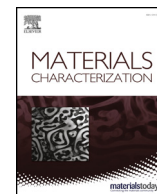




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## Editorial

## Highlights of the special issue on metal additive manufacturing

## A B S T R A C T

This editorial introduces a special issue on metal additive manufacturing (AM) discussing the main highlights and contributions to the field of each paper of the special issue classified by alloy type. These papers treat a range of additive manufacturing technologies.

## 1. Introduction

Metal AM is a rapidly expanding research field. Looking at scopus records in 2010, 96 papers treated that specific topic while in 2017, 860 papers were reported. Research on metallic alloys like aluminum and titanium are clearly taking the lead but new materials are slowly emerging. Yet even with these more classical materials, a lot remains to be studied to understand and improve the performance of manufactured components. This special issue on metal additive manufacturing has gathered the work of leading teams in the field worldwide.

Many different materials may be processed using AM technologies. In this special issue the following AM metals and alloys are discussed: aluminum alloys [1–7], titanium alloys [8–14], iron based alloys [15–17], Cu alloy [18], Inconel [19,20], NiTi [21] and TiAl [22] based composites, but also more complex types of engineered materials [23, 24].

The papers mainly discuss microstructural characterization and mechanical properties. However, a few papers also present results about magnetic [16], thermal [18] and electrical [24] properties as well as bio-compatibility [14] characterization of these new materials.

These materials could also be called structures. Indeed, one cannot dissociate material from structure as will be shown in several papers [2, 7, 10, 11, 15]. The material is built as the structure is built with a specific thermal history associated with the part geometry, in particular its wall thickness. AM can also be used to build nanoporous structures combining AM and selective etching [23] or as substrates to grow smart devices by inkjet printing [24].

Post AM processing [1, 9, 10, 16] is also an emerging field of research as more demanding industrial sectors, such as the aerospace industry, are showing increasing interest in qualifying these technologies. They require improved mechanical performance for these parts, in particular better fatigue performance [9]. Now post-processing can also help to tune magnetic properties of Fe-Si alloys [16]. Post-processing can even be done in-situ using the laser rescanning strategy [4].

Many additive manufacturing processes are treated in the present special issue: powder bed fusion technologies, i.e. laser beam melting (or selective laser melting, SLM, or direct metal laser sintering technology) and (selective) electron beam melting technology (S)EBM, the wire based technologies, i.e. arc [17], laser [7] or electron beam [5, 6]. Note that while the names given to identical technologies often varies from one paper to another, we have tried here to use a unified terminology, i.e. SLM, EBM and wire. As reported in the introductions of several papers of this special issue [5, 7, 17], wire-based technologies provide larger deposition rates ( $> 1\text{ kg/h}$  compared to  $0.2\text{ kg/h}$  in SLM [7]) with the possibility to build larger structures. Low equipment cost is also an advantage of the arc heating technology [17]. The drawback is of course the dimensional precision of the builds, which is much better with the powder bed technologies.

The effect of the process conditions on the microstructure and properties of the alloys is also an active field of research [3, 4, 5, 18, 22]. Concerning the SLM technology, the pulsed laser technology leads to finer cell size [3] and the laser rescanning refines the grains in the melt pool surface [4]. The special issue also features three papers comparing SLM and EBM technologies [8, 9, 11] for titanium alloys as well as other AM technologies [8]. The oxygen and nitrogen uptake of titanium is also an issue that is treated by Na et al. [12]. The feedstock powder preparation as pre-alloyed or elemental powder mixing and the mixing mode also influences part quality [13].

## 2. Aluminum Alloys

For additive manufacturing, Al–Si or Al–Si–Mg are the classical SLM alloys and three papers treat these materials [1–3]. Emerging alloys for SLM with enhanced performances involve Zr and Sc. In this special issue, the paper of Griffiths et al. [4] concerns Al–Mg–Zr, the so-called Addalloy™.

