

# RAPID ALLOY SELECTION AND ADDITIVE MANUFACTURING OF NICKEL-BASED SUPERALLOYS



Michael M. Kirka, ORNL  
Patxi Fernandez-Zelaia, ORNL  
Christopher Ledford, ORNL  
Julio Ortega Rojas, ORNL  
Quinn Campbell, ORNL

**Oct 2022**

**CRADA FINAL REPORT  
NFE-20-8169**

**Approved for Public Release  
Distribution is Unlimited**



## DOCUMENT AVAILABILITY

Reports produced after January 1, 1996, are generally available free via OSTI.GOV.

**Website** [www.osti.gov](http://www.osti.gov)

Reports produced before January 1, 1996, may be purchased by members of the public from the following source:

National Technical Information Service  
5285 Port Royal Road  
Springfield, VA 22161  
**Telephone** 703-605-6000 (1-800-553-6847)  
**TDD** 703-487-4639  
**Fax** 703-605-6900  
**E-mail** [info@ntis.gov](mailto:info@ntis.gov)  
**Website** <http://classic.ntis.gov/>

Reports are available to US Department of Energy (DOE) employees, DOE contractors, Energy Technology Data Exchange representatives, and International Nuclear Information System representatives from the following source:

Office of Scientific and Technical Information  
PO Box 62  
Oak Ridge, TN 37831  
**Telephone** 865-576-8401  
**Fax** 865-576-5728  
**E-mail** [reports@osti.gov](mailto:reports@osti.gov)  
**Website** <https://www.osti.gov/>

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

Manufacturing Science Division

**RAPID ALLOY SELECTION AND ADDITIVE MANUFACTURING OF NICKEL-BASED  
SUPERALLOYS**

**AUTHOR(S)**

Michael M. Kirka, ORNL  
Patxi Fernandez-Zelaia, ORNL  
Christopher Ledford, ORNL  
Julio Ortega Rojas, ORNL  
Quinn Campbell, ORNL

October 2022

Prepared by  
OAK RIDGE NATIONAL LABORATORY  
Oak Ridge, TN 37831  
managed by  
UT-BATTELLE LLC  
for the  
US DEPARTMENT OF ENERGY  
under contract DE-AC05-00OR22725



## CONTENTS

<b>RAPID ALLOY SELECTION AND ADDITIVE MANUFACTURING OF NICKEL-BASED SUPERALLOYS</b>	<b>3</b>
<b>AUTHOR(S)</b>	<b>3</b>
<b>CONTENTS</b>	<b>III</b>
<b>ACKNOWLEDGEMENTS</b>	<b>4</b>
<b>ABSTRACT</b>	<b>5</b>
<b>1. RAPID ALLOY SELECTION AND ADDITIVE MANUFACTURING OF NICKEL-BASED SUPERALLOYS</b>	<b>5</b>
<b>1.1 Background</b>	<b>5</b>
<b>1.2 TECHNICAL RESULTS</b>	<b>5</b>
1.2.1 Experimental methods – Weld Tracks	5
1.2.2 Optical Imaging – Weld Tracks	6
1.2.3 Powder Production	6
1.2.4 Electron Beam Melting Fabrication	7
1.2.5 Room and High Temperature Tensile Testing	8
1.2.6 Complex geometry fabrication	9
<b>1.3 Impacts</b>	<b>10</b>
<b>1.4 Subject Inventions</b>	<b>10</b>
<b>1.5 Conclusions</b>	<b>10</b>
<b>2. PARTNER BACKGROUND</b>	<b>11</b>

## ACKNOWLEDGEMENTS

This CRADA NFE-20-8169 was conducted as a Technical Collaboration project within the Oak Ridge National Laboratory (ORNL) Manufacturing Demonstration Facility (MDF) sponsored by the US Department of Energy Advanced Manufacturing Office (CPS Agreement Number 24761). Opportunities for MDF technical collaborations are listed in the announcement “Manufacturing Demonstration Facility Technology Collaborations for US Manufacturers in Advanced Manufacturing and Materials Technologies” posted at <http://web.ornl.gov/sci/manufacturing/docs/FBO-ORNL-MDF-2013-2.pdf>. The goal of technical collaborations is to engage industry partners to participate in short-term, collaborative projects within the Manufacturing Demonstration Facility (MDF) to assess applicability and of new energy efficient manufacturing technologies. Research sponsored by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

---

## ABSTRACT

In this work the rapid alloy selection and subsequent processability via additive manufacturing of nickel-based superalloys was investigated. Rapid alloy selection protocols developed within the project produced two viable nickel-based superalloys. This phase I CRADA demonstrates a viable route for selecting new nickel-based superalloys for additive manufacturing.

