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Diffraction and single-crystal elastic constants of Inconel 625 at room and elevated temperatures determined by neutron diffraction ☆

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ABSTRACT

In this work, diffraction and single-crystal elastic constants of Inconel 625 have been determined by means of *in situ* loading at room and elevated temperatures using time-of-flight neutron diffraction. Theoretical models proposed by Voigt, Reuss, and Kroner were used to determine single-crystal elastic constants from measured diffraction elastic constants, with the Kroner model having the best ability to capture experimental data. The magnitude of single-crystal elastic moduli, computed from single-crystal elastic constants, decreases and the single crystal anisotropy increases as temperature increases, indicating the importance of texture in affecting macroscopic stress at elevated temperatures. The experimental data reported here are of great importance in understanding additive manufacturing of metallic components as: diffraction elastic constants are required for computing residual stresses from residual lattice strains measured using neutron diffraction, which can be used to validate thermomechanical models of additive manufacturing, while single-crystal elastic constants can be used in crystal plasticity modeling, for example, to understand mechanical deformation behavior of additively manufactured components.

1. Introduction

Nickel-base superalloys, such as Inconel 625, Inconel 718, and Waspaloy, have excellent mechanical properties and corrosion resistance, especially at high temperature [1–4]. The production of nickel-base superalloy components involves mechanical and thermal processing, which may introduce strain gradients or thermal gradients, which could in turn result in the development of residual stresses. Mechanical processes that introduce residual stresses include traditional thermomechanical manufacturing like rolling and forging [5,6] and post processing methods like machining [7,8], grinding [7,9], and shot peening [10,11]. Thermal processes that introduce residual stresses include manufacturing processes that involve rapid solidification of materials such as welding [12–14] and additive manufacturing (AM) [15,16], as well as post processing heat treatment followed by quenching [7,17]. Mechanically generated residual stresses result from non-uniform plastic deformation, wherein compressive or tensile residual stresses are introduced on the surface of component by local plastic deformation [7]. Thermally generated residual stresses result from non-uniform heating and cooling, or thermal treatments with mechanical constraints [7]. For example, in welding and AM, residual stresses build up from the contraction of melt pool during cooling [12–16]. For materials being subjected to heat treatment followed by quenching, the outer surface cools more rapidly than the inner core, resulting in gradients in tensile and compressive residual stresses. Residual stresses may lead to distortion of a component, which results in a geometry that deviates from its design, and may introduce micro-cracks and local yielding within the component, impacting the component's mechanical performance [15,18]. Therefore, it is important to be able to accurately measure and predict residual stresses during manufacturing and post processing of materials in order to combat them, or account for them in design.

Here, we focus on nickel-base superalloys produced by AM. AM can be used to fabricate complicated near-net shape nickel-base superalloy components that cannot be fabricated through traditional casting or subtractive machining methods; thus researchers are investigating the possibility of fabricating solid solution

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strengthened Inconel 625 through AM [19–21] . Current obstacles in AM of Inconel 625 include the expense, due to the high initial cost of the powder feedstock and the fact that the number of times unused powder can be recycled is an open area of research [22–24]. Additionally, during AM, significant residual stresses are built up due to the complex thermal cycles – specifically, due to the material contraction as the melt pool solidifies and the additional contraction of the material during cooling [15,16]. In order to mitigate these stresses, thermomechanical modeling can be used to model the additive manufacturing process, predict the stress buildup and resulting distortion during fabrication, and modify the build process to minimize or counteract these stresses and distortion [16,18,25]. However, the prediction of residual stresses using thermomechanical models requires validation of these models. One way to measure residual stresses is using neutron diffraction, which requires grain orientation dependent, or *hkl*-specific, diffraction elastic constants (DECs) to convert the measured *hkl*-specific lattice strains to macroscopic residual stresses [12,15,26,27].

Diffraction elastic constants, such as Young's modulus, E_{hkl}, and Poisson's ratio, υ_{hkl}, describe the relationship between *hkl*-specific elastic lattice strains measured using neutron diffraction and applied macroscopic stress for a polycrystalline material. DECs can be determined by uniaxially loading the material in the elastic regime, and measuring each *hkl*-specific lattice strain responses to the applied macroscopic stresses in the loading and transverse directions simultaneously using neutron diffraction. For a highly anisotropic material like a nickel-base superalloy, at a given applied macroscopic stress, the stress and strain are distributed non-uniformly among differently oriented grains with respect to the loading direction. The *hkl*-specific oriented grains have their own intrinsic stiffness, single-crystal elastic moduli $E_{hkl,\ SC}$, defining the constitutive relationship between local stress and strain in a *hkl*-specific oriented grain or single crystal. The direction dependent single-crystal elastic constants comprise a 4th order stiffness tensor, c_{ijkl}, or compliance tensor, s_{ijkl}; these tensors can be expressed as 6×6 matrices, c_{ij} or s_{ij}, using Voigt notation. Therefore, materials with strong texture, such as additively manufactured Inconel 625, have a different macroscopic Young's modulus, meaning different macroscopic stress for a given applied elastic strain, compared to isotropic or weakly textured materials, resulting in different diffraction elastic constants between textured and isotropic materials [28]. A study conducted by Tayon et al. [29] also showed that the macroscopic Young's modulus depended on the orientation of highly textured Inconel 718 specimens made by AM. They used electron backscatter diffraction (EBSD) data and an Orientation Imaging Microscopy (OIM) software to estimate macroscopic Young's modulus in specimens with different orientations, in which single-crystal elastic constants were necessary inputs for the OIM software. As such, quantification of single-crystal elastic constants is essential for understanding anisotropic deformation behavior, especially at elevated temperatures where the anisotropy factor increases significantly. In addition, single-crystal elastic constants are used to provide grain-level constitutive information in crystal plasticity modeling, to calculate elastic energy for understanding dislocation interactions, and to determine the stability of phases to understand the occurrence of phase transformation [30–32].