Zhou et al. [1] studied the microstructural modifications during classical solution heat treatment ( $520\text{ °C}$ ) and aging at  $160\text{ °C}$  on Al–Si10Mg alloy. They showed the segregation of Mg at the small ( $2\text{–}4\text{ }\mu\text{m}$

size) cell boundaries. Fine needle-like Si precipitates are observed inside the cell structure. The cell size is up to 5 times smaller than the grain size ( $\sim 10\ \mu\text{m}$  [2]). The Si content in  $\alpha(\text{Al})$  exceeds the Si solubility expected based on the phase diagram. As generally observed in these SLM alloys, coarsening and spheroidization of the eutectic structure is observed after the solution heat treatment. This coarsening is compensated by the Mg that is more homogeneously distributed in the matrix allowing for the precipitation of strengthening  $\text{Mg}_2\text{Si}$  precipitates after the aging treatment. In addition, the  $\pi$  Fe-rich phase is transformed to the homogeneous  $\beta\text{-Al}_5\text{SiFe}$  phase that does not tie-up any Mg in contrast to the  $\pi$ -phase.

Takata et al. [2] studied, for the same alloy and technology, the effect of the width of the sample (from 0.3 to 10 mm) on the grain structure, Si particle distribution and hardness. The area fraction of all grain orientations is little dependent on the sample width but the  $\{001\}$  orientation is clearly dominating. A slight decrease of low-angle grain boundaries is evidenced for smaller width samples. The most striking effect they report of the sample width is the larger proportion of very small Si particles finely distributed in the  $\alpha(\text{Al})$  (also reported by [1]) for the small sample width. The authors attribute this effect to longer time at high temperature for thin samples due to a slower cooling rate.

Chou et al. [3] studied a slightly different alloy ( $\text{AlSi7Mg}$  or A356) and proposes the use of a pulsed laser SLM technology with a 5 kW peak power, i.e. much higher than classical SLM machines. The most significant difference they report concerns the cell size wherein they found a mean size in the melt pool equal to 423 nm versus the  $2\ \mu\text{m}$  reported by Zhou et al. [1] for continuous laser technology. The fine microstructure allowed them [3] to estimate the solidification front velocity using the KGT model to be  $1.4 \times 10^{-1}\ \text{m/s}$  in the melt pool. In addition, even if epitaxial growth is also observed between melt pools in the pulsed technology, it did not extend through a large number of melt pools, i.e. the columnar grains are not as elongated. The authors attribute this difference to the faster solidification due to the short burst in energy during each individual laser pulse.

Griffiths et al. [4] studied the effect of rescanning each layer on the microstructure of Al-Mg-Zr alloy processed also by SLM. They found that the melt pool depth decreases in the rescanning step even if the laser parameters are unchanged. This is attributed to more heat dissipation on all sides of the already consolidated solid compared to the first scan where powder is observed on one side. The consequence is a nice refinement of the grains also on the top part of the melt pool while retaining the fine grains at the bottom of the melt pool. Indeed, Griffiths et al. [4] have shown that  $\text{Al}_3\text{Zr}$  precipitates are only present at the bottom of the melt pool. They also have evidenced that the refinement effect is more pronounced if the rescanning is performed perpendicular to the first scan.

Wire based additive manufacturing technologies are also emerging for aluminum alloys. In this special issue, three papers concern this topic [5–7].

Yu et al. [5] studied the Al-3Ti-Sc alloy processed by a bead-on-plate electron beam (EB) scan with various scan speeds. Similar to the alloy studied by Griffiths et al. [4] but with different precipitate compositions, this alloy offers the possibility of fine  $\text{Al}_3\text{Sc}$  and  $\text{Al}_3\text{Ti}$  precipitates. The as-cast material presented very coarse grained intermetallic phases showing little hardening potential. The EB processed zone is highly homogenous and hard, with hardness increasing with scan speed. The increased hardness with scan speed is attributed to the very fine  $\text{Al}_3(\text{Ti,Sc})$  precipitates and more Sc and Ti retained in solid solution within  $\alpha(\text{Al})$ .

