Cast Components for High Temperature Concentrated Solar Power Thermal Systems



G. Muralidharan Shivakant Shukla Roger Miller Donovan Leonard Oak Ridge National Laboratory

Jim Myers
Paul Enders
MetalTek International

August 2022



DOCUMENT AVAILABILITY

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

Website 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

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
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.

Concentrated Solar Program

CAST COMPONENTS FOR HIGH TEMPERATURE CONCENTRATED SOLAR POWER THERMAL SYSTEMS

G. Muralidharan, Shivakant Shukla, Roger Miller, Donovan Leonard Oak Ridge National Laboratory

> Jim Myers Paul Enders MetalTek International

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, TN 37831
managed by
UT-BATTELLE LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR227

CONTENTS

Abstract	4
Background	5
Introduction	6
Project Results and Discussion	15
Summary and Conclusions	65
References	67

ABSTRACT

Concentrating Solar-Thermal Power (CSP) components such as piping, valves fittings are required for use at temperatures up to 800°C. These are anticipated to be made using Nickelbased alloys such as Haynes[®]230[®], Haynes[®]282[®], or IN[®]740H[®] and can contribute significantly to the cost of a CSP Gen 3 plant. Thus, there is a significant motivation to lower the cost of materials and components so that the capital costs can be minimized. In all these cases, the cost of the component has two contributions: 1. Materials cost, and 2. Manufacturing cost. Both materials cost and manufacturing costs must be kept low to attain the lowest possible cost. Materials cost can be lowered by using materials that have the ideal combination of properties at the lowest cost. Another avenue to lower cost is chose a manufacturing process that has the potential to lower the cost. Traditionally tubes are made from billets through a wrought process and can be expensive. An alternative process to consider is the centrifugal casting process where the tube is directly fabricated from molten metal. In this case, the molten metal is poured inside a cylindrical metallic mold with an insulating layer and is spun rapidly. A wide range of sizes (diameter, wall thickness, and length) can be cast using this process. The objective of this project was to develop the process for fabricating pipes and related components using a centrifugal casting process and to measure the properties of alloys fabricated using this process.

In the first two years of the project, small laboratory scale heats of Haynes®230® (baseline alloy), Haynes®282® and IN®740H® were cast and their tensile were evaluated between room temperature and 800°C. Haynes®230®, and Haynes®282® were down-selected and industrial scale heats were cast using investment casting by our industrial partner MetalTek International. High temperature mechanical testing on heat-treated investment cast Haynes®230®, Haynes®282® performed at ORNL showed that cast Haynes®282® satisfied the tensile and creep property requirements required for the end application.

Pipes of Haynes®230®, Haynes®282® were successfully centrifugally cast by MetalTek International and tensile properties of the base alloy and Haynes®282® have been measured. Results confirm that Haynes®282® can satisfy the tensile property requirements required for the end application in the centrifugal cast and heat-treated condition. Creep testing shows that the centrifugally cast Haynes®282® has creep properties comparable to or better than that of wrought Haynes®282® except at high stress levels. Creep testing also shows that the centrifugally cast Haynes®282® will have a creep rupture live of 10,000 hours at 750°C at a stress level of at least 134 MPa.

In the final year of the project, welding trials have been successfully completed at ORNL using plates fabricated from centrifugally cast pipe. In addition, centrifugally cast pipe was also welded by the industrial partner MetalTek International using typical manufacturing procedures. Tensile tests at room temperature and at higher temperatures have been completed this year on post weld heat-treated centrifugally cast Haynes[®]282[®].

