches, considering various microstructural (as-built, heat-treated) and surface conditions (net-shape with varying contour-scan strategy, pre-corroded net-shape, polished, pre-corroded polished, single-artificial-defect polished).
- Studied defects are natural process-induced defects (lack of fusion, spatter, gas pore, hole, surface micro geometry) and artificial surface defects (corrosion pit, electric discharge machined defect). Their size is between 15 and 700  $\mu\text{m}$ .
- Residual stresses seem to be the most influential factor on the fatigue strength of net-shape L-PBF 316L over other surface parameters.
- Local microstructure seems to play a minor role for predicting fatigue life of polished L-PBF 316L.
- We are still investigating the relative effects of surface roughness, defects and microstructure near the surface and surrounding defects.

## **3. Anisotropy of behavior and fatigue crack growth in the material IN718 L-PBF**

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The Laser Powder Bed Fusion (L-PBF) process produces oriented microstructures. Although it is now possible to optimise the process parameters to reduce the porosity of many superalloys, this oriented microstructure can still be used. In particular, some industrial applications (lattice

structure or thin-walled tubes) limit the use of heat treatments to eliminate the anisotropy induced by these microstructures. On the other hand, from a more academic point of view, the L-PBF process can be used to generate highly oriented microstructures, approaching directed solidification (DS) materials. The aim of this study is to characterise the role of strong anisotropy on fatigue crack growth for a superalloy produced by additive manufacturing.

We focus on the case of INCO718 superalloy, produced by L-PBF in the presence of an oriented structure, for a direct aging treatment. Two batches of specimens were studied, highlighting different crystallographic textures representative of the variations of microstructures potentially present in real parts: we go from an alloy close to a DS (a single crystallographic orientation preferred) to a more isotropic alloy, retaining elongated grains in the direction of fabrication.

The cyclic mechanical behaviour is strongly anisotropic and is effectively modelled by orthotropic elastic behaviour, nonlinear kinematic strain hardening associated with a Hill criterion. Crack growth measurements made in situ at high temperature show that cracks propagate preferentially along grain boundaries, irrespective of crystallographic orientation. The finite element method was used to account for the complex 3D crack paths. To determine the driving forces for crack propagation, the J-integral evaluated by the G- $\theta$  method is used for such cracks with anisotropic behaviour.

Thus, an intrinsic fatigue crack propagation rate can be characterised for an intergranular cracking mechanism. On the other hand, transgranular cracking was observed for loads applied in the direction of manufacture. In this case, the crystallographic texture appears to be a first order factor governing the fatigue crack growth rate. This study reviews a synthesis of the key parameters of crack growth analysis for a highly anisotropic material.

## Sixième session. Microstructures, propriétés

### **1. Development of $\gamma/\gamma'$ hardened cobalt-based superalloys by laser additive manufacturing**

Thibaut Froeliger<sup>1</sup>, Louise Toualbi<sup>1</sup>, Didier Locq<sup>1</sup>, Rémy Dendievel<sup>2</sup>

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This work focuses on the elaboration of two-phase  $\gamma/\gamma'$  cobalt-based superalloys by Laser Directed Energy Deposition (L-DED). Due to their complex chemical composition, these alloys are sensitive to liquid phase cracking mechanisms during additive manufacturing. In order to elaborate crack-free alloys, the causes of the cracking mechanism were identified by various characterization methods (EDS, WDS, EELS, ASTAR, etc.). The role of microstructure and minor elements on the sensitivity to cracking was highlighted, which allowed the development of processing strategies leading to crack-free alloys by DED. This includes in particular chemical composition adjustments but also the analysis of the thermal history of the process leading to new deposition strategies.

## 2. Influence of the microstructure of additively manufactured Ni20Cr microstructure on aging under irradiation

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The metal additive manufacturing is considered in the nuclear field, for example to replace outdated parts. However, the microstructure of additive manufactured parts is particular. For example, the formation of precipitates or a structural anisotropy due to columnar grain growth are often observed. These differences can strongly affect the properties of the matter, so it is necessary to investigate the response under irradiation. After a brief description of the evolution of metallic alloys under irradiation and, the use of ion beam to simulate neutron will be discussed. Then, the evolution of microstructure of a Ni20Cr under irradiation will be described for different conditions.

Defects induced by irradiation are characterized by Transmission Electronic Microscopy (TEM). Observations in “two-beam” conditions highlight that irradiation mainly induce the formation of Frank dislocation loops. The evolution of these defects is followed by measuring their density and size.

## 3. Fast model of DED from thermal analysis to microstructure, and part-scale residual stress estimation

Daniel Weisz-Patrault<sup>1</sup>, Quentin Dollé<sup>2</sup>, Michèle Bréhier<sup>3</sup>, Laurane Preumont<sup>1</sup>, Rafael Viano<sup>4</sup>

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Directed energy deposition additive manufacturing (DED) gathers several processes (e.g., laser metal powder directed energy deposition (LMPDED), wire arc additive manufacturing (WAAM) or wire laser additive manufacturing (WLAM)). Extreme computation costs are associated to comprehensive models dealing with the interaction between the heat source and the deposited matter, the hydrodynamic problem taking place in the melt pool, crystallization with competitive grain growth during fast solidification, solid state evolution of the microstructure through thermal cycling, and part-scale residual stresses. In practice one can only carry out such simulation for single beads.

In view to better control either the microstructure or large scale residual stresses resulting from the process, fast modeling strategies have been developed and tested against in-situ measurements. These models are able to simulate the temperature field history in the entire part,



to determine a simplified melt pool geometry, which in turn leads to the final microstructure by taking into account competitive dendritic growth, and to address solid state phase transformations (e.g., diffusion of alloying elements). Furthermore, residual stresses are computed thanks to a new uni-dimensional enriched model in order to capture the stress field complexity while limiting computational cost.

