Solid Freeform Fabrication 2019: Proceedings of the 30th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference
Reviewed Paper

Elevated Temperature Mechanical and Microstructural Characterization of SLM SS304L

G. R. Hecht*, S. P. Isanaka*, and J. W. Newkirk**

*Department of Mechanical and Aerospace Engineering, Missouri University of Science and Technology, Rolla, MO 65409
**Department of Material Science and Engineering, Missouri University of Science and Technology, Rolla, MO 65409

Abstract

SLM built SS304L was annealed and water quenched to minimize residual stress and avoid carbide precipitation. Mini-tensile characterization of strength and elongation at temperature conditions up to 800°C, along with observations of the associated microstructural transformations were utilized to understand the changes produced in SLM SS304L. As-built and annealed specimens were found to exhibit decreasing strength and elongation with increasing temperature as expected. Carbide precipitates appeared after short times at high temperatures within both as-built and annealed specimens for all cases, but no brittle intermetallic phase development was observed for any of the temperatures investigated. While the lack of Sigma, Chi or Laves phases were anticipated, the premature formation of carbides is unexpected behavior for this composition of SS 304L. It is an indication of higher sensitivity of SLM made material. An additional change in the etch response was also observed between as-built and annealed specimens. It is theorized that annealing caused all ferritic and other residual phases present in as-built SLM SS 304L to fully transform into austenite. The cellular structure observed in the as-built specimen was also dissolved due to annealing and water quenching possibly leading to the strength loss observed.

Introduction

Austenitic stainless steels provide excellent high-temperature corrosion resistance and mechanical strength of any alloy group, only bested by nickel-based alloys. For this reason, they are often the ideal choice for applications involving elevated temperatures and extreme oxidizing or reducing atmospheres where mechanical strength is a priority. Austenitic stainless steels also exhibit good elongation at high temperatures, another very favorable characteristic, especially for applications where sudden failure is undesirable.

At elevated temperatures, conventionally manufactured austenitic stainless steels are expected to experience several phenomena leading to reduction in mechanical strength and ductility. As temperatures increase, the required energy for dislocations to slip past one another decreases, allowing plastic deformation to take place at increasingly lower values of applied stress leading to a reduction in mechanical strength. Even at low temperatures, thermal activation affects lattice resistance to dislocation glide. Increased temperature results in a lower required temperature-assisted force to overcome obstacles to dislocation motion [1]. Chromium carbide may also precipitate at elevated temperatures if carbon within the material reaches levels of

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

supersaturation and diffusion rates sufficient for carbon and chromium to segregate into precipitates, causing a reduction in ductility and an increase in hardness [2]. If then subjected to an oxidizing atmosphere, these precipitates are then prone to rapid oxidation which can lead to the virtual disappearance of these metal carbides [3] also causing a reduction of mechanical strength and further reduction of ductility. A third detrimental effect that may be seen in austenitic stainless steels at elevated temperatures is embrittlement due to the formation of a sigma phase. Sigma phase can form in austenitic stainless steels subjected to temperatures ranging from 500˚C to 980˚C after a few thousand hours in service under normal conditions, or almost instantly if the material has been cold worked [4].

Due to limitations of conventional machining and manufacturing, selective laser melting (SLM) is an attractive alternative, allowing geometrically complex three-dimensional parts to be manufactured to a near end-use state, directly from computer-aided design files. Selective laser melting is a powder-bed fusion technique in which successive layers of pre-placed powder are bonded together with the use of a laser. The low powder particle sizes and small input energy spot size in SLM allows parts to be made with high dimensional accuracy and thus require less post-processing when compared to other additive manufacturing techniques [5]. These advantages of increased design flexibility have been adopted by researchers across a range of conventional and novel materials to serve industries like aerospace and healthcare. For example, Concept Laser has produced bracket connectors to be used in the Airbus A350 XWB. This component was previously milled from aluminum but is now fabricated from titanium using an SLM process, reducing the component's weight by over 30% [7].

While SLM and other AM processes create three-dimensional geometries by metallurgically bonding layers of metals and their alloys, they do so in a unique manner. Owing to the small volume of phase change driven by high energy density, AM parts are composed of a complex framework of connected prior meltpools. While these meltpools can be observed microstructurally, their effect on AM-made materials at high temperatures warrant further investigation. Their cooling rates are also extremely high, leading to the possibility of creating unconventional microstructures. Before SS304L components manufactured using SLM can be used extensively at elevated temperatures, a thorough understanding of how the material behaves under these conditions is required. The goal of this research is to characterize the effects of elevated temperature on the mechanical and microstructural properties of SLM made SS304L. SLM made SS304L has been previously shown to exhibit high reactivity when aged at elevated temperatures when compared to its conventionally manufactured counterpart [6]. Thus, similar reactivity is expected to be observed at elevated temperatures.