Background

Components such as piping, valves fittings are required for use at temperatures up to 800°C. These are anticipated to be made using Ni-based alloys such as Haynes [®]230[®], Haynes [®]282[®], or IN®740H® and can contribute significantly to the cost of a CSP Gen 3 plant. Thus, there is a significant motivation to lower the cost of materials and components so that the capital costs can be minimized. In all these cases, the cost of the component has two contributions: 1. Materials cost, and 2. Manufacturing cost. Both materials cost and manufacturing costs have to be kept low to attain the lowest possible cost. Materials cost can be lowered by using materials that have the ideal combination of properties at the lowest cost. Another avenue to lower cost is chose a manufacturing process that has the potential to lower the cost. Traditionally tubes are made from billets through a wrought process and can be expensive. An alternative process to consider is the centrifugal casting process where the tube is directly fabricated from molten metal. In this case, the molten metal is poured inside a cylindrical metallic mold with an insulating layer and is spun rapidly. A wide range of sizes (diameter, wall thickness, and length) can be cast using this process. The objective of this project was to develop the process for fabricating pipes and related components using a centrifugal casting process and to measure the properties of alloys fabricated using this process. Both developmental and commercial alloys were initially considered alloys with the best combination of properties and lowest cost were down selected for final property measurements and weldability testing. Although previous work has been performed on sand and centrifugal casting of Haynes[®]282[®], the diameter of the tube that was cast was much larger than the one that is likely to be used in Gen 4 CSP systems.

Introduction

The following are the descriptions of the various tasks that were conducted in this project.

- **Task 1:** Task 1 focused on making laboratory scale castings of candidate alloys, microstructural characterization, evaluating effect of homogenization and aging heat-treatments, and measuring high temperature tensile properties. Results from the tests on cast alloys were compared with that from the wrought equivalent of the same alloy.
- **Task 1.1. Fabrication of laboratory scale castings**: An initial group of four alloys that included Hayes $^{\$}230^{\$}$, Haynes $^{\$}282^{\$}$, Inconel $^{\$}740H^{\$}$, and one other alloy selected from several candidate alloys that include development alloys such as 161 were fabricated in laboratory scale castings of $\sim 1"x \ 1"x \ 3-4"$ in size using arc melting and drop casting. 2-4 ingots were cast for each alloy to ensure that sufficient material was available for high temperature tensile property evaluation.
- Task 1.2 Microstructural characterization of laboratory scale castings: Microstructure of the alloys were characterized in the as-cast condition to understand secondary dendrite arm spacing and the effect of casting on the formation of interdendritic phases. Optical and scanning electron microscopy along with x-ray microchemical characterization were used to evaluate the microstructure and interdendritic segregation during solidification. Effect of homogenization annealing treatment on reducing segregation in these alloys was also evaluated.
- Task 1.3 Evaluation of tensile properties of laboratory scale castings: Tensile properties (uniaxial- yield strength, tensile strength, and elongation at failure) were evaluated at room temperature, 750°C, and 800°C in the solution annealed (in the cast non-age hardenable alloys), and solution annealed + aged condition in the cast age-hardenable alloys. Solution annealing + aging treatments conditions were selected on the basis of literature data and based upon previous experience with wrought alloys. Two to three specimens were tested at each temperature of interest. Two alloys that met the tensile property requirements and a baseline alloy were down-selected for industrial-scale casting trials. The criterion for down-selection was that highest measured yield strength of the alloys between 750°C and 800°C should be ≥80% of the lowest yield strength of the wrought alloy Haynes®282® between 750°C and 800°C obtained from the literature.
- **Task 2**: Task 2 was focused on making industrial scale castings (both static and centrifugal), measurement of high-temperature mechanical properties including short-term (time independent) tensile testing, uniaxial creep testing, and fatigue testing. Results from the tests on cast alloys were compared with the wrought equivalent of the same alloy.
- Task 2.1. Fabrication of static industrial scale castings: The baseline alloy Haynes[®]230[®] and two additional alloys from the initial set of alloys that meet the tensile property requirements in Milestone 1.2 were melted in industrial scale heats of about ~200 lb or greater and plates were cast in graphite or other molds for microstructural characterization and mechanical property testing. Thickness of the plates would be similar to that required for piping in CSP systems. Multiple castings were obtained from each heat and were used for testing.
- Task 2.2 Microstructural characterization static industrial scale castings: Microstructure of the alloys were characterized in the as-cast condition to evaluate the effect of industrial

process conditions on the as-cast microstructure. Optical and scanning electron microscopy along with x-ray microchemical characterization were used to evaluate the as-cast microstructure and interdendritic segregation during solidification. Effect of annealing treatment selected using previous work with laboratory scale heats in Task 1.2 on homogenization and microstructural evolution were also evaluated.

