



Fatigue crack growth rate and tensile strength of Re modified Inconel 718 produced by means of selective laser melting



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ABSTRACT

The paper presents results of mechanical tests, namely fatigue crack growth (FCG) rate and tensile tests – of Inconel 718 produced by Selective Laser Melting (SLM) in pure form and with Re addition. SLM method was used to manufacture “comb like” structures, simulating small parts with thin walls, of which final mini-samples were cut out (two types of samples for tensile and one for the FCG rate test were used). A fraction of samples underwent a standard procedure of a heat treatment designed for Inconel 718 alloy. The influence of samples orientation to the laser beam direction, samples size and heat treatment on the tensile strength, yield strength and elongation to fracture were investigated. FCG rate tests were carried out using mini-samples with notches. Cyclic loading of samples was synchronized with CDD camera trigger for registering images of samples surfaces at the moments of maximal loading. Digital Image Correlation (DIC) was used to determine near crack tip displacement fields. The results of DIC measurements were analyzed using the inverse method to automatically determine the stress intensity factor and crack tip coordinates. Additionally, fracture surfaces SEM observations, X-ray Diffraction (XRD) analyses, X-ray fluorescence (XRF), light microscopy (LM) and Transmission Electron Microscopy (TEM) observations have been done to understand mechanical properties variation revealed during mechanical testing.

1. Introduction

Rapid prototyping methods have gained growing interest in recent years. This is related to the possibility of manufacturing near net shape products of complex geometry, often with thin walls, by layer-by-layer powder melting. The usage of such methods may lead to material waste reduction, and therefore production cost savings [1]. Saving potential could be shown on buy to fly ratio often calculated in the aerospace industry. This means the ratio of the mass of material that is needed to machine a part compared to the mass of material in the finished component. For compressor and ring sections it is approximately 12:1 and the analysis performed by Pratt & Whitney indicated that the ratio could be reduced by 41% to 7:1 by using the cold spray method (considered as the method belonging to a rapid prototyping group, recently) for parts manufacturing [2].

There are reported many successful attempts of the utilization of the methods of rapid prototyping group to produce blocks of material made, for example, of steels, Ti, Cu, Co and Ni alloys [2–5] including

Inconel 718 [3,4] present in literature. However, it is often underlined that obtained materials may have inhomogeneous properties and more work should be devoted to the optimization of scanning strategy for each material. Moreover, reports concerning mechanical properties of thin wall parts are rarely available [5], as investigations usually focus on parts of regular shapes such as prisms or cylinders [2–4].

One of widely used methods of the rapid prototyping is Selective Laser Melting (SLM) in which consecutive powder layers are melted by a focused laser beam. Properties of final products manufactured in the SLM process strongly depend on powder type and morphology as well as on processing parameters, such as: scanning strategy, laser power, distance between two scanning lines, exposure time in a point and layer thickness. Another factor of the crucial influence on material properties is the object orientation to the laser beam direction (or building plane).

Relatively fast heating and cooling rates used in SLM process and directional character of final shape production may influence microstructure and mechanical properties of the final objects. Also, a slight porosity presence is expected - as usually noticed for powder metallurgy

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methods. This may have a detrimental influence on mechanical properties as well as cause larger flaws.

Current research is aimed to investigate mechanical properties of Inconel 718 alloy with rhenium addition (2%, 4% and 6% wt) produced by Selective SLM in the form of thin wall structures. Presented methodology, utilizing mini-samples and optical non-contact strain measurement method, may be applied to different materials of smaller size. This includes materials produced by means of rapid prototyping methods, especially when properties and fatigue crack growth (FCG) rates should be well determined. Only room temperature properties are investigated in this study – presented results may be treated as reference data for high temperature tests [6].

In the case of superalloys with Ni being the main component, strengthening is achieved by precipitates of γ' (face-centered cubic – $\text{Ni}_3(\text{TiAl})$) and γ'' (body-centered tetragonal – Ni_3Nb) phases which both are coherent with γ matrix [7–9]. The γ' phase is an example of superstructure when the appropriate constituent element ratio in solution is present. This superstructure has advantageous properties due to its strength growth accompanied with temperature increase, while still maintaining good plasticity, contrary to the strengthening by phases with high hardness. The γ'' phase occurs in the form of nanometer size disc-shaped precipitates, however longer exposure to temperature above 650 °C transforms it to incoherent δ phase which morphology and location should be carefully controlled. Generally, δ phase in the form of intragranular acicular precipitates limits ductility, however when δ phase of spherical morphology is present in grain boundaries prevents from grain growth at elevated temperature. Topologically Closed Packed (TCP) [10], Laves phases as well as carbides are detrimental in terms of material strength and durability. These phases are not coherent with matrix and their presence increases brittleness of superalloys.

Rhenium addition to superalloys improves their mechanical properties – especially at elevated temperature. Rhenium dissolved in γ phase is responsible for γ' coarsening retardation and the increase in misfit between γ and γ' [11]. These effects are present due to very low diffusion rate of Re in γ related to the large size of Re atoms [12].

