Materials Science & Engineering A 714 (2018) 75–83

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Stress relaxation in a nickel-base superalloy at elevated temperatures with in situ neutron diffraction characterization: Application to additive manufacturing☆

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ARTICLE INFO

Keywords:
Inconel 625
Stress relaxation
Neutron diffraction
Additive manufacturing

ABSTRACT

The complex thermal histories in additive manufacturing (AM) of metals result in the presence of residual stresses in the fabricated components. The amount of residual stress accumulated during AM depends on the high temperature constitutive behavior of the material. The rapid solidification and repeated thermal cycles with each laser pass result in material contraction, and subject the surrounding, constrained material to both elevated temperatures and internal stresses, providing driving forces for stress relaxation. In this study, the stress relaxation behavior and mechanisms of conventionally processed and additively manufactured Inconel 625 (CP-IN625 and AM-IN625) at 600 °C and 700 °C were investigated via compression tests up to an engineering strain of 9% with in situ neutron diffraction characterization. The stress decayed to a plateau stress equivalent to 18% of the peak stress in CP-IN625 and 16% in AM-IN625 at 600 °C, and 39% in CP-IN625 and 44% in AM-IN625 at 700 °C. At the same temperature, the stress relaxation rate in AM-IN625 was twice as high as that in CP-IN625, and the magnitude of the plateau stress in AM-IN625 was slightly lower than that in CP-IN625, as the textured AM-IN625 had much larger grains than the texture-free CP-IN625. The stress relaxation in CP- and AM-IN625 was deduced to be controlled by dislocation glide and climb, where dislocations interact with grain boundaries, solute atoms, and secondary phases. The stress relaxation constitutive behavior reported here is a necessary input for the development of accurate thermomechanical models used to predict and minimize residual stresses and distortion in AM, as well as to predict the stress relaxation behavior of Inconel 625 in high temperature structural applications.

1. Introduction

Nickel-base superalloys (e.g., Inconel 625 and Inconel 718) are widely used in aerospace, marine, and petrochemical applications due to their high mechanical strength, creep resistance, and corrosion resistance at elevated temperatures [1–3]. However, fabricating complex components from nickel-base superalloys using traditional subtractive machining can be expensive, as the hard nickel-base superalloys wear out machine tools easily. In addition, given the high cost of nickel-base superalloys, it is of interest to use additive manufacturing (AM) to fabricate near net-shape components with minimal waste of material [4–6]. In powder-based directed energy deposition (DED) AM with a laser heat source, a laser beam melts the substrate or previously deposited layer, and powder feedstock is delivered to the melt pool. As the laser advances to continue depositing the layer, the melt pool rapidly solidifies and fuses to the layer below [7–9].

During deposition, as subsequent layers are added, the surrounding material is subjected to thermal cycling with each additional laser pass. The material in the component being fabricated acts as a constraint, resulting in the generation of internal stresses during deposition [10–12]. The extent to which these stresses are relieved during fabrication depends on the stress magnitude and temperature as a function of time at each point within the component during fabrication, and the corresponding stress relaxation behavior [13]. Thermomechanical modeling can be used to predict residual stresses that develop during AM, as well as to optimize build parameters to minimize distortion in

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https://doi.org/10.1016/j.msea.2017.12.058
Received 11 July 2017; Received in revised form 26 October 2017; Accepted 13 December 2017
Available online 15 December 2017
0921-5093/

the fabricated components. However, for accurate modeling, the stress relaxation behavior, on the timescale over which the temperature during AM is elevated, is a required input. In addition, an accurate description of stress relaxation is necessary for predicting the behavior of components that will be loaded in service at elevated temperatures [14]. For example, compressive stresses are intentionally introduced into Inconel 718 blade fixtures to hold the blades tightly in turbine engines, but these stresses may relax during a component's lifetime at high service temperatures, leading to undesirable vibration or failure [15].

Prior research has been performed to investigate the microstructure and creep/stress relaxation behavior in conventionally processed nickel-base superalloys at elevated temperatures [16–21]. The creep resistance in nickel-base superalloy Inconel 625 is due to solid solution strengthening by niobium and molybdenum solute atoms in the nickel-chromium γ matrix. Additively manufactured Inconel 625 contains small amounts of niobium-, titanium-, and molybdenum-rich carbides, nitrides, and Laves phase in interdendric regions and along boundaries of columnar γ grains [4,5,22], while conventionally processed Inconel 625 contains small amounts of niobium-, titanium-, and molybdenum-rich carbides primarily along boundaries of equiaxed γ grains [23,24].

Diehl and Messler [16] investigated the stress relaxation behavior of annealed Inconel 625 welds under tension, with applied stresses approximately equal to the temperature-dependent yield stress, between 566–982 °C. They found that the stress relaxed exponentially, with 13% of the initially applied stress relieved at 566 °C, 87% at 871 °C, and 90% at 982 °C all after 8 h.

Rodriguez et al. [17] studied creep in ultrafine-grained Inconel 625 from 538 °C to 650 °C over a period of 36 h. Through investigation of the microstructure of deformed samples after testing using scanning and transmission electron microscopy, they concluded that creep was controlled by dislocation glide and climb, as dislocation tangles were found within grains. Mathew et al. [18] evaluated creep behavior and rupture life of conventionally processed Inconel 625 from 760 °C to 815 °C. These data indicated that dislocation creep was active under these conditions. Evans et al. [19] studied microstructural changes in conventionally processed Inconel 625 of varying thickness subjected to creep tests at 750 °C for 1310 h. They showed that the creep resistance decreased with decreasing thickness due to fewer grain boundaries in the thinner material. Scanning and transmission electron microscopy analyses of tested samples showed secondary phases, including carbides, δ phase, and σ phase, precipitated along grain boundaries and inhibited grain boundary sliding.