For the short-term tensile tests used for this characterization study, the main effect on mechanical properties at elevated temperatures is anticipated to be material softening. Due to the short time scale of the tensile tests, carbide precipitation and embrittlement due to sigma phase are very unlikely to be observed. For austenitic stainless steels with a carbon content less than 0.019% by weight, such as the samples used for this study as shown in Table 1, chromium-carbide is only expected to form after 100 hours at temperatures between 500˚C and 600˚C [2]. Sigma phase in austenitic stainless steels, again, only forms after a few thousand hours at temperatures ranging from 500˚C to 980˚C, with the exception of cold worked material for which formation begins instantly. Due to the complex stress-strain field present in SLM made

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

specimens, sigma phase could potentially begin forming in the investigated elevated temperature tensile tests. Simulations based on JMatpro indicate that sigma phase formation is unlikely for conventionally manufactured SS304L with the same chemical composition as the SS304L powder used within this study. Thus, sigma phase is not expected to form in either the as-built or annealed samples of SLM material exposed to high temperature as part of this research. In order to confirm this, both as-built and annealed SLM SS304L samples have been characterized at temperatures ranging from 200˚C to 800˚C for changes in mechanical properties and microstructure. Samples were annealed at 1050˚C and water quenched to minimize possible residual stresses and to avoid carbide precipitation. In the past, researchers have reported high dislocation presence in SLM materials, but annealing and water quenching specimens is theorized to reduce dislocation density and consequently anticipated to increase ductility with the tradeoff of reduced strength in these SLM materials.

Experimental Procedure

Specimens used for this characterization study were built on the Renishaw AM250 additive manufacturing system located at the Missouri University of Science and Technology. The process parameters used for the fabrication of the specimens have been previously optimized to obtain maximum density and include a laser power of 200W, hatch spacing of 85µm, point distance of 60µm, and an exposure time of 75µm. It should be noted that the Renishaw AM 250 uses a point exposure scan strategy. The SS304L powder used for the fabrication of the specimens was purchased from LPW Technology. Table 1 displays the chemical composition of the powder.

Table 1: Chemical Composition of SS304L Powder [9]

Element	C	Mn	S	Si	Cu	Ni	Mo	Cb	Cr	Ti	N
Wt.	0.018	1.4	0.005	0.6	0.05	9.8	0	0	18.4	0	0.06
Element	Al	P	Sn	Pb	Co	V	B	O	Ca		
Wt.	0	0.012	0	0	0	0	0	0.02	0		

Specimens were built as rectangular columns that were then used to make mini-tensile specimens from the interior of each block. This strategy was deployed to ensure that any effects of oxidation were not present on the tensile specimens and thereby avoided from the characterization study. All cutting was performed using a Sodick Electrical Discharge Machine (EDM) to minimize any possible work hardening effects that other harsh machining processes would impart on the properties of the material. The use of mini-tensile specimens rather than an ASTM standard allows many data points to be acquired while minimizing the volume of material needed. This is ideal when dealing with SLM powder that is expensive due to the need for spherical particles with high purity [5] or in other situations where material costs and production volumes come at a premium. Figure 1 displays a drawing of the mini-tensile specimens used for this study which have been developed at the Missouri University of Science and Technology and have a thickness of 1 mm [8].

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

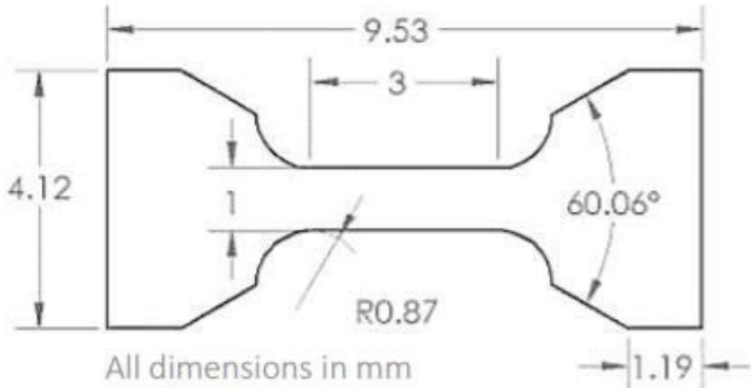

Figure 1: Dimensions of the Mini-Tensile Specimen Developed at the Missouri University of Science and Technology and Used for This Study. [8]

Additionally, before the cutting of mini-tensile specimen, several rectangular columns were annealed by heating at 1050˚C for 30 mins before quenching in water. This was done to avoid carbide precipitation and to minimize the residual stresses inherent to all SLM made specimens due to the rapid thermal cycling, phase change and directional building inherent to all metal AM processes. Prior to running tensile tests, all mini-tensile specimens where polished with 600 grit Silicon Carbide paper to remove any oxide formation from the EDM process.

All of the tests were carried out on an Instron UTM using a custom built elevated temperature testing setup. An Applied Test Systems 3210 series tube furnace was fitted to the Instron UTM machine to allow specimens to be heated to temperatures of up to 1100˚C. Custom built pull rods were manufactured using the nickel based super alloy Inconel X due to its high temperature creep strength and low thermal conductivity. This ensured that high temperature tensile tests could be run for the full operational temperature range of the furnace while ensuring that the sensitive load cell remains well within the recommended calibrated temperature range for accurate measurements. Figure 2 displays the full elevated temperature set up used for this study.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

Figure 2: Elevated Temperature Setup

A total of six short-term tensile tests each were run at room temperature, 200˚C, 400˚C, 600˚C, and 800˚C for the as-built samples, and the annealed and water quenched samples. Samples were held at temperature long enough to ensure uniform temperature and were then pulled at a constant strain rate of 0.06 mm/mm/s while the engineering stress-strain relationship was recorded. After completion of each test, the broken specimens were saved for further analysis.