The earliest information about Re additions to Ni-based superalloys can be found in the patent submitted by Smashey in 1975 [13]. The aim of his work was to improve high-temperature properties of unidirectionally solidified eutectic Ni-based superalloys by precipitating fibrous carbide phases aligned with the growth direction. In these superalloys Re does not tend to form carbides and can be used to strengthen matrix and promote the partitioning of Ta and V elements to the γ' phase. The first attempt to introduce Re to single-crystal superalloy was made by Giamei [14]. In this work, he tried to modify MAR-M200 alloy by replacing a fraction of W by Re, while keeping summarized the content of these elements constant. As a result, he obtained four different alloys with Re content equal to 0%, 2%, 4% and 6% of Re respectively. The specimens were assessed in terms of creep resistance, tested at 899 °C under the load of 380 MPa. Results analysis revealed beneficial effects of Re addition on creep deformation rate. The creep deformation rate strongly decreased with the increasing content of Re addition. This phenomenon is known as rhenium effect and it was confirmed in other works refereeing single crystal alloys [15,16].

Another effect observed in second-generation single crystal superalloys is coarsening kinetics reduction of γ' precipitates with rhenium addition [11]. This is due to the fact that Re prefers partitioning to the γ phase as observed in many single-crystal Ni-based superalloys with high γ' volume content [17,18]. While γ' coarsens, Re has to diffuse ahead of γ/γ' interface. The partitioning of Re to γ is due to the fact the Re is slowest diffusing metal in Ni matrix, and so γ' coarsening rate is severely limited by the presence of Re [19].

Development of third generation single crystal alloys proceeded with the increase in refractory elements content. Second generation superalloys contain 3% Re (F-16 and F-15 engines), while newer single crystal third generation alloys contain 6% of Re (F22 and F35 engines)

[20,21]. One of the first alloy named 5A showed a new microstructural instability [22] associated with the additions of higher levels of refractory elements, Re among others. The problem was not only related to the increase of a topologically close packed (TCP) in comparison to unmodified alloys, but also detrimental instability occurring under coatings. This was named secondary reaction zone (SRZ) because it occurred beneath the primary diffusion zone between the coating and the base metal. The discovery of SRZ raised new issues in the further development of third generation single crystal superalloys. It was found that Re was the element mainly responsible for the formation of SRZ. Thus, the levels of Re and other refractory elements had to be carefully balanced to obtain good microstructural stability and creep rupture strength [23].

Having in mind mentioned features of SLM method and supposed rhenium influence on the properties of superalloys it is important to know microstructure – properties relationship of thin walls structures of modified Inconel 718, and that is what this research aims to address. The need for accurate mechanical properties determination of thin wall structures leads to application of mini-samples technique accompanied with optical non-contact strain measurement method – Digital Image Correlation (DIC) described in Materials and methods section.

2. Materials and methods

“Comb-like” thin wall structures were produced of Inconel 718 and Re powders by means of MCP Realizer II – SLM 250 device. Powder mixtures for SLM processing were prepared in two stages. The first one was mechanical alloying with the 1:1 wt ratio of Inconel 718 and Re. In the second stage, the desired composition of 2%, 4%, 6% of Re was achieved by addition of appropriate amounts of pure Inconel 718 powder. Milling of powder mixture was carried out in a uni-ball mill with constant speed of 200 rpm and with 25 balls of 25 mm diameter. Rotation speed has been experimentally selected to keep the spherical shape of powder and reduce the influence of rhenium particles grind on the mixture flowability. Milling container was made of wear resistant zirconium oxide (ZrO_2) and had 500 ml capacity. Each milling resulted in 200 ml of final mixture production.

Average size of powders grain was determined by Laser Scattering Particle Size Distribution method using the Horiba LA-950 device. Initial and milled powders were examined by means of SEM (Hitachi 3500 and SU8000).

SLM process parameters optimization was realized in two trial stages. First one was aimed to minimize porosity and was carried out for pure Inconel 718 powder only (for economic reasons). In the second stage, optimization criteria considered both, porosity of final samples and Re dissolution level. Optimized parameters used for all Re addition levels are listed below:

- Scanning strategy: XZ (consecutive layer was scanned twice in perpendicular directions).
- Laser power: first scan 100 W, second scan: 400 W
- Scanning speed: first scan: 75 mm/s, second scan 500 mm/s.
- Layer thickness: 50 μm .
- Distance between lines: 160 μm

To qualitatively determine Re solubility ratio and select optimal SLM parameters, the analysis of binarized SEM/BSE sample cross-section images registered with three magnification levels has been used. Additionally, X-ray fluorescence (XRF) analyzes were made with the Bruker S4 Explorer device usage for determining Re content in finally produced samples.

Final samples for mechanical testing were cut out from the produced “comb-like” structures (see Fig. 1) by means of Electro Discharge Machining (EDM). Two geometries of mini-samples for tensile tests (with 5 and 10 mm gauge section length) and one type of samples with notches for Fatigue Crack Growth (FCG) rate tests (see

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