Task 2.3 Evaluation of tensile properties of static industrial scale castings: Tensile properties were evaluated at room temperature, 750°C, and 800°C in the solution annealed (in the cast non-age hardenable alloys), and solution annealed + aged condition in the case of age-hardenable alloys. Solution annealing + aging treatments conditions were selected on the of previous work on laboratory scale castings outlined in Task 1.3. Two to three specimens were tested at each temperature of interest.

Task 2.4 Evaluation of creep properties of laboratory scale and industrial scale static castings: Isothermal, uniaxial creep testing were performed on up to two promising alloys that meet the tensile property requirements and a baseline alloy under constant load conditions. Creep property screening included two completed creep tests for each alloy with one test expected to exceed the creep rupture life of 250 hours and one creep test expected to exceed rupture life of 500 hours in the solution annealed or solution annealed + aged condition as appropriate, at temperatures in the range of 700°C - 800°C. Specimens were selected from laboratory scale and/or industrial scale heats of the same alloy compositions. This work was expected to lead to the down-selection of two alloys for centrifugal casting. It was expected that at least one alloy would satisfy BOTH the tensile strength and creep strength requirements.

Uniaxial tensile property requirement: Highest measured yield strength of the down-selected alloys between 750°C and 800°C should be ≥80% of the lowest yield strength of the wrought alloy Haynes[®]282[®] between 750°C and 800°C obtained from the literature.

Creep property requirement: Creep rupture life of down-selected alloy should be ≥85% of the lower limit in the scatter band in creep rupture life of wrought alloy Haynes [®]282[®] at equivalent stresses and temperatures.

Task 2.5 Fabrication of centrifugal castings: Based upon the tensile and creep properties measured in laboratory scale heats and industrial scale heats, it was expected that **two alloys** (Two alloys (Haynes®282® and a baseline alloy or other) were to be down-selected for centrifugal casting trials. At least one alloy was expected to satisfy BOTH the tensile and creep properties in Milestone 1.4. Centrifugal cast tubes of diameter 3-4" would be fabricated using the two alloys. Cast pipe manufacturing using selected alloys would be subjected to nondestructive testing (NDT), detailed quality checks and basic metallographic study to prove the manufacturing route was successful for producing a (welded) tube acceptable for high-temperature evaluation. The cast pipes would be subject to relevant sections of "Standard Specification for Seamless Nickel and Nickel-Cobalt Alloy Pipe and Tube." B622-17b © ASTM International, 2017.

Task 2.6 Microstructural characterization centrifugal castings: Microstructure of the alloys were characterized in the as-cast condition to evaluate the effect of typical centrifugal processing conditions on the as-cast microstructure. Optical and scanning electron microscopy along with x-ray microchemical characterization were used to evaluate the

microstructure and interdendritic segregation during solidification. Effect of homogenization annealing treatment on reducing segregation in these alloys were also evaluated.

Task 2.7 Evaluation of tensile properties of centrifugal castings: Tensile properties (uniaxial- yield strength, tensile strength, and elongation to failure) were evaluated at room temperature, 750°C, and 800°C in the solution annealed (in the cast non-age hardenable alloys), and solution annealed + aged condition in the cast age-hardenable alloys. Heat treatments (solution-annealing + aging) were performed based upon previous experiments with laboratory and industrial scale heats. Two to three specimens were tested at each temperature of interest.

Task 2.8 Evaluation of creep properties of centrifugal castings: Isothermal uniaxial creep testing were performed on centrifugally cast material of both down-selected alloys under constant load in the solution annealed or solution annealed + aged condition as appropriate. Testing included short-term, and medium-term (up to 1500 hours) creep testing at temperatures in the range of 700-800°C for down-selected alloys. One test was expected to have failure times exceeding 250 hours, and the second test was expected to exceed 1000 hours of rupture life. Based upon three creep tests one alloy would be down selected for additional creep testing, fatigue testing, and weld process development. Creep rupture life of down-selected alloy should be ≥80% of the lower limit in the scatter band in creep rupture life of wrought alloy Haynes®282® at equivalent stress and temperatures

It was expected that atleast three additional creep tests would be completed on down-selected alloy with one test exceeding 500 hours, one test exceeding 1500 hours, and another test exceeding 4000 hours. Cast down-selected alloy pipe creep-rupture performance was expected to be ≥80% of the wrought properties on stress through fit of a minimum of five data points AND the extrapolation to 10,000 hours at 750°C shall exceed 150MPa.