### Septième session. Procédés, durabilité

#### **1. Optimisation of lithography-based metal additive manufacturing of 316L complex parts: from metal powders to sintered parts**

Xavier Boulnat, G. Bonnard, Eric Maire

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We will present a sinter-based additive manufacturing technique called (stereo)lithography metal manufacturing. Initially developed for ceramics, this technology uses the principle of photopolymerization, where metal powder is homogeneously dispersed in a light-sensitive resin and selectively polymerized by exposure with light. The feedstock, which consists in a blend of metal powders mixed with polymeric binders, is applied layer by layer with a coating blade during the printing process. This composition allows for a wide range of metals to be processed, from 316L stainless steel to titanium, copper or precious alloys. Once the parts are printed, they undergo a cleaning process followed by a thermal debinding/sintering step to achieve the final properties of the metallic parts.

In this presentation, we will follow the dimensional and microstructural evolution of the part, from the green (printed) part to the sintered part. First, the effect of printing processes, mainly layer thickness and incident energy, on the green part properties was investigated. The pore distribution was quantified by X-Ray microtomography whereas the strength, determined by 3-point bending tests, was linked to the progress of photopolymerisation using Fourier-transform Infrared spectroscopy (FTIR). Then, the thermal debinding behavior under various gaz atmospheres was determined by thermogravimetric (TGA). The debinding process leads to a competition between the full degradation of the polymeric binder and the oxidation of the metal powders, both increasing with temperature. Finally, sintering was assessed by thermomechanical dilatometric analysis and a multi-scale characterization of the sintered parts. Fully dense parts could be achieved, resulting in a fine austenitic microstructure decorated by delta ferrite. A low carbon content, critical for 316L alloy, confirmed the full degradation of the inorganic binder. This microstructure and density lead to an excellent strength/ductility trade-off of the sintered parts. This optimized process was then used to print complex parts to quantify the anisotropic shrinkage behavior 3D by X-Ray microtomography.

## **2. Impact of the microstructure of a Ni20Cr alloy produced by additive manufacturing on the electrochemical corrosion resistance**

Lydie Mas<sup>1</sup>, Alexis Dujarrier<sup>2</sup>, Philippe Marie<sup>2</sup>, Mamour Sall<sup>2</sup>, Isabelle Monnet<sup>2</sup>, Eric Hug<sup>1</sup>

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Ni20Cr is a binary alloy used for their resistance in aggressive media. Its electrochemical behavior is well known in various electrolyte such as NaCl, H<sub>2</sub>SO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, and borate buffer solution. In this work, the corrosion and the passive behavior in a neutral (Na<sub>2</sub>SO<sub>4</sub>) and chloride (NaCl) environment of a Ni-20Cr alloy elaborated by Laser Power Bed Fusion (LPBF) with different building energies and strategies is compared with the conventional as cast material. The impact of the microstructure generated by additive manufacturing on the corrosion and the passive film behavior is studied thanks to polarization and electrochemical impedance spectroscopy measurements. Moreover, Mott-Schottky analysis is performed to obtain the semiconductor properties of the passive film. The native passive film thickness is evaluated by ellipsometry experiments and compared with Transmission Electron Microscopy observations and results issued from electrochemical experiments. LPBF Ni20Cr alloy exhibits a similar corrosion behavior than the as cast counterpart in Na<sub>2</sub>SO<sub>4</sub> but clearly better performances in NaCl electrolyte. Electrical properties of the passive layer are not very impacted by additive manufacturing, but ellipsometry measurements reveals a thicker passive film in LPBF samples. This thicker oxide layer is responsible of the better properties of additive manufacturing Ni20Cr in chloride environment.

## **3. Powder Bed Additive Manufacturing of alloy 718 – development of dedicated post-fabrication heat treatments**

Bertrand Max

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An increasing number of industrial parts is produced by additive manufacturing and their use is getting closer and closer to critical applications. As the most widely used wrought and cast superalloy, alloy 718 is an excellent legacy alloy for additive manufacturing. Nevertheless, direct transposition of heat treatments specifications developed for conventionally processed alloy is not the most effective to reach the highest properties when performed Laser Beam Melted parts. The presentation describes an approach to better understand the effects of different steps of post-LBM heat treatment sequences, including multi-scale microstructural characterizations and mechanical properties.

#### 4. Study of powder blend behavior and its application to the PBF-LB process

Paul Bourot<sup>a</sup> ; Laurent Weiss<sup>a</sup> ; Didier Boisselier<sup>b</sup> ; Pascal Laheurte<sup>a</sup>

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Cost per part is one of the main impediments to AM adoption, and the need for cheaper Laser Powder Bed Fusion manufactured parts is pulling for a reduction in manufacturing time and material cost. One of the current ways to increase building speed is to increase the layer thickness, while adapting other manufacturing parameters. This approach offers the opportunity to use coarser particle size to value a larger share of the gas atomization produced powder. However, a coarse particle size distribution is known to induce a low packing density of the powder bed. This results in lack of fusion defects in the part which are common due to uncomplete melting and low compacity powder bed. To mitigate this effect, the use of monomaterial blended powder, presenting a bimodal size distribution is investigated. A powder blend is composed of two powders with different sizes, blended with a defined weight ratio. First, different blends, with different sizes and weight ratios are prepared and characterized regarding their densities and flow behavior. Based on this ex-situ characterization, powder blends are identified for powder spreading and powder bed characterizations. Second, the powder spreading process is studied through the measurement of the coverage rate and deposited layer height. This method aims to further identify suitable powder blend for the PBF-LB process. Lastly, samples are printed from powder blend and characterized regarding their density and defects counts/type. Experiments shows a density above 99% for a layer thickness of 150  $\mu\text{m}$ , with lack of fusion remaining in the samples. This study shows the interest of using blended powders for high layer thickness and the associated increase in building speed.