After completion of tensile tests, specimens were polished and etched with an electrolytic nitric acid setup (60% solution at 30V and 5mA for a 3 second exposure) to prepare for optical microscopy. Optical images were then taken of samples from each group of tests to analyze the microstructural characteristics of each.

JMatPro was also used to simulate the effects of elevated temperature on the mechanical and microstructural properties of conventionally manufactured SS304L with the same chemical composition. A cell size of one micron was also used to simulate grain size and distribution in the software. Intermetallic phase development and carbide precipitation were simulated for temperatures ranging from 100˚C to 800˚C at exposure times up to 1×10^{13} hours. The yield strength was simulated for temperatures ranging from 0˚C to 1400˚C. It should again be noted that JMatPro simulations are based on conventional manufacturing and therefore do not take into account the residual stresses, rapid cooling rates and other effects present in the SLM samples, and so some discrepancy is to be expected.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

Results and Discussion

The results of the JMatPro simulation are displayed below in Figure 3. Sigma and Laves phase formation are predicted to form in this composition of SS304L, although neither are expected to within the temperatures or time frame of this characterization study. Sigma phase is predicted to appear after a few hundred to a few thousand hours at temperatures ranging from 400˚C to 750˚C while Laves phase formation takes nearly 500k hours beginning at around 300˚C. JMatPro predicts the formation of a M_2 (C, N) precipitate but again only after a hundred hours of exposure to 750˚C. From these predictions, embrittlement due to the formation of these brittle intermetallic phases is unlikely within this characterization study of SLM SS304L.

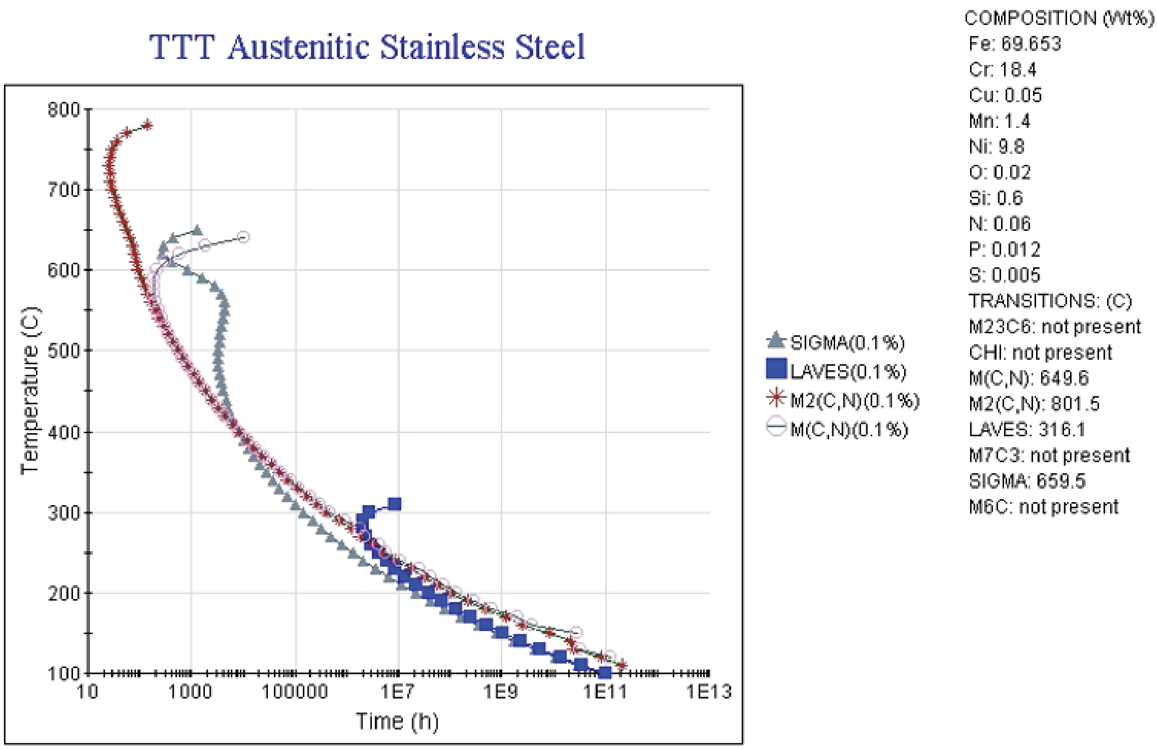

Figure 3: Intermetallic Phase Development Simulated with JMatPro

JMatPro was also used to simulate the effect of elevated temperatures on the yield strength for comparison to the SLM SS304L material. As shown in Figure 4, the software predicts softening of the material with increasing temperature as expected. This behavior can also be confirmed with experimental data, although the physical tests indicate that the as-built specimens exhibit approximately 20 percent higher yield strength when compared to the simulated strength. Simulations are based on conventionally manufactured materials so the discrepancy in the results could be a product of this assumption. Discrepancies could also be due to a higher dislocation density within the as-built specimens compared to the assumptions made within the simulation. Before definitive conclusions can be made, further analysis using TEM is required to get an accurate idea of the dislocation density within the as-built specimen.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

