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Unravelling thermal history during additive manufacturing of martensitic stainless steel

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Article info
Article history:
Received 29 June 2020
Received in revised form 9 October 2020
Accepted 11 October 2020
Available online 13 October 2020

Keywords:
Additive manufacturing
Thermal history
Phase stability
Residual stress
Neutron diffraction

ABSTRACT
In-situ thermal cycling neutron diffraction experiments were employed to unravel the effect of thermal history on the evolution of phase stability and internal stresses during the additive manufacturing (AM) process. While the fully-reversible martensite-austenite phase transformation was observed in the earlier thermal cycles where heating temperatures were higher than Af, the subsequent damped thermal cycles exhibited irreversible phase transformation forming reverted austenite. With increasing number of thermal cycles, the thermal stability of the retained austenite increased, which decreased the coefficient of thermal expansion. However, martensite revealed higher compressive residual stresses and lower dislocation density, indicating inhomogeneous distributions of the residual stresses and microstructures on the inside and on the surface of the AM component. The compressive residual stresses that acted on the martensite resulted preferentially from transformation strain and additionally from thermal misfit strain, and the decrease in the dislocation density might have been due to the strong recovery effect near the Ac1 temperature.

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1. Introduction
The recent COVID-19 pandemic has changed the supply chain for many industries. Additive manufacturing (AM) may offer alternative solutions [1] for the post-COVID-19 era. Specifically, there is an increasing demand for AM for metals in that there is a significant interest in industry for taking up AM as one of the main production engineering routes [2]. The scientific and technological approaches to improve the metal additive manufacturing have changed from being process oriented to microstructure oriented, for example, considering the effect of both the thermal gradient and the solidification rate, simultaneously, on the microstructure of the AM component [3–5].

Metal AM is a repetitive process that enables the metal powders to melt and solidify layer by layer. A melt pool on the top layer thermally influences already-deposited underneath layers during the continuous AM process. The repetitive heating and cooling cycles induce subsequent change in the microstructure of the AM component, which governs the material properties [6]. Therefore, a precise understanding of the thermal cycling effect during the AM process is of great importance to optimize the final microstructure and to remove harmful factors (e.g., defects, residual stresses) that influence the quality of the component.

First of all, a thermal history of the component being subjected to continuous heating and cooling needs to be defined to understand the thermal effect on the AM component. Bohlen et al. [7] experimentally revealed that a certain layer of the AM component undergoes damped thermal cycles by attaching thermocouples on it. The thermal cycle has been numerically examined by modeling a temperature distribution profile induced by a melting pool of post deposit layers [8–11], which was useful to optimize the heat source
parameter. Promoppatum et al. [12] investigated the thermal history of the AM component experimentally and numerically, and Rodgers et al. [13] simulated the microstructure imposed by the AM process. The investigation of internal stress development on the AM component is an important issue in the integrity of the material, as in the case of the welding process [14,15]. In general, the compressive residual stresses are developed inside and tensile residual stresses are developed on the surface of the AM component along the building direction [16]. Mukherjee et al. [17] reported that inhomogeneous residual stress distribution was developed from tensile on the surface to compressive on the inside, and its quantity depends on the location along the building direction. In spite of many worthy investigations on the AM component, the in-situ experimental study on the microstructure and residual stresses evolved during the AM process has been rarely attempted. The current study focuses on the evolution of phase constituents and internal stresses using in-situ thermal cycling experiments.

Real time measurements are necessary to observe the microstructural evolution induced by thermal cycling. With a dilatometer it is often hard to determine accurate transformation temperature due to the reduced difference in density between multiple phases at elevated temperatures [18]. X-ray diffraction and electron backscatter diffraction (EBSD) have been used to provide information caused by the damage and stress relaxation on the surface of the sample [19–21]. On the other hand, since neutrons make it possible to monitor internal stress and phase evolution in real time during thermal transformation in a bulk material [19,22], it is a powerful analytical technique for the study of the microstructural changes in the bulk AM component during thermal heating and cooling.