**Jeudi 29/06/2023. Demi - journée couplée**

**GT1 (Interactions Energie-Matière) – TT1 (Caractérisations, Grands Instruments)**

Huitième session

#### 1. Temperature distribution and weld pool dynamic in WAAM transfer

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Laboratoire de Mécanique et Génie Civil UMR5508 de l'Université de Montpellier

Quality and geometry of bead in parts obtained by WAAM Process depend of heat transfer during the mass transfer. In this presentation, we will present in-situ temperature measurement to analyze transient effect during the deposit.

## **2. Wetting and spreading of a binder droplet on a powder bed: towards understanding the dimensional inaccuracies of parts manufactured by Metal Binder Jetting**

Agnès Schnell<sup>1,2</sup>, Christophe Colin<sup>1</sup>, Benjamin Sangouard<sup>2</sup>

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Metal Binder Jetting (MBJ) is an indirect and non-melting powder bed additive manufacturing process. MBJ involves selectively jetting a binder, composed of a polymer and solvents, layer-by-layer onto the powder bed. The binder infiltrates between the particles, which aggregate and form the shape of the final part. The "job box", containing the printed part and free powder, is then placed in a curing oven where polymerization and solvent evaporation occur. This results in a so-called "green" part which, after depowdering, is placed in a debinding furnace to remove any trace of polymer. Once debinding is complete, the resulting "brown" part is very porous and its mechanical properties are weak. Gradual thermal treatments (sintering) are required to obtain a strong and dense part.

The interaction of the binder droplets with the powder affects the properties of the "green" part and the quality of the final part obtained by MBJ. Through pendant drop tests performed on Inconel 718 powder, the wetting and spreading of the drop on the powder bed before and during infiltration were studied. The following four stages were identified: (i) after returning to equilibrium following impact, the binder droplet wets the surface of the powder bed, (ii) a pure spreading regime is then established, followed by (iii) a mixed regime of spreading and infiltration, which finally leads to (iv) a pure infiltration regime within the powder bed. The effect of the porosity of the powder bed on binder/powder interactions was investigated. A kinetic model of droplet spreading was established on a porous medium with variable density. In addition, the study of the wetting angle of the droplet on the powder bed surface confirmed that the droplet takes the form of a spherical cap only during the pure spreading regime.

## **3. Instrumented benches for additive manufacturing process monitoring and quality assurance**

Robin Kromer, Eric Lacoste, Corinne Arvieu

Université de Bordeaux - IUT Département GMP, Institut de Mécanique et d'Ingénierie de Bordeaux (I2M) - UMR 5295

The presentation focuses on metal additive manufacturing, specifically the Laser Powder Bed Fusion (LPBF) and Wire Arc Additive Manufacturing (WAAM) processes. I am involved in the development of instrumented benches to measure the additive manufacturing processes in-situ. These measurements are used to aid decision-making during manufacturing and to assess the material health of the parts being produced. We are also involved in the development of property ranges for the manufactured parts, taking into account their orientation, support structures used, and necessary finishing. To achieve this, we use predictive models that take

into account the topology of the parts, in order to optimize their design and manufacturing. Perspectives will be proposed to ensure quality results at an industrial scale.

#### **4. Contribution of instrumentation to the understanding and control of the LPBF process**

Pierre Lapouge, Patrice Peyre, Matthieu Schneider, Corinne Dupuis, Frédéric Coste, Morgan Dal

PIMM, Arts et Metiers Institute of Technology, CNRS, Cnam, HESAM Université, 151 Boulevard de l'Hopital, Paris 75013, France

Laser Powder Bed Fusion (LPBF) is the most developed additive manufacturing process for metals. The laser interaction with the metal during the process is focused on a small area ( $\varnothing < 100 \mu\text{m}$ ) and last over a few tens of ms resulting in complex hydrodynamic phenomena. On the other hand, a build can reach dimensions of several tens of cm with durations exceeding several days. The instrumentation of the process serves two complementary purposes: 1) providing experimental data at a fundamental level to understand and model the interaction, 2) monitoring the process to control the quality of the build. The same sensors and setups will not be able to achieve at the same time a high resolution of the interaction, spatial and temporal, and a complete monitoring of a build. Hence depending on the objectives of the studies, different trade-off can be chosen leading to the development of different experimental benches. This talk will introduce the developments done at the PIMM laboratory over the last few years through some notable studies.

#### Neuvième session

##### **1. New insight into additive manufacturing with multi-scale resolution synchrotron x-ray tomography**

Camille Pauzon, Pierre Lhuissier, Rémi Daudin, Guilhem Martin, Maxence Buttard, Jean-Jacques Blandin

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The maturity level and robustness of Additive Manufacturing technologies, and especially LPBF, are primarily bound to the current understanding of the many process parameters and the complexity of the obtained microstructures, which is also inherent to the part design. Examples of perspectives offered by synchrotron X-ray tomography to narrow this knowledge gap will be presented. In particular, nano-tomography permits to dive into the microstructures at the melt pool scale and document their evolution upon heat treatment, while micron-scale resolution enables to reveal the presence of defects. In addition, the design of miniature process replicator brings new perspectives for in situ measurement, e.g. defect tracking, and thereby to address aspects such as scanning strategy optimisation. Finally, the combination of micron-

scale resolution and large sample sizes on the ESRF's beamline BM18 offers new perspectives for geometry inspection of complex and *designed for AM* components.