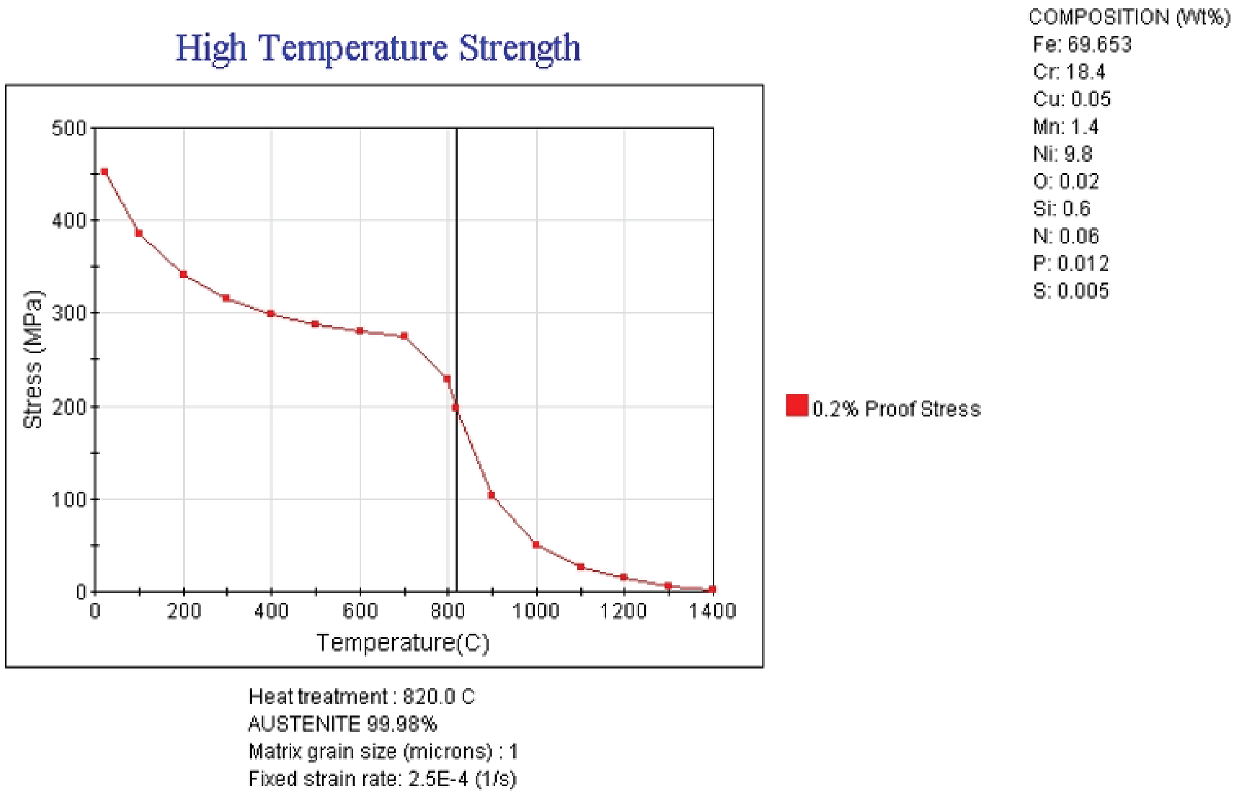

Figure 4: Yield Strength Simulated with JMatPro

Figures 5, 6, 7, and 8 show the experimental results acquired for the as-built and the annealed specimen. As can be seen, the annealed specimens exhibit lower strengths compared to the simulated and as-built data. Inversely, the annealed specimens exhibit much higher elongation at elevated temperatures with remarkable stability for the full range of temperatures investigated. This behavior could be attributed to the dislocation density possibly being reduced during annealing. Additional microstructural transformations could also be contributing to this mechanical performance behavior.