### 1. RAPID ALLOY SELECTION AND ADDITIVE MANUFACTURING OF NICKEL-BASED SUPERALLOYS

This phase I technical collaboration project (ORNL/TM-2022/2787) began on August 1, 2020 and concluded on October 31, 2022. The collaboration partner Carpenter Technology Corporation is a large business. This CRADA was associated with a larger program that included ORNL in collaboration with AMES (lead), Sandia, Kansas City National Security Campus (KCNSC), Argonne National Laboratory, Siemens Energy, and General Electric. This project demonstrated the ability for rapid alloy selection techniques to identify two viable high temperature nickel-based (Ni-based) superalloys for processing via additive manufacturing.

#### 1.1 BACKGROUND

The overall Science-based Acceleration of the Full Value Stream for Metal Additive Manufacturing (AM): Expedited Powder Development (“X-P4AM”) project objective is to drastically reduce the time-to-market barriers for new additive alloys of interest in automotive and aerospace applications, through computational alloy design with rapid screening and down-selection via synthesis of candidate alloys with rapid solidification. The project will also refine the technology in high pressure gas atomization to improve the production of commercial quantities of selected powders with high powder yields and enhanced powder quality. New alloy compositions, enhanced powder production, and optimized AM build parameterization will provide a critical step in widespread adoption of AM technology for automotive and aerospace applications. This CRADA project is part of a larger program between ORNL and AMES (lead), Sandia, KCNSC, Carpenter Technology Corporation, Siemens Energy, and General Electric.

#### 1.2 TECHNICAL RESULTS

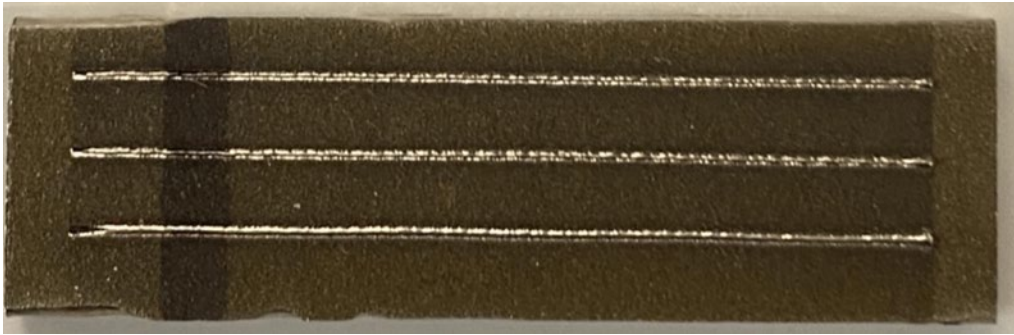
##### 1.2.1 Experimental methods – Weld Tracks

The initial task of this program was to utilize single line melt tracks in an electron beam melting system to determine crack susceptibility of various Ni-based superalloy chemistries. The base alloy samples were arc melted castings which were thinned by wire EDM. A total of 3 weld tracks were melted on 11 different chemistries in a GE Additive Arcam Q10plus. See Table 1 for melt parameters of the 3 weld tracks along with Figure 1 showing a representative sample.