Brice et al. [6] studied the Al-Cu-Mn-Mg-Ag alloy processed by EB additive manufacturing with a single track on the plate. They varied the Mg content from 0.2 to 0.8% and showed significant effects on the precipitation structure analyzed with transmission electron microscopy (TEM), small angle neutron scattering (SANS) and differential scanning calorimetry (DSC). The precipitate size and hardness increase with Mg content. This is attributed to the larger promotion at large Mg content

of the  $\Omega$  phase to replace the less hardening  $\theta'$  phase. They also evidenced the evaporation of Mg during the deposition, which should thus be accounted for to ensure sufficient Mg content for the formation of the desired hardening precipitates.

Froend et al. [7] studied the wire-based laser metal deposition of two 5xxx series aluminum alloys. The hardness was found to be lower on top of the small wall structure. This was attributed to longitudinal and unidirectional oriented grains in the center of the structure compared to randomly oriented and smaller grains on the top structure.

### 3. Titanium Alloys

Presently, Ti6Al4V is the most commonly researched alloy associated with AM. Two powder bed technologies are emerging in parallel for this metal class: the laser beam (SLM) and the electron beam (EBM) based technologies. However, other laser powder and wire based technologies are also available.

Neikter et al. [8] and Chastand et al. [9] compared the SLM and EBM technologies for Ti6Al4V. Neikter et al. [8] also compared other powder (also called laser cladding) and wire based technologies in a wide technological study. Generally, the powder bed fusion technologies (SLM and EBM) lead to finer microstructures. In the laser metal wire deposition (LMwD) technology, they found that a 2 min pause between tracks also favors a finer microstructure. In general, a significant effect of the cooling rate is reported on the  $\alpha$  phase size. Comparing SLM and EBM concludes for a finer intertwined microstructure and less elongated  $\beta$  grains with the SLM technology.

Chastand et al. [9] compared the fatigue life of SLM and EBM Ti6Al4V with various post-processing treatments (hot isostatic pressing – HIP, machining and polishing). They actually noticed a limited difference in the intrinsic fatigue life of the SLM and EBM parts. Machining and polishing significantly improved the fatigue life after removing the surface roughness. Machining also removes the near-surface defects related to the part contour strategy. After polishing, the fatigue life of powder bed fused samples reached a level similar to a cast alloy. After HIP and removing the surface roughness, the fatigue life is close to what is reported for wrought Ti6Al4V.

Persenot et al. [10] studied the tensile properties after post-processing of small struts representative of lattice structures manufactured by EBM Ti6Al4V. Such structures cannot be subjected to machining or mechanical polishing as Chastand et al. [9] applied. They thus studied chemical etching and HIP. Based on  $2.5\ \mu\text{m}$  resolution 3D X-ray tomography they observed no more porosities after HIP. However, this post-treatment leads to the coarsening of  $\alpha$  phase lamellae leading to a decrease in yield strength. The suppression of excessive porosities observed near the sub-surface by chemical etching leads to an improvement of the effective tensile properties.

Dzuzan et al. [11] studied the effect of the sample geometry on stress relieved SLM as well as EBM Ti6Al4V similar to work on Al alloys included in the special issue [2, 7]. In particular, they built thin walls and performed tensile tests. The sample size has shown significant effect on the ductility but the effect is actually quite unusual because thicker samples showed lower ductility. This was identified to be due to more volume process-induced defects in larger samples. Most probably, statistically large defects are more likely to be present in large samples and will be the source of premature failure. They found that the surface roughness plays a significant role on the load carrying regions of a sample. The surface roughness is significantly larger in EBM samples compared to SLM samples. The non-load carrying material was also mapped by analyzing the fracture surface of the tensile samples.