Single-crystal elastic constants can be measured by different methods. Alers et al. and Kanrar et al. [33,34] determined c_{ij} in Fe-Ni alloys using acoustic methods, in which the speed of ultrasonic waves propagating through single crystals was measured. However, this method is limited by the purity and size of single crystals, the difficulty in fabricating single crystals, and the availability and accuracy of sensors used to detect ultrasonic waves at high temperatures or pressures [35]. Single-crystal elastic constants can also be determined using *in situ* diffraction methods during mechanical loading of polycrystalline materials at prescribed temperatures or pressures. Aba-Perea et al. [4] measured E_{hkl} and υ_{hkl} using neutron diffraction in Inconel 718 and plotted $1/E_{hkl}$ and $-\upsilon_{hkl}/E_{hkl}$ *versus* direction cosines of (*hkl*) to determine the linear interpolation coefficients for single-crystal elastic constants, rather than c_{ij}. No literature has reported diffraction elastic constants or single-crystal elastic constants of nickel-base alloys, in particular, Inconel 625, at elevated temperatures.

The present study focuses on measuring *hkl*-specific DECs and determining single-crystal elastic constants, c_{ij}, at room and high temperatures in Inconel 625 (IN625), a face-centered cubic (fcc) material that is used in additive manufacturing. Macroscopic stress and *hkl*-specific lattice strain measured by *in situ* neutron diffraction of IN625 upon elastic loading were used to determine the DECs, E_{hkl} and υ_{hkl}, which were then used to calculate c_{ij} and the single-crystal elastic moduli, $E_{hkl,\ SC}$ using a theoretical model proposed by Kroner [36]. The macroscopic elastic modulus, E_M, which describes macroscopic stress-strain relationships in isotropic materials, was measured from the experiments and also computed from c_{ij}. A comparison between the measured and computed E_M can be used to assess the accuracy of the selected model.

2. Materials and methods

We investigated conventionally rolled and annealed IN625 and IN625 deposited using directed energy deposition (DED). In powder-based, laser-based DED AM, powder is delivered through nozzles to a molten pool, on a substrate or layer below, produced by a laser beam [37–40]. A 101 mm long, 28 mm tall, 7 mm thick IN625 wall was deposited, using pre-alloyed IN625 powder, onto an annealed IN625 (AN IN625) substrate (ASTM B-443 Grade 1 [41]). A laser power of 2 kW, scanning speed of 10.6 mm/s, powder feed rate of 16 g/min, and argon gas flow rate of 9.4 L/min were used to deposit the IN625 wall by additive manufacturing (AM IN625) [16]. Energy dispersive spectroscopy (EDS) was used to determine the chemical composition of annealed and AM IN625, with the measured compositions given in Table 1. EDS analysis and scanning electron microscope (SEM) images indicated that AN IN625 consisted of a small amount (< 1 vol%) of carbides rich in Nb, Mo, and Ti distributed in an fcc γ matrix, and AM IN625 consisted of a small amount (< 2 vol%) of Nb- and Mo- rich carbides and Laves phase distributed in an fcc γ matrix [42]. These primary and secondary phases in AN and rapidly solidified IN625 are consistent with the literature [19–21,43,44].

To measure mechanical behavior of IN625 under compression, 5 mm diameter and 10 mm long cylindrical specimens were extracted from both IN625 made by AM and the annealed substrate. Compression tests at room temperature, 600 °C, and 700 °C at a strain rate of 3×10^{-5} were performed with *in situ* neutron diffraction using the VULCAN instrument at the Spallation Neutron Source at Oak Ridge National Laboratory [45,46]. Two detector banks in VULCAN allowed for collecting diffraction spectra simultaneously from grains whose lattice planes (*hkl*) were perpendicular to two orthogonal scattering vectors, which were along, and perpendicular to, the loading axis [47]. The *hkl*-

Table 1
Elemental composition in wt% of AN IN625 and AM IN625 measured by EDS.

	Ni	Cr	Mo	Fe	Nb	Co	Mn	Si	Ti	Al
AN IN625	59.2	22.3	9.5	4.7	3.5	0.4	0.3	0.3	0.1	0.2
AM IN625	60.2	23.5	8.7	4.6	2.3	0.1	0.4	0.4	0	0