## 2. Do dislocations evolve during additive manufacturing? – A synchrotron X-ray diffraction study

S. Gaudez<sup>1</sup>, K. A. Abdesselam<sup>1</sup>, H. Gharbi<sup>1</sup>, Z. Hegedüs<sup>2</sup>, U. Lienert<sup>2</sup>, W. Pantleon<sup>3</sup>, M. V. Upadhyay<sup>1</sup>

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During additive manufacturing (AM) of alloys, just after melting of feedstock, the material rapidly solidifies. Then, for the remaining build time, it is subjected to heating-cooling cycles in the solid-state i.e., solid-state thermal cycling (SSTC), at varying temperature amplitudes and rates. These highly non-equilibrium processes together determine the microstructure of as-built alloys, which exhibit heterogeneities at multiple length scales.

Amongst the different possible microstructural features, dislocations are ubiquitous in any as-built alloy. They mainly organize in intragranular dislocation cells with a high dislocation density in the cell walls and a low density in the cell interiors. Currently, there are two main hypotheses on their origin in as-built alloys. One hypothesis is that they form in inter-dendritic regions during rapid solidification and do not significantly evolve, if at all, during SSTC. The opposing hypothesis is that thermo-mechanical forces occurring during SSTC result in significant changes in the dislocation structures formed during rapid solidification. Both hypotheses are based on studies performed on as-built samples; yet, it is impossible to univocally separate the role of SSTC from that of solidification on dislocation structure and internal stress formation and evolution during AM by solely analyzing as-built samples. A clear insight can only be gained by tracking their evolution during the AM process.

To that end, we have conducted an experiment that involves performing high resolution reciprocal space mapping (HRRSM), a synchrotron-based X-ray diffraction technique, while additively manufacturing a thin wall from a single-phase alloy (in order to avoid effects of solid-state phase transformations) [1]. The experiments are performed using a miniature Laser Metal Deposition (mini-LMD) machine that has been specially designed for synchrotron X-ray diffraction and radiography experiments. In this talk, I will present the results of this experiment and answer the question posed in the title of this talk.

### 3. Bichromatic melt pool thermal measurement based on a RGB camera: application to additive manufacturing processes

Loïc Jegou (a,b), Joel Lachambre (a), Nicolas Tardif (a), Mady Guillemot (a), Anthony Dellarre (c), Abderrahime Zaoui (a), Thomas Elguedj (a), Valerie Kaftandjian (b) and Nicolas Beraud (c)

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Measuring temperature fields during additive manufacturing processes typically requires expensive equipment such as infrared cameras. The full-field sensor presented in this talk is very compact and consists of a single RGB camera. Based on a dual-wavelength radiometric model, it is designed to measure temperatures ranging from 700 to 2200 °C. Thanks to dynamic exposure time approach, it is possible to observe phenomena with high thermal gradients. The accuracy of the measurements is ensured by calibration on a black body and a tungsten ribbon lamp. The method is applied to wire arc additive manufacturing and laser metal deposition with powder, and an accurate measurement of thermal gradients in the melt pool and the surrounding solid phase is obtained. The good resolution of the camera gives a precise overview of the shape and size of the melt.

## Poster session

### 1. Interactions hydrogène-microstructure des alliages 625 et 718 fabriqués par fabrication additive

Dylan Cozlin<sup>1</sup>, Francisco Medina<sup>2</sup>, Kurtis Watanabe<sup>2</sup>, Abdelali Oudriss<sup>1</sup>, Xavier Feugas<sup>1</sup>, Jamaa Bouhattate<sup>1</sup>

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Du fait de sa petite taille et sa grande mobilité, l'atome d'hydrogène est très préjudiciable à la durabilité des matériaux métalliques. La fragilisation par l'hydrogène (FPH) est un mécanisme d'endommagement par lequel le matériau, exposé à un environnement concentré en hydrogène, voit sa ductilité diminuée, le rendant plus fragile [1]. La microstructure du matériau joue un rôle crucial dans le processus de fragilisation par l'hydrogène. Dans le cadre de ce travail, nous nous concentrons sur les effets des différents types de joints de grains et leurs distributions spatiales, ainsi que leurs connectivités afin d'améliorer notre compréhension de l'impact de l'hydrogène sur la rupture intergranulaire [2, 3].

Dans ce contexte, il est essentiel de rassembler des informations sur le comportement des joints de grains vis-à-vis de la mobilité de l'hydrogène. Les données expérimentales récoltées sont ensuite utilisées dans un modèle numérique de type éléments finis afin de simuler les différentes distributions et connectivités de chaque type de joint de grains. Les simulations numériques nous donneront ainsi accès à une microstructure moins sensible à la fragilisation par l'hydrogène basée sur l'impact du réseau de joints de grains sur la mobilité de l'hydrogène [4]. Pour obtenir une telle microstructure, les nouveaux processus de fabrication apparaissent comme les plus adaptés. En effet, la fabrication additive (FA) permet le contrôle des paramètres de fabrication afin d'obtenir des architectures originales. Sur ce thème, ce travail se focalise sur deux alliages base nickel, l'Inconel 625 et l'inconel 718 dont nous étudierons l'impact du réseau de joints de grains sur la mobilité de l'hydrogène et la fragilisation par l'hydrogène.

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[2] B. Osman Hoch, A. Metsue, J. Bouhattate, et X. Feugas, « Effects of grain-boundary networks on the macroscopic diffusivity of hydrogen in polycrystalline materials », Computational Materials Science, vol. 97, p. 276-284, févr. 2015.

