



High temperature oxidation of IN 718 manufactured by laser beam melting and electron beam melting: Effect of surface topography



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ABSTRACT

The oxidation behaviour of IN 718 alloys produced by laser beam melting and electron beam melting was compared to that of the wrought alloy at 850 °C in laboratory air. Oxide scales of all alloys were similar in nature and morphology with small differences due to powder particles sintered on the surface of additive manufacturing parts. Nevertheless, major differences in surface topography were noticed, these could affect surface area estimations and consequently mass gain estimations. A quantitative correlation was determined between apparent parabolic rate constant and surface area.

1. Introduction

Recent progress in the field of additive manufacturing (AM) has led to the development of “3D printed” metallic alloys and ceramics parts. The microstructure of materials produced using these new manufacturing methods is different from those made using traditional methods. The aerospace industry and aircraft manufacturers seek to control AM processes in order to optimize the “buy-to-fly” ratio by lowering production costs and material loss during manufacturing while improving component functional design [1,2]. The Powder Bed Fusion (PBF) process is the AM method mainly used for metals. It consists in melting a powder bed and fusing it layer by layer, using either a laser beam – Laser Beam Melting (LBM) otherwise known as Selective Laser Melting (SLM) – or an electron beam – Electron Beam Melting (EBM). The microstructure of materials produced in this way is oriented following their build direction. As a result AM materials have anisotropic properties [3,4].

There have been recent attempts to produce parts of aircraft gas turbines by AM. But real gas turbine environment involves complex and aggressive mixed reactants that can lead to high temperature corrosion [5,6]. Before studying high temperature corrosion of AM parts, it is essential to characterize their high temperature oxidation behaviour in air since the first protection of the alloy from hot corrosion is the oxide scale formed onto its surface.

1.1. Microstructure of IN 718 alloys produced by additive manufacturing

The 718 alloy or IN 718¹ (NiCr19Fe19Nb5Mo3 – UNS N07718) is a solution annealed and precipitation hardened Ni-base superalloy [7]. This material is extensively used in aeronautical applications for its mechanical properties and structural stability at temperatures as high as 650 °C [8]. It was developed to resist creep, fatigue and, to a lesser extent high temperature oxidation [9]. This Ni-base superalloy is composed of austenitic matrix γ (Ni) phase, a disordered face-centered cubic crystal structure (A1, $a = 0.3616$ nm) [10], which gives to this alloy good mechanical strength. The solid solution strengthening of the matrix is induced by insertion of chromium and molybdenum. Composed of around 19 wt.% of chromium, the 718 alloy is a chromia scale former and therefore presents good oxidation resistance [11]. Furthermore, homogeneous precipitation hardening mechanisms are induced by the addition of alloy elements such as aluminium, titanium and niobium [12]. These elements lead to the precipitation of two metastable intermetallic strengthening phases, composed of a large amount of niobium. Their precipitation occurs between 600 °C and 900 °C. The phase mainly responsible for the alloy’s strengthening is γ' (Ni₃Nb) [10], which has a fine oblate spheroid form [13,14]. The second one, is the metastable γ'' (Ni₃(Al, Ti, Nb)) phase [10], which has a cuboidal or spherical form. After optimal heat treatment, the volume fraction of these two phases is 15–17 vol.% [15], the volume fraction of γ'' being three to four times larger than that of γ' [16]. After an ageing or a heat treatment, between 650 °C and 950 °C, δ -Ni₃Nb phases, the

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¹ IN 718 stands for INCONEL 718, designated alloy trademark of Special Metal Corporation.

