Microstructure evolution during metal additive manufacturing

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Motivation and objective

Additive manufacturing (AM), also known as 3D-printing, holds the potential to revolutionize the metal and alloy manufacturing sector through its ability to simultaneously (a) generate the material from powder/wire form, and (b) manufacture the desired part geometry without the need of tooling. The process involves reading the 3D part geometry from a computer-aided design file and building it in a layer-by-layer manner by locally melting the material using a moving heat-source (laser or electron beam). Based on the material feeding approach, metal AM processes can be divided into: (a) direct energy deposition (DED) and (b) powder-bed fusion (PBF) methods. In DED, the feedstock material in powder/wire form is directly fed into the heat-source as it scans the build surface whereas in PBF, the powder feedstock is deposited onto a powder-bed between two successive layer scans. For both processes, the final as-built part contains surface and volumetric defects, residual stresses and a heterogeneous non-equilibrium microstructure. These phenomena depend on AM process parameters, material composition, and desired part geometry which together determine the material properties and part performance.

In this study, we are concerned with better understanding the evolution of the non-equilibrium microstructure during the AM process. Thus far, most experimental and modeling efforts in this field have been dedicated towards studying the heat-matter interactions in the melt-pool and rapid solidification [1–4]. While these are important topics to study, it is equally important to understand the microstructure evolution in the heat-affected solid zone which undergoes thermal cycling. To better understand this, consider the layer-by-layer building of a single-track (one heat-source scan per layer) wall and focus on the material at a point X on a deposition layer L (>1). Immediately after the localized deposition, the material at X cools down at a very high temperature rate (ranging from 10^3 to 10^6 °C/s). Addition of more layers (i.e. L+1, L+2, …) results in thermal cycling at X with varying temperature rates and amplitudes. The transient thermal gradients result in the formation of high-amplitude cyclic thermal stresses which must affect the microstructure at X, for example, through dislocation density evolution. At later stages of thermal cycling (i.e. during deposition of layer N >> L), a steady-state heat transfer could occur at X resulting in dynamic recovery/recrystallization. Furthermore, different material points in the as-built part will be subjected to different thermal histories resulting in heterogeneous microstructural evolution. ***The main objective of this study is to identify and quantify the microstructural evolution occurring due to thermal cycling in the heat-affected solid during AM.***

Project description

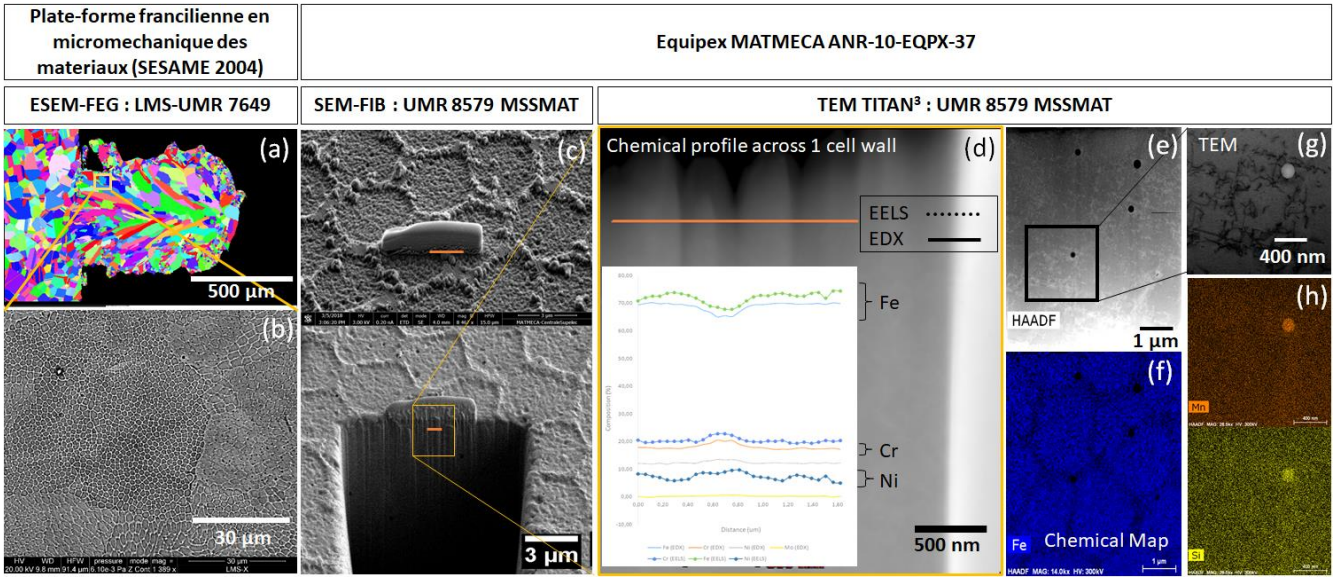

Figure 1*: (a) EBSD grain orientation map of our 3-layer 316L SS wall fabricated using LMD. (b) SEM map showing cellular structures in different grains. (c) Preparation of TEM lamellae in SEM-FIB, before (top) and after (bottom) extraction. (d) Chemical profile across one cell wall via EELS and EDX in TEM showing differences in composition in the cell wall and cell interior. (e) HAADF STEM map showing precipitates in TEM lamellae. (f) EDX map demonstrating lower Fe content in cell walls. (g) TEM map showing dislocations in cell walls. (h) EDX maps revealing higher Mn and Si content in precipitates.*

We will study the microstructural evolution of 316L stainless steel (SS) samples fabricated using the new Laser Metal Deposition (LMD: a DED process) machine at LMS. 316L SS is an important engineering material with diverse applications in household, medical and structural sectors. Conventionally formed 316L SS is ductile (30–40% strain) but suffers from low initial yield strength (~200 – 300 MPa). However, AM 316L SS typically exhibit superior strength without significant loss of ductility [5,6]. The origin of these desirable mechanical properties can be found in their non-equilibrium microstructure.