Na et al. [12] is the only paper in this special issue about titanium alloys that does not treat the Ti6Al4V alloy but rather pure titanium processed by SLM. They showed that an increasing laser power leads to significant uptake of oxygen and nitrogen in titanium even if the building chamber is filled with flowing argon gas. The oxygen and nitrogen uptake lead to significant hardening.

Simonelli et al. [13] discussed the important question of powder preparation and tried to answer the question: can we start with elemental powders to make alloys by SLM? They studied the Ti6Al4V alloy processed in a classical way, i.e. by pre-alloyed powder and compared this to a mix of pure powders of Ti + Al + V. They showed that the powder preparation is crucial to obtain a near-homogenous microstructure. The simple powder mixing (turbula type) did not generate a homogeneous microstructure and also leads to the highest volume porosity and coarse grains. They proposed a “satelliting” method (fine Al and V decorating larger Ti powder) that lead to a more homogenous microstructure but still some observable heterogeneities at the scale of the melt pool.

Luo et al. [14] performed an unusual study for metallurgists but highly relevant to the common bio applications of the Ti6Al4V processed by SLM. They showed that the cytocompatibility (antibacterial resistance) of the alloy can be improved by Cu addition. Fortuitously, the Cu release rate under body-like fluids was found to be far below the recommended daily Cu absorption amount.

#### 4. Iron Based Alloys

Leicht et al. [15] discussed the effect of thickness and build orientation on the resulting grain size and crystallographic texture of walls built of 316 L stainless steel with SLM technology. They identified a small (150  $\mu\text{m}$  thick) surface zone of fine and randomly oriented grains present in the wall samples thinner than 0.4 mm. The bulk of the walls is made of elongated grains following the build direction and not the part orientation when different. The vertical walls were significantly more textured (with a  $\langle 101 \rangle$  preferential orientation) than the inclined walls.

Garibaldi et al. [16] studied the microstructure, hardness and magnetic properties of a Fe-6.9%Si soft magnet ring manufactured by SLM. Powder bed fusion technologies are potentially interesting for Fe-based magnet production as these technologies lead to highly textured materials. In Fe-based alloys, a  $\langle 100 \rangle$  texture is expected to be an easier axis of magnetization. Garibaldi et al. [16] have shown that a stress relieving heat treatment performed after processing homogenizes the Si distribution. Indeed, Si tended to be micro-segregated in as-built samples. Homogenization, residual stress relief and a potential phase ordering are the expected reasons for the significant hardness and magnetic properties improvement compared to the as-built state. However, the small grain size typical of additive manufacturing technologies limits the magnetic performance of so-built rings.

Xu et al. [17] performed wire arc additive manufacturing of maraging steel and compared the microstructure and hardness of walls before and after an aging treatment. They observed a gradient in microstructure from the top to the bottom of the built wall. Less columnar grains, little austenite and less intermetallic compounds (Fe–Mo, Ni–Mo and Ni–Ti) were formed at the top of the build, leading to a softer material on top. More intermetallic compounds were formed after the aging treatment with a large proportion of discrete austenitic islands along the inter-dendritic boundaries. The hardness is increased and more interestingly homogeneous from top to bottom. The elongation at fracture of the wall samples is lower than that of wrought maraging steel, which presents a more equiaxed grain structure with fewer impurities.

#### 5. Other Alloys

In this section, highlights will be given for papers about Cu [18], Inconel [19,20], NiTi [21] and TiAl [22] based composites processed by powder bed fusion technologies (EBM and SLM). The last two papers [23,24] concern architected type materials where additive manufacturing is one step in the processing.

Guschlbauer et al. [18] performed EBM of pure copper, which is quite novel research as few literature papers report on EBM copper. The

paper is mainly focused on the identification of optimal process parameters with the originality of not using the pre-defined varying parameters of the Arcam machines (the so called “themes”) but actually controlling and maintaining the parameters constant to identify their influence on part soundness. In addition to density, microstructural observations, impurity distribution and mechanical property measurements, thermal conductivity is also measured as this is of particular importance for copper applications. The best process conditions lead to a very narrow window (1.5–2 m/s of scan speed and about 600 W of beam power) to reach high thermal conductivity as well as high overall density and low crack density. The crack density does not exceed the requirements for cast copper.