Task 3.1 Weld process development: Welding processes were developed in collaboration with industrial partners for the final down-selected alloy. Weld feed wire composition was determined based upon computations and prior experience with similar alloys or wrought alloys. Trial welds were performed on centrifugal cast pipe for testing and evaluation after appropriate heat-treatment. Welds had to pass the ASTM flattening and eddy current quality standards for welded tube as defined in ASTM ["Standard Specification for General Requirements for Nickel and Nickel Alloy Welded Pipe." B775-13 © ASTM International, 2013].

Task 3.2 Microstructural characterization of welded specimens: Microstructure of the weld zone and the heat-affected zone were characterized, and microhardness tests were performed to understand hardness variation across the joint.

Task 3.3. Evaluation of cross-weld tensile properties: Tensile properties of the welded joints (cross-welds) were evaluated at room temperature, 750°C, and 800°C in the solution-annealed + aged condition. Tensile property data were compared with literature data from comparable welds in the wrought alloys.

The following table shows the milestones for this project.

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification
1.1	Complete initial laboratory scale casting	Complete laboratory scale casting of up to 4 alloys. 2-4 ingots (~ 1"x 1"x 3-4") and ~ 1 lb heats will be fabricated of each alloy	Compositions of castings	Yes	Compositional analysis to ensure conformance to target composition ranges of the alloys	Alloy compositions of cast alloys must be within specification of target wrought compositions
1.2	Down-select one baseline alloy and two alloys for industrial scale static castings	Complete initial evaluation of tensile properties in triplicates in the solution annealed or solution-annealed + aged condition as appropriate and downselect one baseline alloy and two alloys that meet tensile property downselection criterion	Highest yield strength between 750°C and 800°C	≥80% of the lowest yield strength of the wrought alloy Haynes ®282® between 750°C and 800°C obtained from the literature	students t-test, 95% confidence interval	Strength of cast alloy may be different from that of the wrought alloy due to grain size effects, non-equilibrium segregation and non-equiibrium phase formation.
1.3	Fabricate 3 alloys in industrial scale static castings	Fabricate industrial scale static castings of plates from 3 alloys (one baseline alloy + two down- selected alloys) from M1.2	Composition of castings	Yes	Compositional analysis to ensure conformance to target composition ranges of the alloys	Strength of Alloy compositions of cast alloys must be within specification of target wrought compositions

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification
1.4	Down-select maximum of two alloys for centrifugal casting	Complete tensile tests in triplicates on industrial scale alloys in the solution annealed or solution-annealed + aged condition as appropriate and two uniaxial tensile creep-rupture tests for each alloy one test exceeding creep rupture life of 250 hours and one creep test exceeding life of 500 hours in the solution annealed or solution annealed or solution annealed + aged condition as appropriate, in the range of 700°C - 800°C. Downselect maximum of two alloys for centrifugal casting with at least one alloy satisfying both tensile and creep properties	highest measured yield strength of alloy between 750°C and 800°C	≥80% of the lowest yield strength of the wrought alloy Haynes® 282® between 750°C and 800°C obtained from the literature ≥80% of the lower limit in the scatter band in creep rupture life of wrought alloy Haynes® 282® at equivalent stresses and temperatu	t-test	Alloy properties required to match or exceed performance of current best alloy Alloy properties required to match or exceed performance of current best alloy
2.1	Complete centrifugal casting of two alloys	Complete centrifugal casting of two alloys at the industrial casting partner	Compositions of castings	Yes	Compositional analysis to ensure conformance to target composition ranges of the alloys	Alloy compositions of cast alloys must be within specification of target wrought compositions