[3] A. Oudriss et al., « Grain size and grain-boundary effects on diffusion and trapping of hydrogen in pure nickel », Acta Materialia, vol. 60, no 19, p. 6814-6828, nov. 2012.

[4] J. Sayet, B. O. Hoch, A. Oudriss, J. Bouhattate, et X. Feugas, « Multi-scale approach to hydrogen diffusion in FCC polycrystalline structure with binary classification of grain boundaries in continuum model », Materials Today Communications, vol. 34, p. 105021.



## **2. Influence of processing parameters on the fracture behavior of 316L austenitic steel processed by Laser Power Bed Fusion**

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The relationship between the process parameters and fracture behaviors of 316L SS printed by Laser Power Bed Fusion is explored. Five different test sets were considered in a vertical building configuration: standard parameters,  $\pm 20\%$  power, and  $\pm 20\%$  scanning velocity. The mechanical properties were determined by ultrasonic surface wave analysis. Fracture testing followed ASTM E1820 for Single Edge Notch Bending. The influence of local plastic deformation, quantified by a microscopic measurement of a surface profilometry, significantly depends on the process parameters. A correlation between energy density, fracture toughness and the dimensions of the Fracture Process Zone is observed. The critical fracture toughness for the AM configurations was calculated and discussed along with the plastic deformations at the crack tip. The R-curves show different fracture behaviors depending on the energy density. The energy required to grow a crack is associated with larger plastic zones resulting in fracture toughness values ranging from 43 (P-20%) to 427 kJ/m<sup>2</sup> (P+20%). From the obtained results, one may conclude that the fracture toughness of 316L SS printed by LBPF can be tailored/optimized by adjusting the energy density.

## **3. Influence du procédé de fabrication sur le comportement à rupture d'un alliage nickel-chrome élaboré par méthode conventionnelle et fabrication additive (L-PBF)**

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Le comportement à rupture des alliages base-nickel élaborés de manière conventionnelle est depuis longtemps étudié dans le cadre du dimensionnement des structures en service. Le but de cette démarche est de prévenir toute rupture prématurée, notamment dans les domaines aéronautique, nucléaire et maritime. Cependant l'essor de la fabrication additive, qui s'inscrit durablement comme un procédé d'avenir, soulève de nombreuses problématiques en termes de mécanique de la rupture. En effet, le vaste choix de paramètres d'élaboration ainsi que l'histoire thermique subie par une pièce lors de sa fabrication induisent des microstructures complexes

en raison de forts gradients thermiques, lesquelles vont influencer les mécanismes de propagation de fissure.

Dans le cadre de ces travaux, l'attention est portée sur le comportement à rupture d'un alliage nickel-chrome élaboré par fusion laser sur lit de poudre (L-PBF). Deux paramètres de fabrication ayant une influence de premier ordre sur la microstructure sont investigués : la direction de fabrication (horizontale et verticale) et l'angle entre les couches de lasage (67° et 90°). Le matériau coulé est étudié pour constituer une base de données de référence représentative des méthodes de fabrication conventionnelles. Des essais de flexion trois points sont menés classiquement en accord avec la norme ASTM E1820, portée sur les matériaux métalliques ductiles. Une comparaison de la ténacité, c'est-à-dire la résistance à la propagation de la fissure, est alors réalisée entre les différentes configurations de fabrication. Des observations microstructurales sont effectuées à différentes échelles afin de retracer le chemin de la fissure et comprendre les mécanismes physiques à l'origine des comportements obtenus.

#### **4. Development of Ni-20wt.%Cr metamaterials by additive manufacturing: towards the optimization of mechanical and acoustic properties**

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The research on metamaterials has made significant progresses thanks to hierarchical and multi-porous architectures. These architectures opened up new perspectives in terms of the mechanical properties (materials with negative Poisson's ratio) and the control of acoustic wave propagation (negative refraction, perfect absorption). The aim of this study is to build Ni-20wt.%Cr architectural structures by laser powder bed fusion (L-PBF) additive manufacturing in order to obtain innovative mechanical and acoustic properties.

The first axis of this study is devoted to build architectural metal diffusers for acoustic absorption by metal L-PBF. The diffusers are spheres with cylindrical pores parallel to the build direction.

The second axis focuses on the study of the acoustic and mechanical properties of the metamaterial. The acoustic properties of the diffuser are determined by diffusion and absorption measurements. Regarding the macroscopic mechanical properties, the mechanical strength of the structure is assessed using compression tests.

A third axis is devoted to the elementary deformation mechanisms of the thin metallic parts of the diffuser, in relation to the manufacturing conditions and the resulting microstructure. Thin walls (range thickness from 0.2 mm to 2 mm) are produced by L-PBF for various processing parameters. The mechanical behavior of thin walls is studied by tensile tests. The main mechanical properties of the alloy are next correlated to microstructure (grain size and dendritic cells, crystallographic texture, inter-dendritic precipitation, residual stresses) which can evolve

with the build thickness. The aim of this work is to observe if a size effect exists concerning the mechanical properties of thin parts, and its potential consequences on the mechanical integrity of the metamaterial.

## **5. Evolution of the microstructure and the hardness of additive manufacturing pure nickel samples processed by High Pressure Torsion**

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Additive Manufacturing (AM) alloys often exhibit complex microstructures with a hierarchy from nanometric to micrometric scale, such as solidification cells with element segregation and nanoscale precipitation along their grain boundaries, strong texture and mesostructures that reflect printing strategies. These characteristics lead to an excellent strength-ductility combination.

The possibility of further adapting these complex microstructures to improve mechanical performance by severe plastic deformation (SPD), and resulting grain refinement, is considered in this work. It has been reported that High Pressure Torsion (HPT) process has led to a remarkable increase in the hardness of SLM (Selective Laser Melting) austenitic stainless steels, and cobalt and aluminum alloys.