*Table 1: Melt parameters for individual weld tracks*

Weld Track	Beam Current (mA)	Beam Speed (mm/s)	Focus Offset (mA)
1	8	500	15

2	8	1000	15
3	8	1500	15



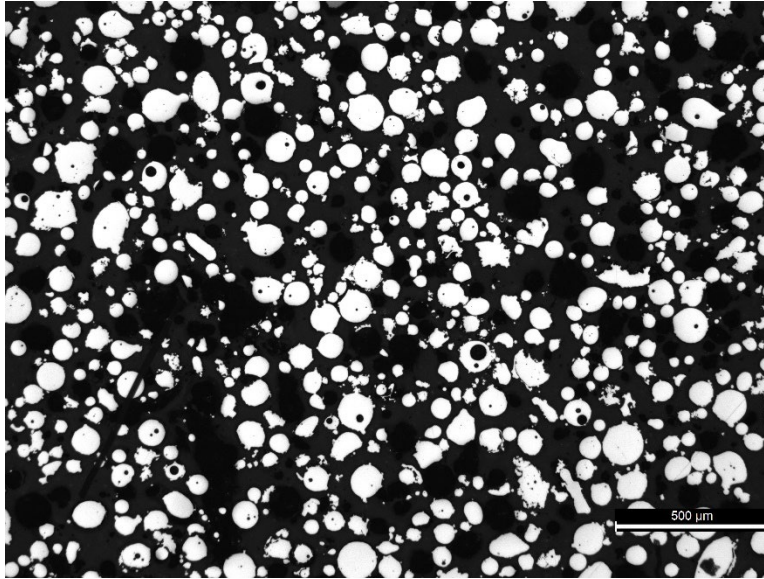
*Figure 1: Representative weld track sample*

### **1.2.2 Optical Imaging – Weld Tracks**

First stitched optical images were taken of the surface of each weld track for all alloy compositions. Each alloy sample was then cut perpendicular to the weld line direction and samples were mounted then polished and optical images were taken on each sample. The optical images were used to characterize the degree of cracking in the weld pool and surrounding cast material. Roughly half the alloys showed some degree of cracking in the weld pools and among those that did not crack, two nickel superalloy chemistries were chosen for further investigation for additive manufacturing.

### **1.2.3 Powder Production**

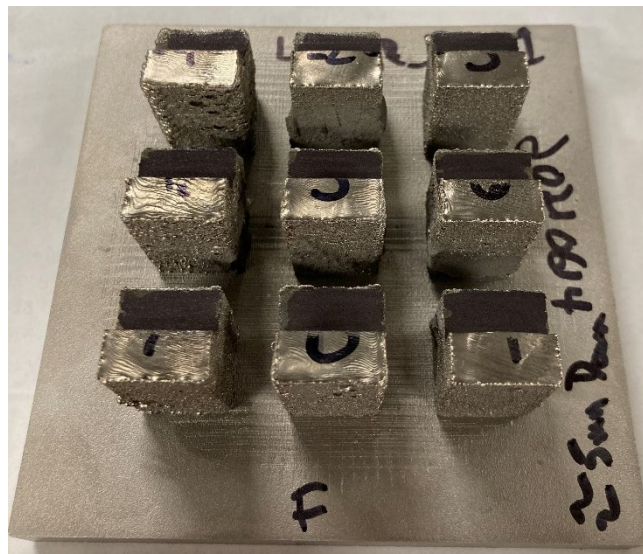
For each of the two selected chemistries, a custom powder lot was procured in the 53-106 um size range. These powders will be referred to as Nickel 0 and Nickel 1. A representative image of the sectioned powder is shown in Figure 2. A fair amount of trapped gas porosity is seen in the sectioned micrograph and was consistent across both powder lots.



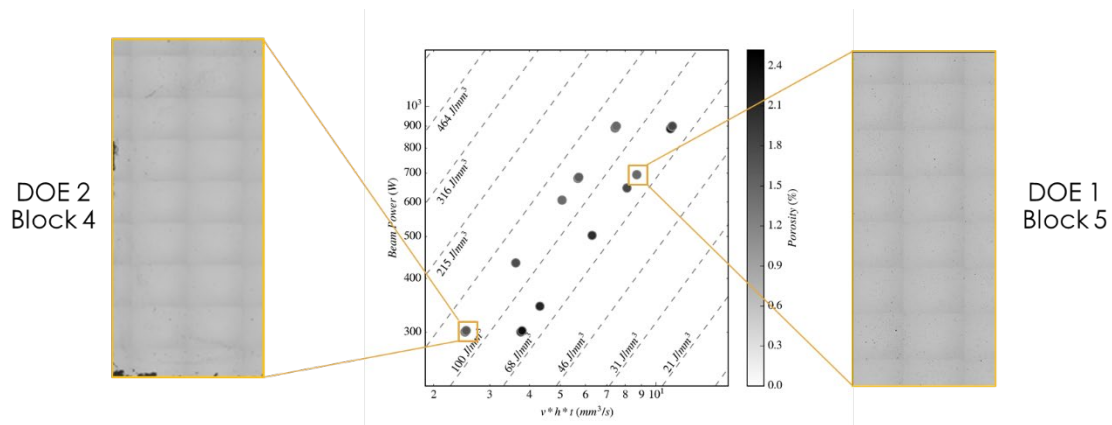
*Figure 2: Representative cross sectioned image of the custom powder lots*