SLM made specimens have been known to possess higher yield strengths when compared to their wrought and cast counterparts while wrought and cast characterization studies are typically used as the basis for the JMatPro simulations. This is confirmed in this study, with the experimental yield strengths of SLM specimens being higher than the predictions from JMatPro. Interestingly, the trends from both the as-built and the annealed strength investigations in Figure 5 match the prediction of conventionally manufactured SS 304L from JMatPro. This implies that the SLM material behaves similar to its conventional counterparts when exposed to high temperatures. Were it not for the starting yield strength being higher in the SLM specimens, the trend might match the JMatPro predictions.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

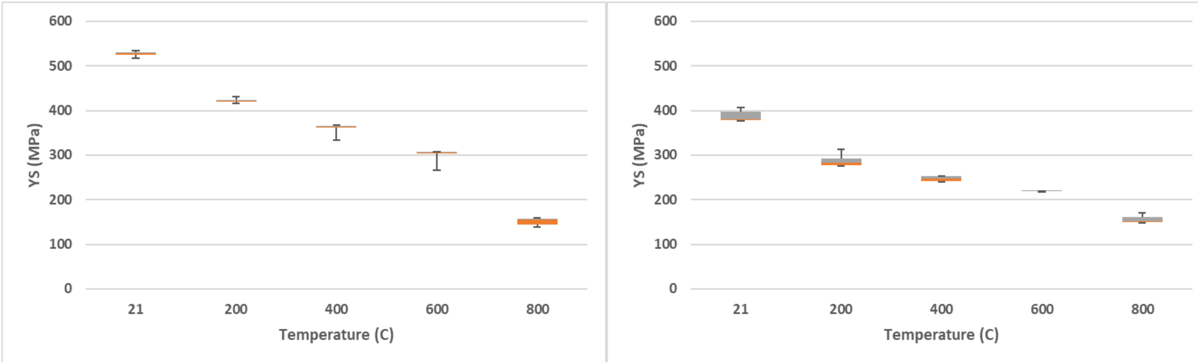

Figure 5: As-built (left) and Water Annealed (right) Yield Strength.

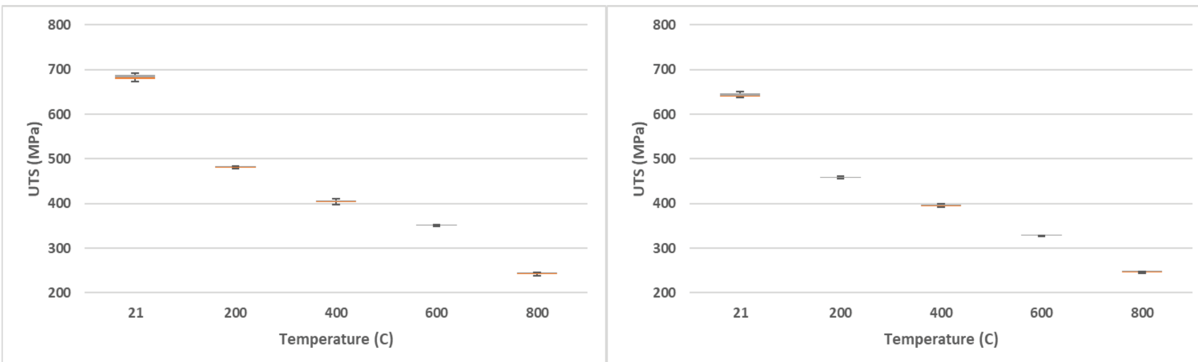

Figure 6: As-built (left) and Water Annealed (right) Ultimate Tensile Strength

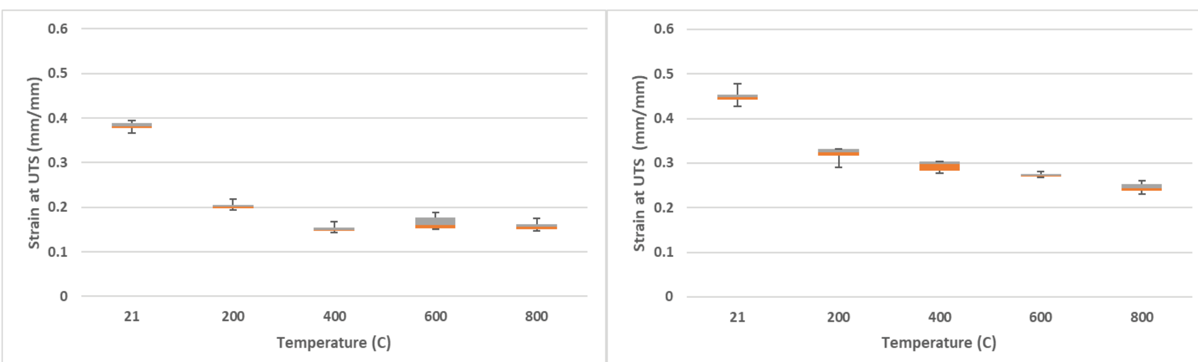

Figure 7: As-built (left) and Water Annealed (right) Strain at Ultimate Tensile Strength