A typical HPT microstructure consists of elongated grains, usually well below 1  $\mu\text{m}$  in grain size, and exhibit typical shear texture components. Applied to samples produced by AM, HPT leads to distortion, elongation and refinement of the melting pools, with increasing number of tool rotations. A significant reduction of the porosity and a chemical homogenization of the material are also observed.

The evolution of the microstructure and the mechanical properties of pure Nickel AM samples treated by HPT is studied, as a function of the building strategy and the location in the produced bars. Grain size, grain boundary misorientation and texture have been investigated by EBSD, and micro- and nano- hardness have been measured in order to assess the mechanical properties. Grain fragmentation process, with the increase in high misorientation grain boundary density, has been observed to develop non-homogeneously in the sample, starting at sample periphery where the applied deformation is stronger. A homogeneous distribution of nanoscaled grains, with grain size around 250 nm, is obtained after 10 revolutions, leading to a strong increase in the Ni hardness.

## **6. Comparative study of pulsed and continuous wave laser powder bed fusion of AlSi10Mg alloy**

Pierre Hébrard<sup>1</sup>, Patrice Peyre<sup>1</sup>, Bassem Barkia<sup>1</sup>, Pierre Lapouge<sup>1</sup>, Frédéric Coste<sup>1</sup>, Émilie Le Guen<sup>2</sup>

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Le contrôle de la forme temporelle du faisceau laser dans le procédé PBF-L/M est un sujet peu exploré à l'heure actuelle. Les quelques recherches sur ce sujet sur Inconel718 et AISI316L semblent montrer l'intérêt d'un régime de faisceau laser pulsé pour réaliser des pièces. La précision géométrique des conceptions et la texture de la microstructure des matériaux se démarquent de celles obtenues en régime de faisceau laser continu généralement utilisé.

Nous cherchons à comparer les résultats issus du régime continu à ceux du régime pulsé par la fabrication de cubes de quelques millimètres de hauteur sur une machine prototype instrumentée développée au laboratoire. Le matériau d'étude est un alliage d'aluminium de fonderie adapté au procédé PBF-L : AlSi10Mg. Les alliages d'aluminium ont une faible absorptivité des rayonnements et demandent de fortes puissances laser pour être mis en forme, ce qui différencie l'étude des matériaux précédemment évoqués.

## **7. Tuning the mechanical and microstructural behavior of metastable-beta titanium alloys thanks to the in situ laser powder bed fusion manufacturing process.**

Hugo Schaal, Philippe Castany, Thierry Gloriant

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Titanium alloys are appropriate candidates for biomedical applications due to their high specific strength and high corrosion resistance. However, controversies over the cytotoxicity and the poor mechanical compatibility of the widely used Ti-6Al-4V (%wt) and Ti-6Al-4V-ELI call for the development of new metallic alloys for biomedical applications such as dental and orthopaedic implantations. Beta and metastable-beta titanium alloys are particularly fitted for this kind of applications due to their high biocompatibility, and their low Young's modulus.

Additive manufacturing technologies are very promising for the elaboration of medical devices because of the capability of these processes to elaborate patient-specific devices, as close as possible of the patient morphology. However, the lack of diversity of precursor materials is hindering the elaboration of "exotic" materials via these technologies.

In our work, we show how the in situ method, which promotes the utilisation of a blend of elemental powder instead of pre-alloyed powders, can be used in the laser powder bed fusion process to elaborate titanium alloys with low Young's modulus and interesting mechanical properties, with the possibility to tune the chemical compositions and the microstructures of these alloys via stress-induced martensitic transformation or heat treatments, changing

thoroughly the mechanical behaviour of these alloys. Some results about metastable-beta Ti-Zr-Nb-Sn titanium alloys will be presented in this communication.

## **8. In-situ micromechanical testing of alloys processed by Laser Power Bed Fusion**

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The specific microstructure of alloys manufactured by additive manufacturing leads to particular mechanical properties. Mechanical testing at the microstructure scale could help to understand the overall properties of these alloys.

## **9. Prévion de la durée de vie en fatigue des pièces issues de la fabrication additive WAAM : effet de l'état de surface**

Mathilde Renault<sup>1,2</sup>, Lorenzo Bercelli<sup>2</sup>, Sylvain Calloch<sup>2</sup>, Cédric Doudard<sup>2</sup>, Bruno Levieil<sup>2</sup>, Julien Beaudet<sup>1</sup>

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La fabrication additive par arc-fil est un processus de dépôt rapide de matériau, mais qui peut entraîner des hétérogénéités matérielles affectant la résistance à la fatigue des pièces produites telles que des porosités et une surface dégradée. Afin de remédier à ces problèmes, divers traitements de parachèvement peuvent être utilisés comme l'usinage, le martelage, le grenailage laser ou conventionnel, ainsi que des méthodes spécifiquement développées pour la fabrication additive.

Il est essentiel d'évaluer les propriétés de fatigue des pièces fabriquées par ce procédé afin de garantir leur fiabilité et d'établir des normes de qualité appropriées. Cependant, en raison de la complexité inhérente à la fabrication additive et des multiples options de traitement de surface disponibles, il est nécessaire de disposer de méthodes d'évaluation rapides. Une approche consiste à développer des modèles pour réduire le nombre d'essais requis, tout en nécessitant une identification du mode de rupture tant sur le plan qualitatif que quantitatif.