We specifically designed an experimental study to describe the effect of thermal history including a continuous thermal damped cycle using in-situ thermal cycling neutron diffraction experiments. Such an experimental approach made it possible to reproduce the thermal history which AM martensitic stainless steel experienced during AM processing. In this study, we examined the thermal stability and the developments of internal stress and dislocation density on phase constituents using neutron diffraction. This study provides meaningful data on microstructural evolution during the AM process and post heat treatment, although it is not perfectly identical to the actual thermal history of the AM component.

2. Materials and methods

2.1. Additive manufacturing conditions

A 15-5 PH martensitic stainless steel with superior strength and corrosion resistance was additively manufactured using a direct metal laser sintering. The metal powder for AM is composed of 14.8 wt%Cr, 4.5 wt%Ni, 3.2 wt%Cu, 0.07 wt%C, and Fe balance. The cylindrical dog-bone specimen was fabricated with the building direction parallel to the tensile axis with a spot size of ~70 μm, a layer thickness of ~40 μm, orthogonal scanning strategy, and ~1200 mm/s in scanning speed under nitrogen atmosphere.

2.2. Microstructure characterization

For the EBSD measurement, the specimen was mechanically polished with 1 μm diamond suspension, followed by electro-polished under 2 V for 30 s using 30% nital etching reagent. The EBSD measured a plane parallel to the building direction with a voltage of 20 kV and a step size of 0.4 μm, and grain boundary was determined by high angle grain boundary, θ > 15°, from an inverse pole figure and phase maps. Since EBSD and diffraction were insufficient to resolve body-centered cubic (BCC) and body-centered tetragonal (BCT) structures, both structures were considered as BCT for this study.

2.3. Neutron diffraction experiments

In-situ thermal cycling neutron experiments were performed under the stress-free condition in an air atmosphere using the mechanical testing system on the VULCAN engineering materials diffractometer at the Spallation Neutron Source of the Oak Ridge National Laboratory (Fig. 1d) [23,24]. An induction heating coil was used to control the heating and cooling with a thermocouple attached to the sample. The entire time-of-flight (TOF) diffraction patterns were acquired continuously during heating and cooling sequences from two detector banks situated on diffraction angles of $2\theta = \pm 90^\circ$. Hence, the axial detector bank collects the diffraacted grain families whose scattering vectors are parallel to the axis of dog-bone specimen, and the transverse bank collects the diffraction grain families whose scattering vectors are perpendicular to the specimen axis. The diffraction spectra were in a range from approximately 0.5 to 2.4 Å in the wavelength using a 30 Hz chopper setting, and each spectrum from $5 \times 5 \times 5$ mm$^3$ gauge volume size corresponded to 1 min of data collection. The interplanar spacings (d-spacings) from the axial and transverse banks were obtained as a function of temperature during the heating and cooling sequence. Rietveld refinement using General Structure Analysis System (GSAS-II) determined the average lattice parameter and phase fraction, and single peak fitting determined the d-spacing and full width at half maximum (FWHM) of hkl reflections [25].

Thermal cycling was designed to contain appropriate repetition of heating and cooling process with a temperature range available, and to ensure enough time to collect neutrons during in-situ neutron diffraction experiment. The specimen was heated up to 1000 °C from room temperature (RT) with ~17 °C/min and cooled down to RT with ~17 °C/min in the 1st cycle. In the 2nd cycle, the temperature increased up to 900 °C with ~18 °C/min and kept for 20 min, then decreased to RT with an average of 10 °C/min. In the 3rd, 4th and 5th cycles, it was heated up to 600 °C, 370 °C, 180 °C, respectively, and cooled down to RT with 13–22 °C/min.