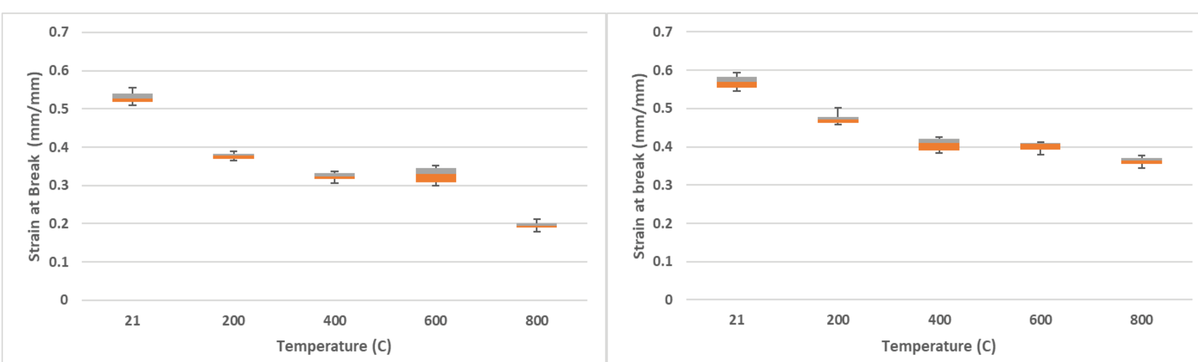

Figure 8: As-built (left) and Water Annealed (right) Strain at Break

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

The microscopy images are shown below in Figures 9 and 10. As can be seen, the etch responses of the as-built and annealed specimens were dramatically different.

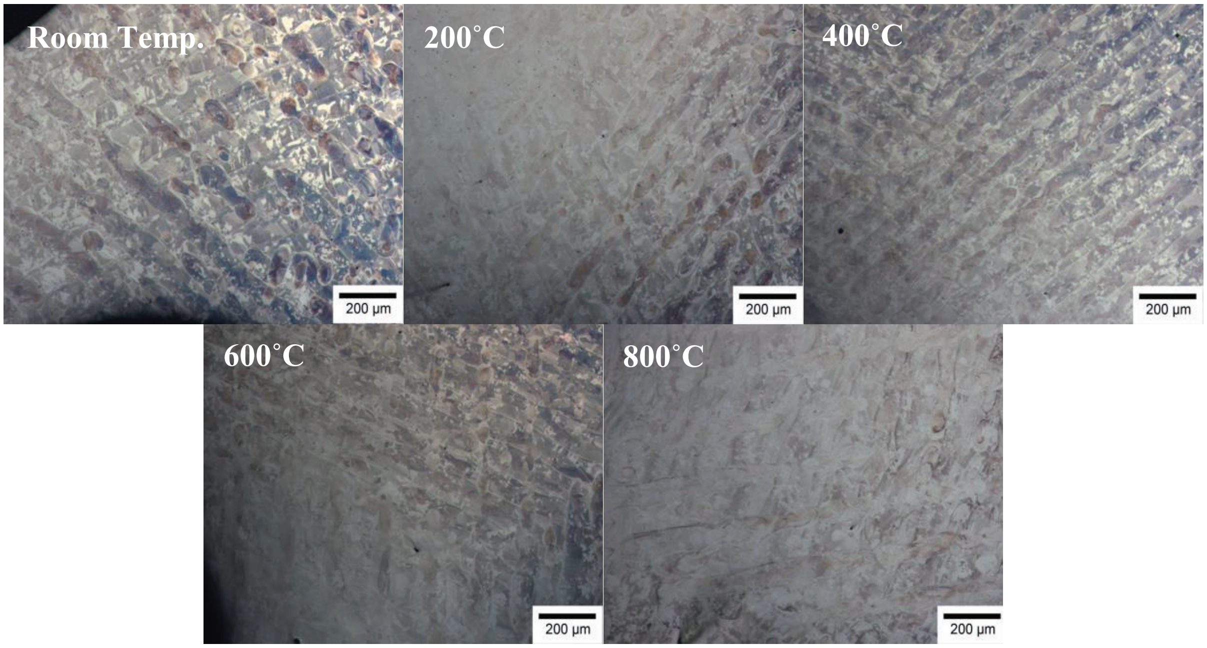

Figure 9: As-Built Specimen Etched with Electrolytic Nitric Acid Setup Under 5x Magnification.