Bercelli et al. ont mis en évidence que l'état de surface constitue le facteur le plus critique pour les pièces brutes issues du procédé de dépôt, en raison des concentrations de contraintes induites en surface par cet état. Afin de détecter et de suivre les fissures, la thermo élasticimétrie (ou Thermoelastic Stress Analysis - TSA) se révèle particulièrement efficace, ne nécessitant pas une connaissance a priori du lieu d'amorçage. Cette technique a été appliquée lors d'essais de fatigue par flexion à quatre points sur des éprouvettes en CuAl9. En observant l'amorçage et la

propagation des fissures au cours de ces essais, il devient possible de développer un modèle probabiliste basé sur la mécanique linéaire de la rupture. Ce modèle permet ainsi d'estimer la durée de vie en fatigue des pièces produites par le procédé de fabrication additive.

## **10. Simulation de la radioscopie du bain fondu lors de la fabrication additive d'une pièce en acier inoxydable 316L par projection laser**

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LaMCoS / LVA

Présentation d'un modèle analytique pour simuler le bain fondu, puis simulation d'une radioscopie du bain à partir des résultats obtenus.

## **11. Development of heat-treatments for functionally graded superalloys obtained by LMD-p**

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In modern aircraft engines, thermomechanical solicitations can be extreme and quite different depending on the considered component and targeted application. Hence, materials used for each part are carefully chosen to withstand specific service loading conditions. Additive Manufacturing of Functionally Graded Materials can be used to combine components into a single one, but also to improve already existing parts. In this study, microstructural evolutions during heat-treatments and their impact on mechanical properties of a functionally graded structure between two different superalloys have been investigated. To do so, a  $\gamma/\gamma'$  Ni-based superalloy with high  $\gamma'$  fraction has been deposited on a commonly used C&W  $\gamma/\gamma'$  superalloy by Laser Metal Deposition of powder, generating a transition zone using a controlled mixing ratios of powders of the two alloys. Several heat treatments derived from "state-of-the-art" heat-treatment sequences of each alloy have been performed and characterized using SEM analyses, while mechanical properties have been assessed using mainly hardness tests. A closer look was also given to the chemical and microstructural homogeneity of the deposited part, and to the possibility of reducing it using heat-treatments without degrading the substrate. Finally, the main purpose of this work will be to propose a heat-treatment sequence enabling optimal mechanical properties for both the substrate and the deposited parts, at room temperature as well as at service temperatures.

## 12. Influence of a methane flame aggression on the mechanical behavior of an austenitic stainless steel produced by LPBF

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The objective of this work is to understand the influence of a methane/air flame aggression on the mechanical properties and the microstructure of an austenitic stainless steel (316L). The main interest is to get closer to the real conditions of temperature and chemical reaction between the material and the fuel inside a combustion chamber. This study is focused on four parameters: the methane/air equivalence ratio, the sample manufacturing process (cast or produced by SLM), the duration and the temperature of exposure. More precisely, for the latter parameter, we will seek to determine the influence of the thermal gradient induced by the flame exposure on the mechanical properties.

In a context where additive manufacturing (AM) is one of the main subjects of research in the aeronautical field, only researches concerning the influence of oxidation [1] or temperature [2][3] have been carried out. This study allows us to better understand the evolution of mechanical behavior, induced by a flame aggression[4], in a framework more representative of reality.

As expected, several effects on the mechanical behavior are observed. Indeed, the exposure to the flame causes a strong decrease in the yield strength and the ultimate tensile stress for the AM specimen (100 MPa), contrary to the cast specimen which present only a small decrease (20 MPa). After the flame exposure, the same tendencies can be seen for the hardness evolution, and the AM samples present the same hardness as the cast samples. This decrease in macroscopic properties is certainly due to exposure to hot combustion gases, which will cause the same evolution as annealing, and in addition will increase the O<sub>2</sub> and carbon content in the steel. In order to verify this modification, a chemical analysis (EDS) and grain size analysis (EBSD) will be carried out.

[1] S. H. Kim, G. Obulan Subramanian, C. Kim, C. Jang, et K. M. Park, « Surface modification of austenitic stainless steel for corrosion resistance in high temperature supercritical-carbon dioxide environment », Surface and Coatings Technology, vol. 349, p. 415-425, sept. 2018

[2] G. Marchese et al., « The role of texturing and microstructure evolution on the tensile behavior of heat-treated Inconel 625 produced via laser powder bed fusion », Materials Science and Engineering: A, vol. 769, p. 138500, janv. 2020

[3] M. L. M. Sistiaga, S. Nardone, C. Hautfenne, et J. V. Humbeeck, « Effect of Heat Treatment of 316L Stainless Steel Produced by Selective Laser Melting (SLM) »

[4] P. A. Król et M. Wachowski, « Effect of Fire Temperature and Exposure Time on High-Strength Steel Bolts Microstructure and Residual Mechanical Properties », Materials (Basel), vol. 14, no 11, p. 3116, juin 2021.

### **13. Effet de la direction de déposition sur la propagation de fissures dans un acier inoxydable Duplex fabriqué par DED**

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Des volumes denses d'acier inoxydable Duplex ont été fabriqués par DED (directed energy deposition). Des échantillons CT (Compact tension) ont été usinés à partir de ces volumes pour évaluer la ténacité du matériau dans deux orientations : soit parallèlement soit perpendiculairement aux couches déposées.