3. Results and discussion

3.1. Phase stability

EBSD and neutron diffraction were utilized to examine the microstructure of the as-built sample. The average grain size determined with a high angle grain boundary above 15° in the inverse pole figure map was ~2.2 μm. Although very small amount of 0.3% austenite was found in the miniaturized phase map (Fig. 1a and b), no austenite was observed in the diffraction pattern of the as-built sample (Fig. 1c) whose microstructure was fully composed of martensite with a lattice parameter of 2.874 Å. More details on the sample and its texture can be found in earlier studies [26,27]. Fig. 2 is a conceptual diagram that represents the thermal history during the AM process. The temperature variations at the designated location (marked in X in Fig. 2a) were examined in Fig. 2b [17]. The thermal cycling test was conceptually and specifically designed using neutron diffraction (Fig. 2c).

Although the suggested cycling test was somewhat different from the real situation involving rapid heating and cooling during AM process, sufficiently-diffracted neutrons precisely traced the structural change of the alloy at the designed temperature, revealing the phase stability evolved during damped thermal cycles.

The weight fraction between martensite and austenite phases is presented as a function of temperature in Fig. 3a. Upon heating in the 1st cycle, a martensite phase observed at RT started to transform to the austenite phase at 533 °C ($A_s$), and finished at 928 °C...
Upon cooling, the martensitic transformation started at 175 °C (Ms) and completed at 74 °C (Mf). As, Af, Ms, and Mf in the 2nd cycle were 624 °C, 881 °C, 208 °C, and 101 °C, respectively. The difference in As and Af temperature between the first two cycles is attributed to the difference of austenite reversion generated below the Ac1 temperature. The as-built specimen was very heterogeneous than the specimen undergoing the 1st thermal cycle including a fully austenitizing process. Indeed, the as-built specimen has the smallest grain size [28] and higher dislocation density (described in section 3.3) than the heat-treated specimen. Thus, it has more nucleation sites for reverted austenite, resulting in the lower As temperature in the 1st cycle. The specimen, experiencing the 1st cycle, underwent fully austenite-martensite phase transformation, and showed a little bit lower As than the Ac1 of general PH steel [18], which was attributed to the reflection of austenite reversion below the Ac1 induced by a relatively low heating rate. The difference in Ms temperature might be due to the difference in the cooling rate and residual stresses developed as a function of...
number of cycle (described in section 3.2). Upon heating to 600 °C in the 3rd cycle, approximately 38% of martensite changed into reverted austenite below Ac1, and then during cooling, phase transformation of austenite to martensite resulted in the final retained austenite fraction of 13% at RT. In the 4th and 5th cycles, where the sample was heated at temperatures lower than Ac1, the phase fraction between the martensite and retained austenite remained constant.

The lattice parameter and the coefficient of thermal expansion (CTE) for each phase are presented in Fig. 3b and Table 1 [29]. Upon heating in the 1st cycle, the lattice parameter of the BCT increased linearly with a CTE of 12.297 °C⁻¹, after which the slope of the lattice parameter vs. temperature decreased slightly in the period of 393 °C–642 °C. This region was also found at a similar temperature range, as observed in the 2nd and 3rd cycles. This was due to the formation of the Cu-precipitates of Cu-bearing PH steel [18,30,31]. Above 642 °C, the lattice parameter of the BCT increased gradually (almost linear) with a higher CTE of 19.110 °C⁻¹. This was due to the combined effect of the transformation strain of FCC austenite (smaller volume than martensite) within the martensite matrix and thermal misfit strain [19,20,32]. During the repetitive heating and cooling up to the 5th cycle, the CTE of martensite increased slightly from 12.297 °C⁻¹ (1st) to 13.275 °C⁻¹ (5th) upon heating, and decreased from 14.486 °C⁻¹ (3rd) to 12.276 °C⁻¹ (5th) upon cooling (Table 1). On the other hand, the CTE of austenite in the 1st cycle was 25.557 °C⁻¹ during heating and cooling, but it was largely reduced to 19.156 °C⁻¹ during heating and to 16.144 °C⁻¹ during cooling in the 5th cycle (Table 1). The variation of CTE was significant according to the existence of retained austenite. It was attributed to the fact that the matrix with a lower CTE constrained the volume expansion of the austenite with a higher CTE during temperature variation.