Polonsky et al. [19] performed an elegant 3D characterization of Inconel 718 EBM samples. The 3D characterization involves microscopy (EBSD and SEM) observations of serial sections performed by Focused Ion Beam (FIB). They observed large columnar grains oriented in the build direction with a strong  $\langle 001 \rangle$  texture. The columnar grain growth is interrupted by large lack-of-fusion defects leading to local equiaxed microstructures around defects. The TiN particle distribution is highly clustered near melt pool borders due to surface tension. The more homogeneous carbides show a diameter and number density in line with industrial alloys.

Fang et al. [20] also analyzed the texture and grain misorientations but in Inconel 625 SLM samples as-built and after a stress relieve heat treatment. They start their paper with a nice state of the art about Ni-based superalloys processed by SLM. They observed that elongated grains cross many building layers and this is maintained after heat treatment. The observed texture is different to what Polonsky et al. [19] observed and what is generally found in literature. The authors attribute this difference to the processing conditions. This suggests a perspective research topic. Fang et al. [20] identified a high density of Low Angle Grain Boundaries (LAGB) in the as-built samples associated by the authors to the high level of residual stresses in the samples. This large density is only reduced if the highest temperature heat treatment is applied.

C. Ma et al. [21] manufactured TiC reinforced NiTi based composites by SLM. Their study is focused on the variants formation of the  $\text{Ni}_4\text{Ti}_3$  precipitates that are known to influence the martensitic transformation. They found no difference in martensitic transformation temperatures compared to SLM NiTi without TiC reinforcements. The three identified variants present a selective growth based on difference in the size of the various precipitates. In addition, they performed a finite element study of the process to estimate the thermal residual stresses. They found these stresses are affected by the laser track strategy which is expected to affect the selective growth of the various variants.

Li et al. [22] manufactured reduced graphene oxide (RGO) reinforced TiAl based composites by SLM. They specifically studied the effect of the laser scan line spacing (or hatch space) on the microstructure and behavior under nanoindentation. Mostly high angle grain boundaries (HAGB  $\sim 80\%$ ) are present in the manufactured composite. Some RGO sheets transform to amorphous carbon during SLM. As the laser scan line spacing (or hatch space) increases, the grain size decreases slightly, the HAGB proportion decreases and the proportion of  $\alpha_2$  phase decreases. Consequently, the nanohardness and Young's Modulus also increase.

B. Ma et al. [23] used an original combination of additive manufacturing (SLM) and etching of a Fe65Cu17.5Ni17.5 alloy to build a sub-micron porous ordered honeycomb structure. The pore size is 11  $\mu\text{m}$  and the walls are 200 nm thick. During manufacturing and rapid solidification, a phase separation occurs between the Fe–Ni and Cu–Ni phases leading to a regular pattern of both phases. The Fe–Ni phase is etched off in the subsequent step. The paper also discusses the effect of the etching time on the resulting microstructure.

Faller et al. [24] performed a study on the applicability of substrates manufactured by a large variety of AM techniques. Such substrates will

be subsequently used to build smart devices by inkjet printing. They are built by a lost wax process which was itself built by 3D printing, followed by plaster enrobing and final casting for silver, bronze, brass and copper substrates, by SLM for titanium and aluminum or by lithography-based ceramic manufacturing (LMC) for alumina and zirconia. Characterizations include surface characterization with roughness and surface wettability using the water contact angle, but also electrical resistivity and interface observations after cross sectioning with a FIB. These characterizations reveal that the lost wax casting following 3D printing of the wax is the most suitable technology to build the substrate and have the right surface for inkjet printing.

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