

L-PBF Ni-201 Feasibility Study Materials Characterization Summary

Nickel 200 and 201 are commercially pure nickel alloys with Ni-201 having a lower carbon content than Ni-200. These alloys are useful for their excellent corrosion resistance, good thermal conductivity, and mechanical properties. Very little information has been published on additive manufacturing (AM) of either Ni-200 or Ni-201, so a feasibility study to produce coupons with Ni-201 powder was performed. This work was carried out at NASA MSFC and Aerojet Rocketdyne. Contributors at NASA MSFC included Brian West, Zack Jones, Colton Katsarelis, and Matt Medders in collaboration with Dan Matejczyk and Alison Park at Aerojet Rocketdyne. The data provided in this report was collected by the MSFC. This feasibility study aimed to do the following:

- 1) Develop AM parameters for Ni-201 that produces high density with minimal detrimental defects
- 2) Examine the microstructural behavior of the alloy through thermal treatments to attempt to optimize the microstructure and understand the material's response to high temperatures
- 3) Obtain first order mechanical and thermal properties of the AM material to compare with the wrought product

Using an EOS M100 platform, preliminary parameters were developed through a series of development builds influenced by [1] that produced coupons (Example in Figure 1) with high densities (>99%) when examined in metallography. It was observed that even in samples where the densities were high, there were still some isolated large defects around 75-100 μm ($\sim 0.003''$ - $0.004''$) evident in the material. Further parameter development will be needed to scale this material up to producing parts, however it has been demonstrated that parameters that produce high density parts are attainable. Table 1 contains the parameters tested during this study.

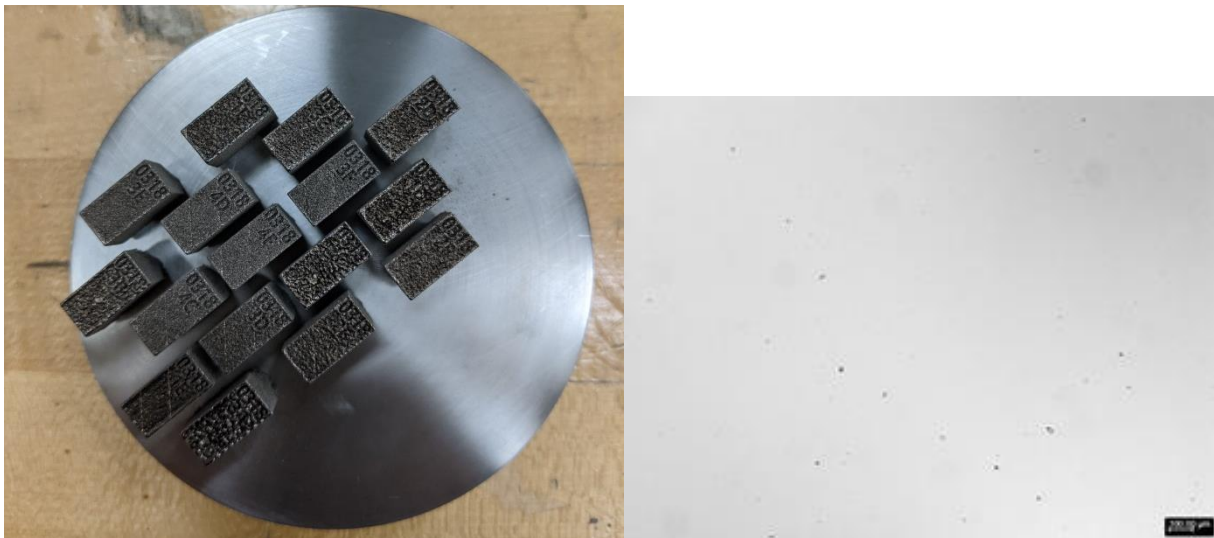


Figure 1: Left: Example of Ni-201 coupons on a build plate from EOS M100. Right: As-polished micrograph of as-built sample. Some minor porosity and lack of fusion defects are apparent.

Table 1: Parameters used to build Ni-201 samples in an EOS M100 AM machine. Constant power and layer thickness were used for parameter development. Data in cells is the volumetric energy density in J/mm³. Colors indicate the severity of defects with red being the worst to shades of green being better.