3.2. Anisotropic residual stresses

Fig. 4a–e shows the relative changes in the interplanar spacing (d-spacing) and lattice parameter for both martensite and austenite at RT after heating and cooling of each thermal cycle. The change in d-spacing for the hkl reflections obtained from axial (parallel to the building direction) and transverse (perpendicular to the building
direction) indicates the development of internal stress for various orientations in polycrystals in terms of direction (axial and transverse). During continuous thermal cycling, nonhydrostatic compressive residual strains developed in the martensite, as revealed in the \( hkl \) strains with different levels in both axial and transverse directions (Fig. 4a and b). It indicated that the residual strains developed along the building direction (axial) and its transverse direction were anisotropic. After the 5th thermal cycling, the highest compressive residual strains were observed in the BCT \( \{211\} \) in the axial direction and the BCT\( \{200\} \) in the transverse direction.

The development of the residual strains in retained austenite, which is only available after the 3rd cycle, is presented in Fig. 4c–e. Internal stresses were developed in the retained austenite due to a martensitic transformation strain below 99 C in the 3rd cycle (Fig. 3), as well as a thermal misfit strain between two phases in the 4th and 5th cycles. The retained austenite showed anisotropic strain distribution among different orientations, and the stress state was nonhydrostatic, as exhibited in different strain levels in both directions.

It is obvious that the repeated heating and cooling developed residual stresses in each phase (Fig. 4). It is well established that interplanar spacings are influenced by thermal expansion, the concentration of interstitial atoms, and internal stresses [19,20,32,33]. Regardless of the existence of retained austenite, the lattice parameter of martensite decreased continuously with an increasing number of thermal cycles (Fig. 3). The compressive stresses acting on the martensite resulted preferentially from transformation strain and additionally from thermal misfit strain induced by the difference in the thermal expansion rate between the two phase constituents [20,32,34–37]. A slight tensile thermal strain acting on the retained austenite was due to the development of tensile stresses that balanced the compressive stresses acting on the martensite [19–21,33,34,38].

Based on the change in magnitude in the residual stresses with an increasing number of thermal cycles (shown in Fig. 4), we can understand that the inside of the AM component, which undergoes many thermal cycles, exhibits relatively higher compressive residual stresses than the surface of the AM component that undergoes fewer thermal cycles [16,18]. In addition, inhomogeneous residual stress distribution caused by damped thermal cycles may be correlated with the anisotropic evolution of residual strains in Fig. 4. The anisotropic residual stresses in terms of direction and location in the AM component reported by Mukherjee et al. [17] coincide well with the anisotropic \( d \)-spacing change of \( hkl \) reflections in this investigation.

### 3.3. Dislocation density

The dislocation densities of austenite and martensite are examined in Fig. 4f using the FWHM of the respective diffraction spectra which relate to lattice imperfection [39]. The detailed calculation procedure can be found in Ref. [35], and a brief explanation is described here with the following equation:

\[
\rho_{\text{cycle}} = \frac{3E}{\mu b_{\text{phase}}^2 \left( 1 + 2v_{\text{phase}}^2 \right)} \left( \frac{\text{FWHM}_{\text{cycle}} - \text{FWHM}_{\text{Ref Temp}}}{d_{hkl}^\text{cycle}} \right)^2
\]

where \( \rho_{\text{cycle}} \), \( E \), \( \mu \), \( b \), \( v \), and \( d \) are the dislocation density at RT after heating and cooling, the modulus of elasticity, the shear modulus,
Burgers vector, Poisson’s ratio, and d-spacing, respectively. The grain size was negligible due to less effective in Williamson-Hall equation [40], and \( E/\mu = 2.5, b_{\text{BCC}} = 2.49 \, \text{Å}, b_{\text{FCC}} = 2.54 \, \text{Å}, \rho_{\text{BCT}} = 0.296, \rho_{\text{FCC}} = 0.222, T_{\text{ref,BCT}} = 747 \, ^\circ\text{C}, \text{and } T_{\text{ref,FCC}} = 191 \, ^\circ\text{C} \), were used, respectively [26,35]. The selected reference temperatures were due to a strong recovery effect in the BCT and the absence of microstrain induced by martensitic transformation in the FCC. The dislocation densities calculated from each hkl reflection were arithmetically averaged.