Les deux configurations ont fourni un comportement élastique isotrope, mais des valeurs de ténacité à rupture différentes. Afin de comprendre l'origine de cette anisotropie de rupture, des analyses post-mortem ont été effectuées. Une analyse métallographique classique révèle la structure hautement orientée du matériau, ainsi que la localisation des phases. L'étude des surfaces de rupture révèle plusieurs points. À l'échelle macroscopique, les surfaces sont relativement planes pour le cas parallèle, tandis que dans le cas perpendiculaire des lèvres de cisaillement prononcées sont obtenues. À l'échelle de la microstructure, la fracture est gouvernée par la coalescence de micro-cavités. La croissance de fissure est pilotée par l'échelle mésoscopique, qui est héritée de la stratégie de déposition employée lors de la fabrication. En effet, les frontières entre la région de solidification primaire des bains de fusion et les régions ré-affectées thermiquement sont des zones préférentielles pour la croissance de fissures. Ces frontières correspondent également aux limites entre les couches déposées. L'analyse de la rugosité des faciès de rupture permet de retrouver les longueurs caractéristiques de la mésostructure, confirmant son rôle majeur dans la fissuration. Ceci permet d'expliquer les différences observées dans les deux directions de fissuration : dans le cas parallèle, la fissure est alignée avec les plans de faiblesse constitués par les intercouches, canalisant la croissance des fissures. Dans le cas perpendiculaire, des excursions de la fissure hors du plan central deviennent possible, lui permettant de suivre une trajectoire tridimensionnelle tortueuse résultant en une ténacité plus élevée que dans le cas parallèle.



## **14. Les secrets de l'acier inoxydable super-duplex 2507 produit par procédé LP-DED**

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L'acier inoxydable super-duplex 2507 est un acier biphasé austéno-ferritique connu pour ses bonnes propriétés mécaniques, notamment en fatigue, et sa bonne tenue à la corrosion, conséquence de sa microstructure mixte ferritique et austénitique. La méthode d'élaboration par fabrication additive LP-DED (Laser Powder - Direct Energy Deposition) permet aujourd'hui de produire de tels aciers sous réserve de disposer des poudres métalliques ad hoc. L'utilisation d'un laser permet ainsi de fondre et d'agglomérer les poudres mais les cycles thermiques induits par le procédé génèrent une microstructure et des propriétés spécifiques, différentes de celles obtenues par procédé conventionnel comme le forgeage.

Les objectifs des travaux présentés sont tout d'abord de définir comment élaborer une matière la plus saine possible via une étude de fabricabilité, puis il s'agit d'observer sa microstructure brute de fabrication, et enfin d'évaluer les propriétés du nouveau couple « matériau-procédé » obtenu. Parmi les propriétés, l'aspect mécanique est bien représenté avec l'étude de la dureté et de la résistance à la traction, mais aussi avec une évaluation de la tenue à la fatigue à grand nombre de cycles à partir d'essais d'auto-échauffement. Les propriétés électrochimiques font également partie du panel expérimental avec des études de tenue à la corrosion par piqûration et à la corrosion sous contrainte.

L'ensemble des essais et des observations a été répété sur la même nuance d'acier élaboré par forgeage pour comparer nos résultats à ce procédé conventionnel pris comme référence. Différents traitements thermiques ont aussi été testés sur le matériau brut LP-DED afin d'optimiser les propriétés obtenues via ce procédé d'impression 3D avec des résultats qui varient selon les températures et les temps d'exposition.

## **15. Printability of Titanium Alloy TA6V4 by MELD Friction Stir Additive Manufacturing Process: Study of process-microstructure-macroscopic properties links and optimization of post-printing heat treatments**

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The research aims to explore the potential of Additive Stir Friction Deposition (AFSD) technology for Ti64 alloys, focusing on its impact on microstructure and mechanical properties.

AFSD has emerged as a promising additive manufacturing technique, offering defect-free and homogeneous microstructures with reduced thermal gradients and residual stresses. While AFSD has been extensively studied for Al-based alloys, research on Ti-based alloys remains limited.

Our study seeks to address this research gap by investigating the effects of process parameters on the microstructure and mechanical properties of Ti64 alloys produced using AFSD. Additionally, we will examine the influence of heat treatments on the micro and macro properties of the components. By understanding these effects, we aim to enhance the knowledge and applicability of AFSD for Ti64 alloys.

Furthermore, we will explore the fatigue behavior of the components produced by AFSD. This research will contribute to a comprehensive understanding of AFSD technology for Ti64 alloys and provide valuable insights for optimizing process parameters and heat treatments.

## **16. Étude comparatif des alliages fer-silicium fabriqués par FA - FLLP : analyse adimensionnelle des paramètres de fabrication**

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La Fabrication Additive par Fusion sélective Laser sur Lit de Poudre (FA - FLLP) est devenue une des technologies de FA la plus répandues dans l'industrie. Cette technique a montré la faisabilité de fabrication des composants en acier à haute teneur en silicium avec de bonnes propriétés magnétiques.

Afin de comparer les résultats des différentes publications, la notion de densité d'énergie normalisée a été utilisée. En utilisant cette analyse, un diagramme normalisé est établi ; il synthétise la relation entre une large gamme de paramètres laser et la densité des composants produits pour divers matériaux magnétiques. Le diagramme normalisé peut être utilisé comme un outil de prédétermination pour l'ajustement des paramètres procédé, tels que la puissance laser, la vitesse de balayage et l'espace entre cordons vis-à-vis de la densité. En perspective, l'utilisation de cette démarche d'analyse pourrait permettre l'optimisation du processus de FA - FLLP pour la fabrication des composants magnétiques.

## **17. Defect healing effect of different rescanning laser power on 316L LPBF generated with failing process parameters**

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Cette étude s'intéresse à la capacité corrective d'un relasage (la même couche est fusionnée plusieurs fois) dans le cadre de défauts pseudo-naturellement générées. En effet les défauts

naturels sont générés à l'aide d'une paramétrie volontairement trop peu énergétique pour permettre de fabriquer un matériau dense. Différentes puissances laser de relasage sont testés pour quantifier l'évolution des défauts.