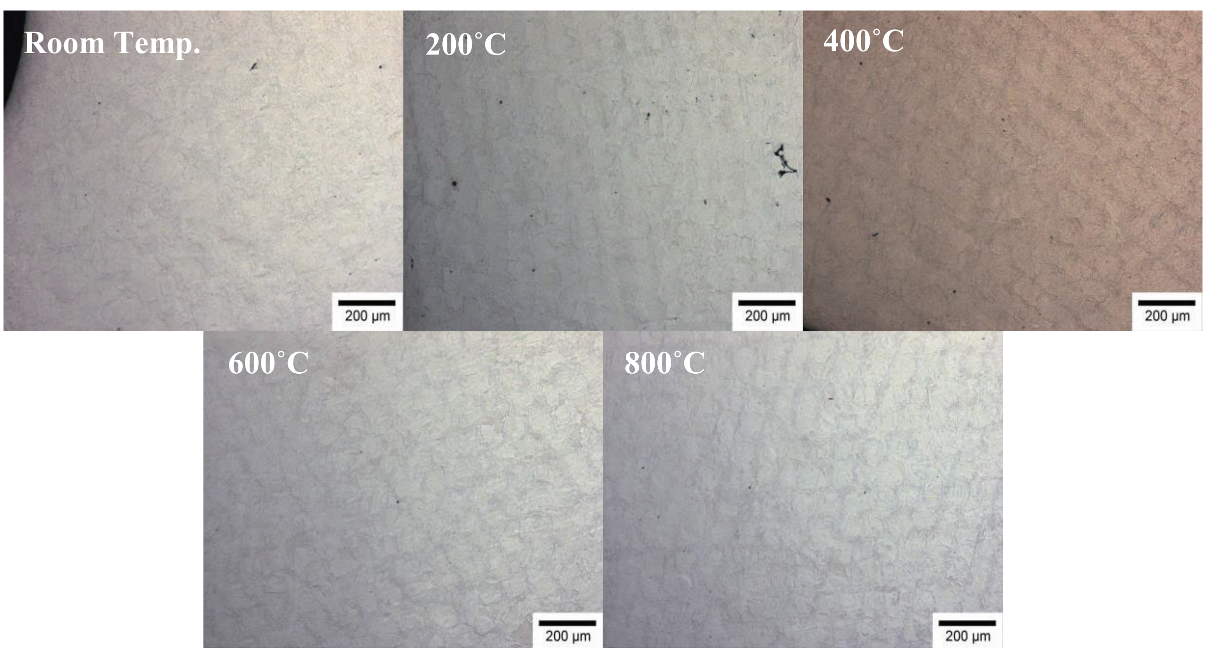

Figure 10: Annealed Specimen Etched with Electrolytic Nitric Acid Setup Under 5x Magnification.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

Within the 100x magnification images of the as-built specimens shown in Figure 11, intermetallic phase development seems to be absent from both the specimens tested at room temperature and 800˚C. Unexpectedly, carbide precipitates do appear to have formed within the as-built specimen tested at 800˚C. The absence of other intermetallic phases agrees with literature and the JMatPro simulations for the short time scale of short-time tensile tests, but the formation of the carbides does not. The loss of strength seen in the experimental characterization could be attributed to the dissolution of the material's cellular structure when exposed to high temperatures, but this also does not seem to be the case of the as-built specimens.

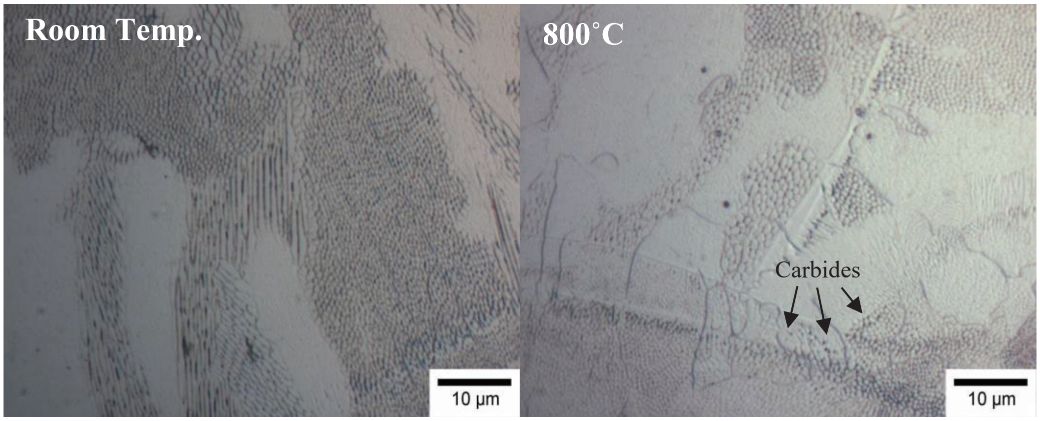

Figure 11: As-Built Specimen Etched with Electrolytic Nitric Acid Setup Under 100x Magnification.

Carbide precipitation was also observed within the annealed and water quenched specimen tested at room temperature as well as at 800˚C as shown in Figure 12. Intermetallic phase development seems to absent from the annealed specimen for both cases shown below. Carbide precipitation was not predicted to occur for either as-built or annealed specimens and is theorized to have been accelerated by the SLM process. Other researchers have found that SLM materials are more sensitive than their conventionally manufactured counterparts and this premature precipitation of carbides could be further indication of that behavior. In both these cases, dislocation slip at high temperatures could be one of the reasons that contribute to the mechanical performance of these materials.