The dislocation density of martensite was 1.53018 \times 10^{15} \text{ m}^{-2} in the as-built condition. In the first two cycles, the dislocation density decreased slightly, but it rapidly decreased after the 3rd cycle. The first thermal cycle would partially eliminate imperfections such as dislocations induced by the AM process, which was reflected on FWDM. The dislocation densities of retained austenite were 0.81433 \times 10^{15} \text{ m}^{-2} (3rd cycle) and 0.44174 \times 10^{15} \text{ m}^{-2} (5th cycle), respectively. After the 5th thermal cycle, the dislocation density of the retained austenite was two times higher than that of martensite.

The continuous thermal cycling influenced dislocation densities as well as residual stresses and CTEs of phase constituents. The reduction in the dislocation density in the 3rd cycle (Fig. 4f) should be due to the strong recovery effect near the \( A_\text{c}1 \) temperature. The target temperature of 600 \, ^\circ\text{C} in the 3rd cycle would be above the critical temperature for the activities of diffusion of vacancy and interstitial atoms, which is consistent with an earlier investigation arguing that dislocation in new-born martensite could be almost eliminated at 500–700 \, ^\circ\text{C} [35].

4. Conclusions

We investigated the effect of thermal history on phase stability and microstructural evolution during additive manufacturing of martensitic stainless steel using in-situ heating and cooling neutron diffraction. While a fully-reversible martensite-austenite phase transformation was observed in the first two cycles where the heating temperature was higher than \( A_\text{c}1 \), the 3rd thermal cycle exhibited irreversible phase transformation forming the reverted austenite. The thermal stability of the retained austenite increased with an increasing number of thermal cycles, resulting in reduction of the CTE. As the number of thermal cycle increased, the lateral parameters of martensite decreased continuously, revealing that higher compressive residual stresses had developed in the martensite. This well simulated the inhomogeneous distributions of residual stresses from the interior of the AM component, that has undergone more thermal cycles, to the surface that has undergone less thermal cycles. The compressive stresses acting on the martensite were mainly due to transformation strain and thermal misfit strain.

CRediT authorship contribution statement

Hoyung Chae: Investigation, Writing - original draft, Visualization. E-Wen Huang: Conceptualization, Methodology, Resources, Writing - review & editing, Supervision. Wanchuck Woo: Validation. Suk Hoon Kang: Validation, Writing - review & editing. Jayant Jain: Validation, Writing - review & editing. Ke An: Validation, Investigation, Writing - review & editing. Soo Yeol Lee: Conceptualization, Methodology, Validation, Investigation, Writing - original draft, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This work was supported by a National Research Foundation (NRF) grant funded by the Korean government (2019R1H1A2080092, 2020M2A2A6A05026873, 2017M2A2A6A05017653). A portion of this research used resources at the Spallation Neutron Source, a DOE Office of Science User Facility operated by the Oak Ridge National Laboratory. EWH acknowledges the Ministry of Science and Technology (MOST), Taiwan for financial support through Grant No. MOST-108-2739-M-213-001 from National Synchrotron Radiation Research Center (NSRRC) Neutron Cultivation Program, in providing the trip to use VULCAN of SNS, ORNL in this work. This work was financially supported by the “Center for the Semiconductor Technology Research” from The Featured Areas Research Center Program within the framework of the Higher Education Sprout Project by the Ministry of Education (MOE) in Taiwan. Also supported in part by the Ministry of Science and Technology, Taiwan, under Grant MOST 109-2634-F-009-029, 108-2221-E-009-131-MY4 and Industrial Technology Research Institute (ITRI) 109A502.

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