stable state of γ'' (Ni_3Nb) phases, nucleate non-uniformly [17]. These δ -phases have a globular form when located at austenite grain boundaries or twin boundaries at temperatures below 930 °C; or an acicular form within the grain at temperatures above 1010 °C [18,19]. The precipitation of this phase is known to increase IN 718 ductility by affecting grain size [20]. As grain size decreases, the volume fraction of δ -phases increases, thus reaching a maximum value of 13% after being aged at 900 °C for 20 h [19]. During the ageing treatment between at temperatures 700 °C and 900 °C, co-precipitation of niobium rich MC carbides [10] and δ -phases occurs at grain boundaries and increases with ageing time, this favours intergranular crack propagation [21]. Last but not least, the Laves phase (Ni,Fe,Cr)₂ (Nb,Mo,Ti) [10], an hexagonal close-packed phase, is also present in the interdendritic regions of 718 welds due to niobium segregation during the solidification process with fast cooling rate [22]. This intermetallic phase, which contains large amounts of Nb (19–26 wt.%), is strongly affected by cooling rate and is known to be a preferred crack propagation site. This tends to reduce the material's tensile properties, ductility, and fatigue crack growth resistance [23].

Recent studies on microstructure showed small differences between AM-produced IN 718 and the wrought (WRG) alloy, partially due to the layer-by-layer melting process and the thermal gradient induced by beam melting. Thus, AM materials present columnar grains parallel to the build direction in LBM [24,25] and EBM processes both [26,27]. The thermal gradient induces epitaxial solidification leading to fine columnar grain architecture and dendrite epitaxial growth inside those grains, both structures are found parallel to the beam direction [28,29]. The formation of dendrites causes the segregation of alloy elements, such as niobium or titanium along interdendritic regions [30]. Besides, Helmer *et al.* [31] demonstrated that the grain structure of IN 718 alloys made by EBM process depends on scanning velocity and hatching space. They also showed that it is possible to have equiaxed grain structure even in the plane parallel to the build direction. Moreover, Strondl *et al.* [32] showed that the IN 718 alloy made by EBM process is a textured material oriented following the $\{200\}_\gamma$ plane. This was further confirmed by Idell *et al.* [33] in a 718Plus alloy made by LBM. Phases present in the AM material are similar to those present in the wrought material but differ depending on the thermal cycle and build parameters. Amato *et al.* [34] found an ellipsoid γ'' (Ni_3Nb) ($D = 100$ nm and $d = 25$ nm) phase platelet coincident with the $\{001\}_\gamma$ matrix in the as-built LBM material. This γ'' phase was also observed by Strondl *et al.* on the as-built EBM material [26]. They also found 5–10 nm long precipitates in the matrix and 50–100 nm long in the grain boundaries as compared to the 500 nm long precipitates found by Unocic *et al.* [35]. They also observed γ' precipitates (2–5 nm [26], 30–100 nm [35]) in the as-built EBM sample. Acicular δ (Ni_3Nb) phases (10 μm long) were found in the interdendritic region above the matrix due to niobium segregation in the as-built EBM sample. And precipitates that were found coarser at the upper side/part near the last solidified layer, than at the lower side/part [35]. Contrary to what was observed in the as-built EBM material, Kuo *et al.* [36] found that δ phases were present in LBM materials only after the AMS 5662 heat treatment. Besides, during LBM process solidification, high cooling rate results in Nb segregation [37,38], and therefore the formation of long brittle Laves phases. However, no Laves phase was reported in 718 alloys produced by EBM process [26].

Furthermore, the nature of the phases in the microstructure depends on manufacturing parameters such as beam power, beam velocity and building chamber temperature. It also depends on the cooling conditions and the heat treatment they undergo. Usually, the AM-produced IN 718 parts present high anisotropic grain orientation and porosities impacting the material's mechanical properties and density of the material [39]. Therefore, the as-built material needs to be hot isotactic pressed (HIP) followed either by an AMS 5662 or AMS 5664 heat treatment (HT), in order to close pores, dissolve weakening precipitate phases, homogenize the material and approach wrought microstructure

[28,30,35]. Rao *et al.* [40] found that HIP followed by AMS 5662 HT had a beneficial effect on the mechanical properties of AM-produced 718 alloys. This method results in significant grain growth, dissolves δ - Ni_3Nb precipitates and increases the material's ductility and yield strength.