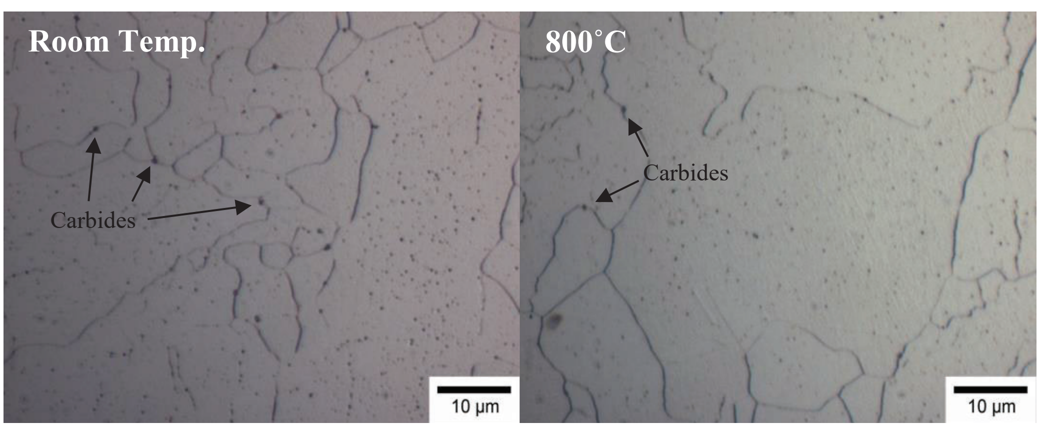

Figure 12: Annealed Specimen Etched with Electrolytic Nitric Acid Setup Under 100x Magnification.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

As stated previously, the etch response of the annealed specimens was dramatically different compared to that of the as-built specimens. Although the electrolytic nitric acid etch procedure was exactly the same, the phase resolution in the annealed case is not apparent. Due to the specimens being SLM made, it is expected to see track and meltpool boundaries in the microstructure. The cellular structure which is present in the as-built SLM specimen is also not apparent in the annealed and water quenched material. It is theorized that annealing the specimens has resulted in a transformation of any ferrite or other residual phases present into austenite, as this could explain the drastic change in etch response compared to as-built specimen. Also, the dissolution of the cellular structure could explain some of the differences within the microstructural images. This could also explain the change in mechanical performance between the as-built and annealed specimen, as the dissolution of the cellular structure could produce a slight loss in strength and a corresponding increase in ductility.

Conclusion

The mechanical and microstructural properties of as-built and annealed SLM SS304L have been investigated at elevated temperatures up to 800˚C. Both as-built and annealed specimens exhibited a decline in strength and ductility as temperatures increased. Intermetallic phase development appeared to be absent within both as-built and annealed specimens for all temperatures investigated. Interestingly, carbide precipitates did appear to be present within the as-built specimens tested at 800˚C, as well as in all of the annealed specimens. Thus, the decline in strength and elongation as temperature increases is theorized to be due to dislocation slip as elevated temperatures reduce the required energy for dislocations to slip past one another, coupled with the loss of the cellular structure in the annealed specimen. Carbide precipitation could also play a role in the decrease in ductility at elevated temperatures. Annealed specimens were observed to exhibit a much different etch response as compared to the as-built specimen. It is theorized that the process of annealing the specimens has resulted in ferritic and other residual phases present to transform fully into austenite as well as the dissolution of the cellular structure.

Future Work

Further investigation is planned to better understand what has been observed during this characterization study. Dislocation studies will be performed employing transmission electron microscopy (TEM). Electron backscatter diffraction (EBSD) will be used to gain a thorough understanding of the microstructural phase transformations observed and the grain size present within the specimens. Finally, further optical microscopy is underway to investigate grain boundary migration and realignment that may have taken place.

Special Thanks

A very special thanks goes out to Alexander Lipari for all of our microscopy imaging, Adam Fleischman for help completing our elevated temperature tensile tests, and Kyle Stagner for preparing all of our samples.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.

References

[1] Courtney TH (2013) Mechanical Behavior of Materials, pp. 298, McGraw Hill Education (India), New Delhi

[2] Kelly, J. (2006). "Stainless Steels." Mechanical Engineers Handbook, pp. 69–78., doi:10.1002/0471777447.ch2.

[3] (1992) High-Temperature Characteristics of Stainless Steels. American Iron and Steel Institute, www.nickelinstitute.org/media/1699/high_temperaturecharacteristicsofstainlesssteel_9004_.pdf.

[4] Kosmač Alenka. (2012). Stainless Steels at High Temperatures, pp.14., Euro Inox, www.bssa.org.uk/cms/File/StainlessSteels_at_HighTemperatures_EN.pdf.

[5] Sutton, A.T., Kriewall, C.S., Leu, M.C., Newkirk, J.W., (2018). Recyclability of 304L Stainless Steel in the Selective Laser Melting Process, in: Solid Free. Fabr. Symp., 2018.

[6] Amine, T.A., (2017). Long-Term Effects of Temperature Exposure on SLM 304L Stainless Steel, JOM Journal of the Minerals, Metals and Materials Society, pp. 384-389, Minerals, Metals and Materials Society (TMS)

[7] Liu, R., Wang, Z., Sparks, T., Liou, F., Newkirk, J.W., (2017). Aerospace applications of laser additive manufacturing, pp. 357-358, Woodh. Pub. Ser. Elect.

[8] Karnati, S., Hoerchler, J. L., Liou, F., Newkirk, J.W., (2017). Influence of Gage Length on Miniature Tensile Characterization of Powder Bed Fabricated 304L Stainless Steel, in Solid Free. Fabr. Symp., 2018.

[9] Home. In: Carpenter Additive. https://www.carpenteradditive.com/.

This work was funded by the Department of Energy's Kansas City National Security Campus which is operated and managed by Honeywell Federal Manufacturing Technologies, LLC under contract number DE-NA0002839.