#### **1.2.4 Electron Beam Melting Fabrication**

To determine the printability of the two selected alloys, a GE Additive Arcam Q10plus was utilized to fabricate rectangular samples following a design of experiments (DoE) approach. Figure 3 shows a representative photo of a DoE sample set. The process parameter space was mapped for the initial alloy chemistry as seen in Figure 4.



*Figure 3: Representative photo of a DoE sample set*



*Figure 4: Process space map showing highest density parameters with micrographs*

A parameter set was chosen (DOE 1 Block 5) which displayed high material density along with minimal to no cracking in the solid samples. Three sets of 9 longitudinal rectangular blocks in each of the 2 powder chemistries using the same parameter set were fabricated for microstructural analysis and tensile testing.

### 1.2.5 Room and High Temperature Tensile Testing

Both room temperature and high temperature (600°C, 800°C, and 950°C) quasi-static tensile testing was performed on longitudinally oriented rectangular bars fabricated from both the Nickel 0 and Nickel 1 alloys. Yield strength, ultimate tensile strength, and elongation as a function of temperature are shown in Figure 5. Both Nickel 0 and Nickel 1 show comparable or increased properties when compared to Inconel 738 superalloy. These results show promise as Inconel 738 is widely used in gas turbine engines but can be quite difficult to process via additive manufacturing due to susceptibility to cracking.