1.2. High temperature oxidation of the IN 718 alloy

The IN 718 superalloy was designed for its mechanical strength at high temperature but also to form a protective chromia scale (Cr_2O_3) by selective oxidation of chromium (around 19 wt.%) when exposed to air at high temperature. The oxide layer growth is then controlled by slow diffusion of chromium cation through the oxide scale reaching the oxide/gas interface and/or by inward diffusion oxygen anions thereby reacting with chromium at the metal/oxide interface or with aluminium or titanium deeper in the alloy [6]. The formation of this chromia layer offers a good protection up to 950 °C under 10^5 Pa of air pressure. At higher temperatures, its growth rate becomes to be quite high and metal consumption is accelerated by the effect of sublimation of $\text{Cr}_2\text{O}_3(\text{s})$ into $\text{CrO}_3(\text{g})$ or Cr-hydroxides $\text{CrO}_2(\text{OH})_2(\text{g})$ [41]. Sublimation kinetics increases with temperature but also with the oxygen and water pressure present in the gas. In the case of turbine engines may be enhanced, thereby leading to early degradation. In aircraft applications, this material is commonly employed at operating temperatures below 650 °C [8]. During air oxidation between 700 °C and 950 °C [42,43], transient oxidation occurred during the first 24 h of the experiment, followed by slow oxidation kinetic during steady state. Between 900 °C and 1300 °C [42], oxidation kinetics have been shown to follow a parabolic rate dependence, ranging from $6.3 \times 10^{-6} \text{ mg}^2 \cdot \text{cm}^{-4} \cdot \text{s}^{-1}$ to $2.4 \times 10^{-3} \text{ mg}^2 \cdot \text{cm}^{-4} \cdot \text{s}^{-1}$. Above 1270 °C, the oxidation became catastrophic, the oxide layer can be spalled easily. At temperature above 1300 °C, the material was rapidly consumed [42]. Furthermore, looking at oxidation layer composition, small amounts of TiO_2 , MnCr_2O_4 spinel [43], and CrNbO_4 [44] were found during the early stages of the 900 °C air oxidation test. Those elements appeared before the establishment of a continuous chromia scale due to rapid diffusion of Ti, Mn, and Nb in the matrix. After the transient stage, a continuous intermetallic Ni_3Nb -rich scale was found under the chromia scale that might act as a chromium diffusion barrier slowing down the oxidation rate [43,44]. Moreover, Al_2O_3 intergranular oxidation was found in the 718 alloy [43].

In the literature, there are as yet few studies on the characterization of high temperature oxidation of the AM-produced IN 718 alloys, namely by Unocic *et al.* [35] and Jia and Gu [45]. Jia and Gu studied the oxidation behaviour of an as-built IN 718 alloy made by LBM, and oxidized at 850 °C for 100 h. They showed oxidation kinetics close to a parabolic growth. The oxide layer was composed of two different oxides: mainly Cr_2O_3 and small quantities of spinel NiCr_2O_4 and NiFe_2O_4 . Moreover, intergranular oxidation was found in the material. The penetration depth of the intergranular oxidation reached 50 μm and decreased with higher material density [45]. However, by comparing AM oxidation kinetics to that of the heat-treated wrought 718 alloy [42], results show that the oxidation kinetics of the as-built LBM 718 alloy studied by these authors is much higher, even for the densest samples. If this happens to be a general finding, the use of these materials. In the second work, performed at Oak Ridge National Laboratory [35], experiences were conducted in wet air at 650 °C, 700 °C and 750 °C on the EBM-produced 718 alloys, so as to compare oxidation rates based on several microstructures: wrought, EBM as-built, EBM HIP and EBM HIP + HT. Results showed that, at 650 °C and 700 °C, all samples had similar oxidation kinetics, and presented spallation. At 750 °C however, the EBM sample without HIP or HT, showed a mass gain without spallation while the wrought one had oxide scale spallation. This higher mass gain was explained by the formation of Fe-rich oxide nodules in the EBM sample, whereas the wrought sample showed mass loss with no Fe-rich nodules, similar to the tests performed at

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