P = 170W t _L = 0.02mm		Hatch Spacing (mm)					
Scan Speed (mm/s)		0.04	0.05	0.06	0.07	0.08	0.09
	550	386.4	309.1	257.6	220.8	193.1818	171.7172
	650	326.9	261.5	217.9	186.8	163.4615	145.2991
	750	283.3	226.7	188.9	161.9	141.6667	125.9259
	850	250	200	166.7	142.9	125	111.1111
	950	223.7	178.9	149.1	127.8	111.8421	99.4152
	1050	202.4	161.9	134.9	115.6	101.1905	89.94709
	1150	184.7826	147.8261	123.1884	105.5901	92.3913	82.1256
	1250	170	136	113.3333	97.14286	85	75.55556
	1350	157.4074	125.9259	104.9383	89.94709	78.7037	69.95885
	1450	146.5517	117.2414	97.70115	83.74384	73.27586	65.1341

During the parameter development, sections from coupons were used to examine the material's response to heat treatment. Heat treatment development examined at MSFC used temperatures ranging from 1300-1900°F with most soaking times at 30 minutes in air while wrapped in foil. Initial experiments up to 1900°F showed that the microstructure remained similar to the as-built microstructure regardless of the build parameters that were tested (Figure 2). It has been hypothesized that a very fine secondary phase may be pinning the grain boundaries of the material preventing grain growth, which could be beneficial in higher temperature forming processes as the microstructure would remain stable up to at least 2050°F. Another hypothesis is that the residual stresses in the samples were not high enough to drive recrystallization during heat treatment. These stresses are influenced by the build parameters, so further adjustment may change the response to heat treatment. As seen with many other alloys in AM, a similar microstructure to the wrought version of Ni-201 may not be attainable through heat treatment optimization [2,3]. Hot isostatic pressing (HIP) was unable to be performed due to time constraints on the process but should be explored if future studies are performed.

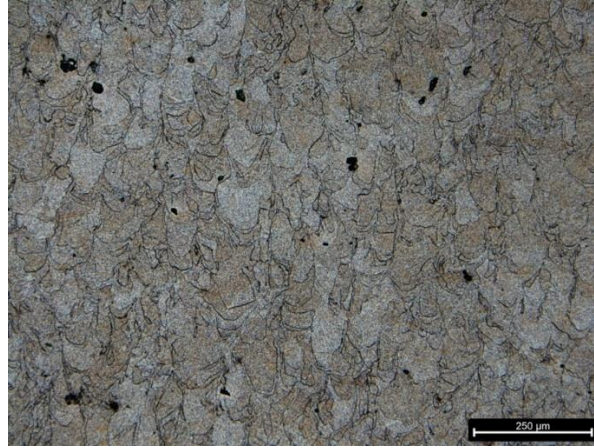


Figure 2: Micrographs of microstructure of AM Ni-201. Left: As polished sample showing porosity and some minor lack of fusion defects in the as-built condition. Right: Etched grain structure after annealing at 1900 °F for 30 minutes.

Upon settling on one of the better parameter sets produced, several subscale tensile samples were produced across two sample builds to test at room temperature and obtain initial tensile properties. The two sample builds are distinguished by build work order numbers 20-0391 and 21-0054. Samples were foil wrapped and heat treated in an air furnace at 1900 °F for 30 minutes. During machining, two samples fractured due to unexpected amounts of porosity in the material. Despite the machining difficulties, many successful tensile tests were performed with the rest of the samples. The average strengths and fracture elongation are shown in Figure 1. The test results showed consistent yield strengths in the range of 25-30 ksi, while ultimate strengths regularly exceeded 40 ksi with the maximum being 52 ksi. The build 20-0391 was observed to have produced the higher strengths and ductility which may have been due to operational differences in the two builds. The fracture elongation of the test samples was impacted the most by the porosity with the 21-0054 build being impacted more than the other creating a wide range of elongations. The 20-0391 build had a fracture elongation ranging from 15-35% while the 21-0054 samples had a range of 8-21%.

During examination of the tensile curves and fracture surfaces, ductile behavior was generally observed even in the lower elongation samples. Many large voids with unmelted powder were observed in the fracture surfaces (Figure 4) showing that further parameter development is needed. The tensile testing results are promising for the material's feasibility, and with further parameter development to improve the defect state of the material improvement of mechanical properties is expected.