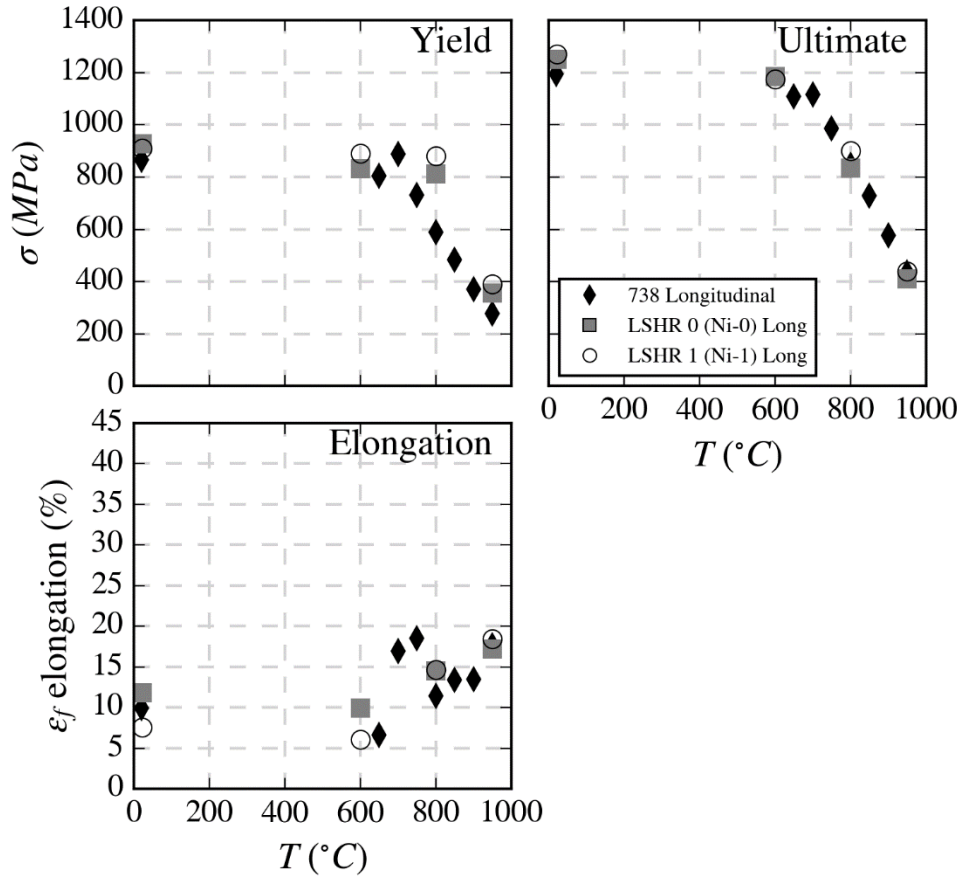
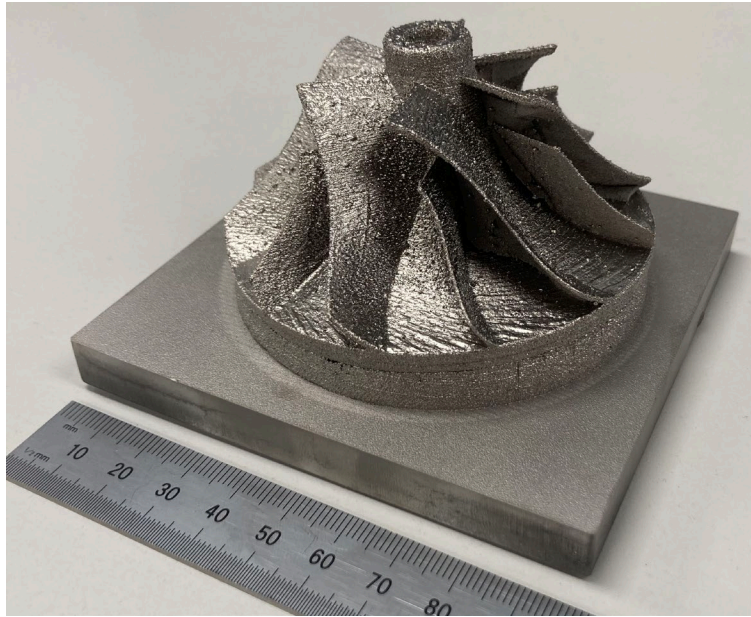


Figure 5: Yield strength, ultimate tensile strength, and elongation as a function of temperature for Nickel 0 and Nickel 1 longitudinal bars

### 1.2.6 Complex geometry fabrication

In order to show proof of concept for the selected alloys, a generic turbo compressor impeller was fabricated as seen in Figure 6. Slight modifications were made to the bulk processing parameters to compensate for the thin walls of the impeller.



*Figure 6: Turbo compressor impeller fabricated in Nickel 1*

### **1.3 IMPACTS**

New alloy exploration is normally a time consuming and expensive process. By developing new techniques to help accelerate the selection process especially for alloys destined for processing via additive manufacturing, new and novel alloys can be quickly transitioned into production. Nickel 0 and Nickel 1 both show promise for use in high temperature applications where the traditional alloys are not quite as amenable to the additive manufacturing process.

### **1.4 SUBJECT INVENTIONS**

NA

### **1.5 CONCLUSIONS**

In phase I of this work, rapid alloy selection techniques were used to identify two nickel-based superalloys that would be amenable to additive manufacturing processes. High density crack free material was produced using electron beam melting. High temperature tensile testing showed favorable properties when compared to legacy alloy Inconel 738. Using the processing parameters developed for the alloys, a turbo compressor impeller was fabricated to show possibilities for complex geometries.

---

## **2. PARTNER BACKGROUND**

Carpenter Technology Corporation is a leading producer and distributor of premium specialty alloys, including titanium alloys, nickel and cobalt based superalloys, stainless steels, alloy steels and tool steels. Carpenter's high-performance materials and advanced process solutions are an integral part of critical applications used within the aerospace, transportation, medical and energy markets, among other markets. Building on its history of innovation, Carpenter's wrought and powder technology capabilities support a range of next-generation products and manufacturing techniques, including novel magnetic materials and additive manufacturing.