A preliminary microstructural characterization of our LMD 316L SS was recently performed via scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It revealed novel physical and chemical phenomena at multiple length scales which don't exist in conventional 316L SS. At the polycrystalline level there is a significant variety in grain orientation, morphology and size (hot-rolled 316L steel has equi-axed grains with a narrower size distribution). For instance, electron back-scattered diffraction (EBSD) map of a 3-layer wall in figure 1 reveals small equi-axed grains near surfaces and long columnar grains in the bulk. At sub-granular level chemical etching followed by SEM and TEM characterization revealed the presence of cellular solidification structures within each grain. The size and orientation of these cells depends on the non-equilibrium solidification kinetics which results in Cr and Mo segregation in the cell walls and the formation of Mn-Si-O precipitates, typically at triple junctions of these walls. Furthermore, the cell walls have relatively high dislocation densities ($\sim 10^{14} - 10^{16}$ /m^2) in comparison to cell interiors. Similar observations have been reported recently in 316L SS built using DED [5] and PBF [6], and other materials: 304L SS [7] and our LMD IN-625. The non-equilibrium phenomena are directly responsible for the enhanced mechanical response of AM materials. However, there have been no coherent efforts to study their evolution during thermal cycling; this is important because AM materials spend most of the processing time undergoing thermal cycling. Our aim is to fill this gap, and in the process answer the following questions:

(1) Do cell walls change their morphology during thermal cycling? If yes, does that occur during the initial or later stages?
(2) Does the elemental segregation in the cell walls change? Do precipitates grow during thermal cycling?
(3) What is the nature and amount of dislocations in the cells and their walls before and after thermal cycling?
(4) How is the polycrystalline texture and morphology affected by thermal cycling? Does dynamic recrystallization occur?
(5) What is the role of LMD process parameters (particularly laser power and speed) on the cellular structure evolution?

Benefits and Outlook

- From a metallurgical standpoint, we would gain a quantitative understanding of the microstructure evolution during thermal cycling. In addition, we would establish a novel experimental procedure that can be used to perform similar studies on other AM materials (other Fe-alloys, Ni-alloys, Ti-alloys, etc.). In the future, this knowledge will be used to suggest modifications to AM process parameters to design parts with improved performance.
- The knowledge gained will also be used to develop advanced crystal plasticity models coupled with recrystallization models to simulate microstructural evolution during AM. Furthermore, current crystal plasticity models assume that local equilibrium is maintained after each time increment, but this assumption may not hold during AM. This study will inform us if it's necessary to develop *extended non-equilibrium thermodynamics*-based crystal plasticity models [8,9] for AM.
- In addition, these results will guide in-situ bulk (as opposed to thin-film in TEM) deformation studies during synchrotron X-ray diffraction [10]; a test is already planned at DESY synchrotron (Germany) with Prof. W. Pantleon (DTU, Denmark).
- Finally, the results of this study will be presented, both in a scientific setting (at conferences) and non-scientific setting (for general public). We will create short (2-3 minute) audiovisual clips to explain the need to study the microstructure evolution during thermal cycling, the procedure used, the results obtained and their significance.

Resources

This project would require the coordinated efforts of the following researchers and equipment:

- AM 316L SS samples will be prepared using the LMD machine at the LMS laboratory by Manas V. Upadhyay and Sylvain Durbecq (AM Engineer, LMS). The main adjustable parameters of this machine are laser power, deposition speed, powder flow rate and layer height. The machine will be equipped with a pyrometer to measure temperature-time curves during AM. This data will be used to guide in-situ thermal cycling experiments in SEM and TEM.
- For in-situ and ex-situ microstructural characterization at the polycrystalline level we will use the QUANTA600 FEG ESEM of the F2M Microscopy platform (SESAME2004) at LMS. It is equipped with thermal and tension-compression devices. This equipment will be used to study the relationship between grain and cell orientations, cell size and process parameters, in-situ microstructure evolution during thermal cycling and mechanical testing. For this purpose, the expertise of Alexandre Tanguy (Engineer, MiMeCA, LMS) and Simon Hallais (Engineer, MiMeCA, LMS) will be solicited.
- We will also use the SEM-FIB Helios 660 nanolab at CentraleSupélec to extract TEM lamellae from the most interesting locations of the microstructure as shown in figure 1. This device, operated by Eva Héripré, will also be used to characterize the microstructure using transmission Kikuchi diffraction.
- In-situ thermal cycling and ex-situ chemical analysis will be performed using the probe corrected TEM Titan^3 G2 60-300 (STEM resolution under 70pm) at CentraleSupélec. This TEM is equipped with a Super-X EDX detector and an EEL Spectrometer for chemical analysis. A new in-situ electro-thermal holder (from Protochips Fusion) was recently developed for this TEM. It is capable of heating (and cooling) samples up to 1200ºC at temperature rates of 10^6 ºC/s. For operating this TEM, the expertise of Lluís Yedra Cardona (Research Engineer, CentraleSupélec) will be solicited.
- A post-doctoral candidate funded via the "coup de pouce F2M" scheme (50,000 euros) for a duration of 1 year.

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