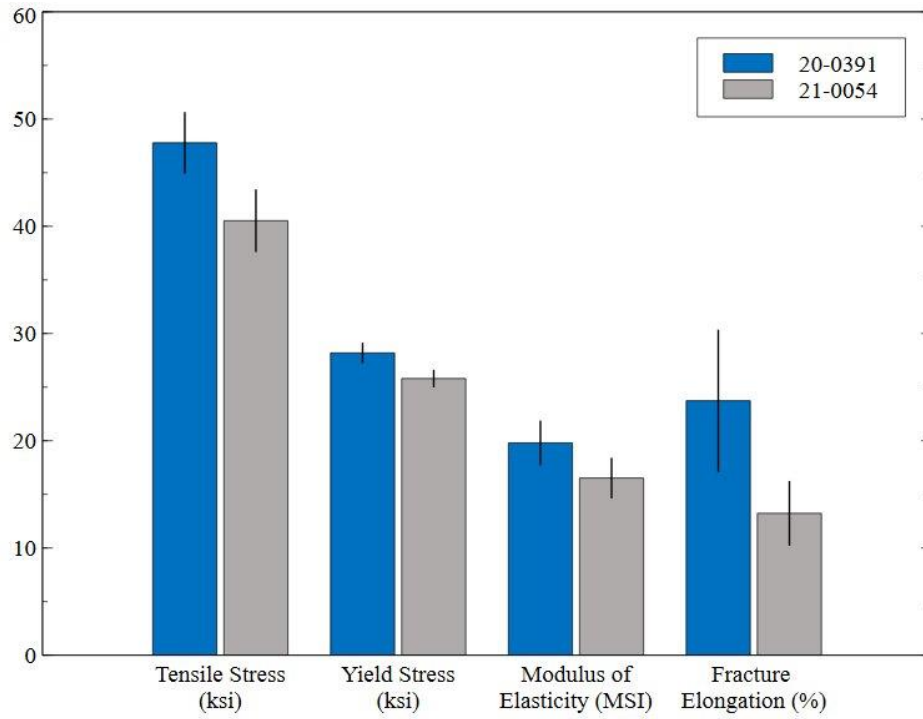


Figure 3: Average ultimate tensile strength, yield strength, and fracture elongation of L-PBF Ni-201 samples tested in EM22 at NASA MSFC.

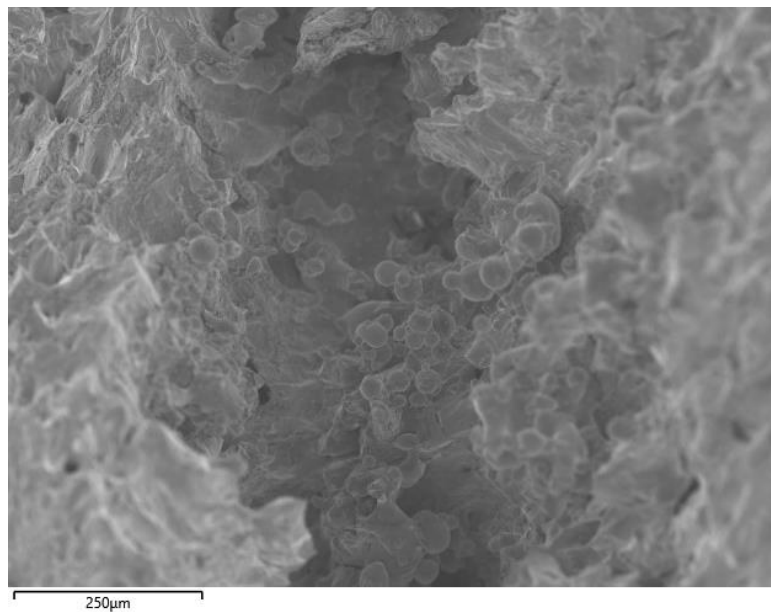


Figure 4: Unmelted powder particles were observed in the fracture surface during SEM examination

Conclusions and Recommendations:

- 1) Through parameter development, high density parts were capable of being produced showing that Ni-201 can be built using AM. However, further development would be needed if the material is to be used for building parts. Large defects were still found in the material with the parameters being used, so better parameters would need to be developed. For building parts with Ni-201, it is recommended to scale up to an EOS M280/290 platform with the size capability for required parts. This scaling will require the parameter development needed for good parts.
- 2) Thermal processing evaluation has shown that AM Ni-201 has a very stable microstructure up to 2050°F that may be excellent for any post processing that may be needed. While 1900°F for 30 minutes was used, it is recommended that other heat treatments be explored for use with parts including HIP.
- 3) First order tensile properties and thermal conductivity were obtained through testing at MSFC and AR, respectively. The tensile properties showed that decent ductility and good strengths are attainable with AM Ni-201 while thermal conductivity results showed a moderate drop from wrought. Both results appeared to be heavily influenced by large defects that were not eliminated from the material. Further testing is recommended with better material in addition to higher fidelity methods of thermal conductivity such as flash diffusivity.

References:

- [1] Yap, C. Y., Tan, H. K., Du, Z., Chua, C. K., and Dong, Z. "Selective Laser Melting of Nickel Powder." *Rapid Prototyping Journal*, Vol. 23, No. 4, 2017, pp. 750–757. doi:10.1108/RPJ-01-2016-0006.
- [2] Shaikh, A. S., Schulz, F., Minet-Lallemant, K., and Hryha, E. "Microstructure and Mechanical Properties of Haynes 282 Superalloy Produced by Laser Powder Bed Fusion." *Materials Today Communications*, Vol. 26, 2021. doi:10.1016/j.mtcomm.2021.102038.
- [3] Leicht, A., Rashidi, M., Klement, U., and Hryha, E. "Effect of Process Parameters on the Microstructure, Tensile Strength and Productivity of 316L Parts Produced by Laser Powder Bed Fusion." *Materials Characterization*, Vol. 159, 2020. doi:10.1016/j.matchar.2019.110016.