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification
2.2	Complete non- destructive testing of centrifugal cast pipes	Manufactured alloy cast pipes will be subject to nondestructive testing (NDT), detailed quality checks, and basic metallographic study to prove the manufacturing route was successful for producing a (welded) tube acceptable for high-temperature evaluation	Quality check	Pass	Relevant sections of ASTM B622-17b	Tubes have to meet quality standards
2.3	Complete tensile testing of centrifugal cast alloys	Complete tensile testing of two centrifugal cast alloys in triplicates. Perform triplicate tests in the solution annealed or solution-annealed + aged condition as appropriate to measure the yield strength of downselected alloys between 750°C and 800°C	Highest yield strength between 750°C and 800°C	≥80% of the lowest yield strength of the wrought alloy Haynes® 282® between 750°C and 800°C obtained from the literature	students t-test, 95% confidence interval	Strength of cast alloy may be different from that of the wrought alloy due to grain size effects, non-equilibrium segregation and non-equilibrium phase formation.
2.4	T2M	T2M for cast high alloy piping as an alternate for mill-run pipe	At least one additional vendor/user willing to consider cast pipe	Yes	Letter from one vendor	Additional application outside of CSP

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification
2.5	Cost Analysis	Cost evaluation for ≥4" pipe manufactured via casting using selected alloy	\$/ft fabricated pipe	≤85% of same alloy- fabricated using seamless pipe	As fabricated cost for at least 2 pipe diameters >10' of pipe	Cost evaluation
2.6	Down-select one promising centrifugal cast alloy	Down-select one promising alloy for further evaluation of creep properties, fatigue properties, and weldability studies based upon tensile properties and creep properties. Creep results will include at	Highest yield strength between 750°C and 800°C	≥80% of the lowest yield strength of the wrought alloy Haynes® 282® between 750°C and 800°C obtained from the literature	students t-test, 95% confidence interval	Strength of cast alloy may be different from that of the wrought alloy due to grain size effects, non-equilibrium segregation and non-equilibrium phase formation.
		2 completed creep-rupture tests with one test resulting in failure times exceeding 250 hours and one test exceeding 1000 hours.	life of down- selected alloy in the range of 700°C - 800°C	≥85% of the lower limit in the scatter band in creep rupture life of wrought alloy Haynes® 282® at equivalent stresses and temperatures	students t-test, 95% confidence interval	Creep strength of cast alloy may be different from that of the wrought alloy due to grain size effects, non-equilibrium segregation and non-equilibrium phase formation.
3.1	Develop initial weld procedures	Develop initial weld procedures for welding tubes of downselected alloy. Weld must pass the ASTM flattening and eddy current quality standards for welded tube as defined in ASTM	ASTM flattening and eddy current quality standards	Pass	ASTM Standard B775-13	Joint must pass weld quality standards.

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification		
3.2	Complete room temperature and high temperature cross-weld tensile testing	Complete room temperature and high temperature cross-weld tensile testing and compare with room temperature and high temperature properties of welded wrought materiall	Cross-weld yield strength	≥85% of the yield strength of equivalent weld in wrought pipe from literature or other data	students t-test, 95% confidence interval	Strength of welds in cast alloy may be different from that of welds in wrought alloy due to grain size effects, non-equilibrium segregation and non-equilibrium phase formation in cast pipes.		
3.3	Complete a minimum of five creep tests on one down-selected alloy	Complete a minimum of five creep tests on one down-selected alloy. One test will have failure times exceeding 250 hours, one test exceeding 500 hours, one test exceeding 1000 hours, one test exceeding 1500 hours of rupture life, and one test exceeding 4000 hours if creep life.	Creep rupture life	≥85% of the wrought properties on stress	students t-test, 95% confidence interval	Creep strength of cast alloy may be different from that of the wrought alloy due to grain size effects, non-equilibrium segregation and non-equilibrium phase formation.		
			Stress for 10,000 hour rupture life at 750°C	≥ 134 Mpa	students t-test, 95% confidence interval	This stress level for 10,000 hour rupture life at 750°C represents 85% of the estimated mean stress level for 10,000 hour rupture life at 750°C for the wrought alloy. Creep strength of cast alloy may be different from that of the wrought alloy due to grain		

Milestone Number	Milestone Title	Description	Metric	Success Value	Assessment Tool	Metric Justification
						size effects, non-equilibrium segregation and non-equilibrium phase formation.

Project Results and Discussion

The first task in this project was related to the laboratory scale casting of initial set of alloys.

