Multiscale characterization of Al-4Fe alloy grown by additive manufactoring

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The advent of Additive Manufacturing (AM) processes in recent years has given us access to parts with complex geometries that are difficult to machine using conventional manufacturing methods. However, Additive Manufacturing leads to microstructures and metallurgical states that differ from those obtained by conventional processes. In this context, it has become necessary to develop new alloy grades dedicated to Additive Manufacturing in order to take full advantage of the specific features of these processes. In the aeronautics sector, there is a strong interest in the study of lightweight titanium- and aluminum-based alloy grades [1,2].

In this work, we are interested in a model Al-Fe alloy, which is not possible to produce using conventional processes due to the precipitation of the embrittling Al13Fe4 phase. The use of the L-PBF (Laser Powder Bed Fusion) additive manufacturing process avoids this pitfall, thanks to a very rapid cooling rate that favors precipitation of the metastable Al6Fe phase. It should also be noted that in the Al-Fe system, due to the very low solubility of Fe in cfc aluminum, it is not possible to carry out post-elaboration homogenization annealing. The aim is therefore to optimize the processing parameters in order to obtain an alloy with the desired mechanical properties at the end of the process. This type of alloy is particularly interesting in the context of repairing parts by AM, since annealing the part is often either not possible or not desirable in order to preserve the mechanical properties of the other parts of the part.

The L-PBF process is an additive manufacturing process involving the successive fusion of layers of powdered material using a laser. This process is characterized by very short interactions between the raw material (powder) and the laser, resulting in very rapid cooling rates (of the order of 106 K/s). The result is a complex microstructure organized on several size scales, which we have characterized in the Al4wt%Fe alloy and which is presented in the figure : At the hundred-micron scale, we observe the melt baths generated by laser melting; Inside the baths, we observe a structure of often columnar grains; Inside the grains, we observe a cellular structure, whose walls are enriched in Fe; Finally, inside the cells, we sometimes observe precipitates, as well as dislocations formed during solidification due to the presence of strong thermal gradients.

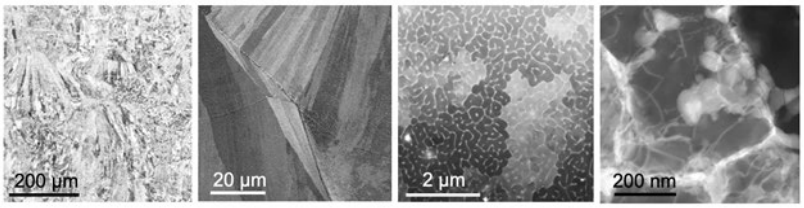
We are particularly interested in the hardening mechanisms operating in this microstructure. In addition to a Hall-Pech effect and hardening caused by iron-rich precipitates and cells and by dislocations, hardening due to the presence of iron solute is likely to be significant. Indeed, it is well known that the very rapid cooling rate during the L-PBF process leads to solute trapping in the matrix [3]. Furthermore, it has been shown that the presence of a low concentration of Fe in an aluminum matrix has a significant contribution to yield strength, far greater than that of usual solutes such as copper [4]. For our alloys, it is therefore necessary to measure the Fe composition inside the cells in order to quantify its impact on alloy hardening.
Another point of clarification is the composition of the cell edges, which are also enriched in Fe: this is expected to deviate from the composition of the metastable Al6Fe phase reported in less rapid solidification processes.

A final point that is not stabilized for our samples, as for similar alloys in the literature [5], is the presence of oxygen. Oxygen is present due to the inevitable oxidation of the powders, and may also originate from the gas present in the preparation chamber. However, its presence and location in Al-Fe alloys produced by L-PBF needs to be investigated. Indeed, the lattice parameters of L-PBF Al-Fe alloys, measured by X-ray diffraction, deviate from the values expected for these alloys, and the explanation put forward in the literature is the presence of oxygen, which would deform the aluminum lattice.

To achieve these goals, detailed characterisation by transmission and scanning electron microscopy (Figure), X-ray diffraction and atomic probe tomography has been carried out on samples produced using different parameters. The impact of manufacturing parameters on microstructure is highlighted, particularly the fact that the size of the solidification cells largely controls the mechanical behaviour. The results also reveals the correlation between the Fe distribution in the microstructure and the material's local hardness. Using transmission electron microscopy enables analysis of the interactions between nanometric precipitates and dislocations resulting from the thermal process, providing a better understanding of the hardening mechanisms. Atomic probe tomography allows to estimate the remaining Fe in the Al matrix in the as-built alloy and local enrichment in iron that lead to precipitation or segregation in the solidification cells.

The results allow the contribution of the different microstructural elements to hardening to be assessed using a phenomenological model. This model, developed based on mechanical tests conducted on various metallurgical states, can be used to propose chemical optimization paths. The purpose is to define an alloy which, in the as-built state, will benefit from a mechanical behaviour combining work and precipitation hardening.

Graphic:



Keywords:

Al alloys, additive manufacturing, solidification

Reference:

1. R. Liu, Z. Wang, T. Sparks, F. Liou, J. Newkirk, 13 - Aerospace applications of laser additive manufacturing, in Laser Additive Manufacturing, Milan Brandt editor, Woodhead Publishing, 2017, 351-371.
2. C. Pauzon, M. Buttard, A. Després, F. Charlot, M. Fivel, B. Chehab, J.J. Blandin, G. Martin, Direct ageing of LPBF Al-1Fe-1Zr for high conductivity and mechanical performance, Acta Materialia 258 (2023) 119199.
3. P. Galenko, S. Sobolev, Local nonequilibrium effect on undercooling in rapid solidification of alloys, Phys. Rev. E 55 (1) (1997) 343.
4. T. Uesugi, K. Higashi, First-principles studies on lattice constants and local lattice distortions in solid solution aluminum alloys, Computational Materials Science 67 (2013) 1–10.
5. X. Qi, N. Takata, A. Suzuki, M. Kobashi, M. Kato, Change in microstructural characteristics of laser powder bed fused Al–Fe binary alloy at elevated temperature, J. Mater. Sci. Technol. 97 (2022) 38–53.