While stress relaxation has been studied in conventionally processed Inconel 625 [16–21], it has not yet been reported in additively manufactured Inconel 625. The microstructure of additively manufactured materials, and therefore, potentially their stress relaxation behavior, differs from their conventionally processed counterparts. In addition, the typical timescales studied for stress relaxation or creep in conventionally processed Inconel 625 are on the order of 10s–1000s of hours, whereas the timescale during which stress relaxation may be an active mechanism during AM processing is much shorter, as discussed in Section 3. The aim of this work was to quantify the stress relaxation behavior at 600 °C and 700 °C over a period up to 2.5 h, and to examine possible stress relaxation mechanisms in conventionally processed and additively manufactured Inconel 625 via in situ time-of-flight neutron diffraction. The stress relaxation behavior measured and reported here can be used in thermomechanical models of Inconel 625 to predict and mitigate residual stresses and distortion in AM as well as to predict the behavior of Inconel 625 in high temperature structural applications.

2. Materials and methods

A 102 mm long × 28 mm tall × 7 mm thick Inconel 625 wall was deposited by DED AM onto a conventionally processed Inconel 625 substrate (ASTM B443 Grade 1 [25,26]). Pre-alloyed Inconel 625 powder was deposited using a powder feed rate of 16 g/min, an argon flow rate of 9.4 L/min, a laser power of 2 kW, and a scanning speed of 10.6 mm/s [27]. Cylindrical specimens measuring 5 mm in diameter and 10 mm in length were extracted from AM-IN625 using wire electrical discharge machining (EDM) such that their loading axes in the subsequent compression tests were aligned with the laser scanning direction. Specimens with the same geometry were also extracted from the CP-IN625 substrate.

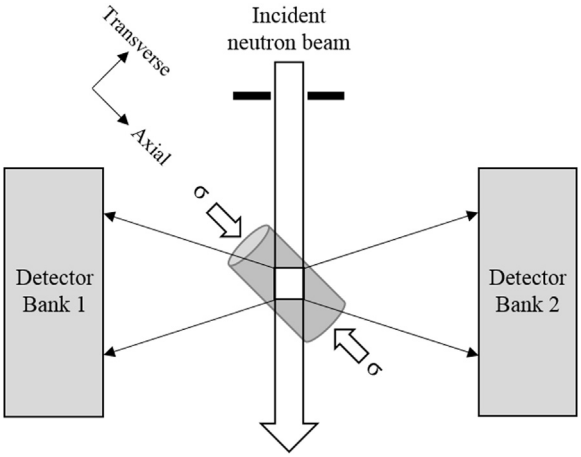

Fig. 1. Schematic of the experimental setup for compression with in situ neutron diffraction, in which diffraction patterns from the axial and transverse directions were recorded in bank 1 and bank 2, respectively.

Stress relaxation tests under compression were performed at 600 °C and 700 °C on the conventionally processed and additively manufacturing cylindrical specimens. These tests were performed with in situ time-of-flight neutron diffraction using the VULCAN instrument at the Spallation Neutron Source of Oak Ridge National Laboratory [28,29]. A schematic of the stress relaxation compression test with neutron diffraction is shown in Fig. 1. VULCAN has two detector banks that, being positioned at $-/+90°$ diffraction angles, simultaneously record diffraction patterns from grains with *hkl*-specific lattice planes whose plane normals are along the axial and transverse directions, respectively. The grain-orientation dependent, or *hkl*-specific, lattice strain, ε_{hkl}, was computed as $\varepsilon_{hkl} = \frac{d_{hkl} - d^0_{hkl}}{d^0_{hkl}}$, where d_{hkl} is the *hkl*-specific stressed lattice spacing, and d^0_{hkl} the corresponding stress-free lattice spacing. In addition to stress relaxation tests, uniaxial compression tests were performed at 600 °C and 700 °C to determine appropriate displacements to apply for stress relaxation tests.

In stress relaxation tests, the specimens were first heated to 600 °C or 700 °C and then compressed to an engineering strain of 0.09. The applied strain at each test temperature corresponded to an applied, or peak, stress of 75–85% of the ultimate tensile strength at that test temperature [30]. The macroscopic strain was held constant, and the evolving stress was recorded. After the stress reached a steady state plateau, defined by a decrease in stress magnitude less than 5 MPa in 5 min, the specimen was unloaded and cooled to room temperature. A type K thermocouple welded to the center of each specimen was used to measure the temperature throughout the test. The test parameters are given in Table 1, while Fig. 2 shows the applied strain, temperature, and resulting engineering stress, as a function of time during a stress relaxation test at 700 °C.

In order to evaluate the microstructure, untested cylindrical specimens extracted from CP- and AM-IN625 were polished using 0.05 μm colloidal silica and electrolytically etched using 10 wt% oxalic acid in DI water at 4 V for 20 s. The specimens were then examined using a scanning electron microscope (SEM, FEI Quanta 200).