Task 1.1. Feedstock procurement: Based upon existing data on wrought alloys, Haynes [®]230[®], Haynes[®]282[®], and IN[®]740H[®] were down selected for initial casting trials. Experimental ORNL alloy 161 and 41M3 were also down selected for laboratory casting trials. To minimize potential effects of compositional variations between laboratory heats when compared to commercial heats and heat-to-heat variations, commercially available rods of Haynes [®]230[®], Haynes [®]282[®], and IN[®]740H[®] were procured for preparation of the laboratory castings. Table 1.1 shows the major elemental constituents of the alloys that were obtained from the certified compositions of the commercial heats.

Table 1.1 Compos	itions of the com-	mercial heats pro	ocured for the project	ct
Table I.I Collipus		mordial modes pri		J

Alloy	Ni	Со	Cr	Fe	W	Mn	Мо	Nb	ΑI	Ti	Si	С
Haynes ®230® (Heat	Bal	2.38	22.14	1.55	14.24	0.53	1.3	0.0	0.39	0	0.37	0.1
830587876) Haynes®282® (Heat 208278368)	Bal	10.18	19.39	0.79	0.06	0.08	8.53	0.0	1.52	2.22	0.06	0.062
IN740H [®] (HT3779J)	50.0 3	20.21	24.51	0.19	0	0.25	0.33	1.45	1.34	1.34	0.14	0.024

Task 1.1 Fabrication of laboratory castings: Laboratory castings of the commercially available alloys were made using the purchased alloy rods as feedstock. The rods were cut into small pieces and were remelted and arc cast into buttons. These buttons were then used to drop cast into \sim 1" x 1" x 3.5" ingots in an inert atmosphere. Figure 1.1 shows an example of a drop-cast ingot of Haynes 282°. A thin slice was cut from the bottom for chemical analysis while another thin slice was cut from the top for microstructural evaluation.



Figure 1.1 Image of a laboratory scale casting of Haynes[®]282[®].

Laboratory scale heats of ORNL alloy 161 and ORNL alloy 41M3 were cast from pure elements. Table 1.2 shows a list of alloys and the number of ingots cast from each alloy. Due to difficulties (splashing of liquid metal) observed during the casting of Haynes 282, casting of one of the four ingots was abandoned. The exact causes for this problem are not clear currently but under investigation. Table 2 shows a summary of the ingots cast as part of this project.

Table 1.2. Summary of ingots cast in Q1 of this project.

Alloy	Number of ingots cast
Haynes ®230®	4
Haynes®282®	3
IN®740H®	4
ORNL Alloy 161	2
ORNL Alloy 41M3	2

Compositional analysis: One slice from one ingot of each of Haynes [®]230[®], Haynes [®]282[®], IN [®]740H [®] and ORNL alloy 161 were sent for wet chemical analysis. Table 1.3 shows a comparison between the certified composition of the alloys and results from the chemical analysis of the ingots. Apart from small variations in alloy compositions, only the composition of Haynes [®]230[®] shows an anomalously large difference in cobalt levels from the feedstock composition. Further investigations will be carried out to understand if this is due to an error in measurement or an actual variation in composition.

Table 1.3. Comparison between feedstock compositions and compositions of samples from ONE ingot of laboratory cast alloy.

Alloy	Ni	Со	Cr	Fe	W	Mn	Мо	Nb	Al	Ti	Si	С
Haynes ®230® -S1	58.89	0.19	21.98	1.37	14.48	0.65	1.33	0.12	0.35	0	0.37	0.1
Haynes [®] 230 [®] (Heat 830587876)	Bal	2.38	22.14	1.55	14.24	0.53	1.3	0.0	0.39	0	0.37	0.1
Haynes [®] 282 [®] - S1	57.52	10.2	19.06	0.77	0.04	0.08	8.25	0.03	1.83	2.07	0.06	0.06
Haynes [®] 282 [®] (Heat 208278368)	Bal	10.18	19.39	0.79	0.06	0.08	8.53	0.0	1.52	2.22	0.06	0.062
IN®740H®-S1	49.32	20.19	24.97	0.2	0	0.29	0.35	1.51	1.58	1.43	0.08	0.02
IN740H [®] (HT3779J)	50.03	20.21	24.51	0.19	0	0.25	0.33	1.45	1.34	1.34	0.14	0.024
ORNL161-S1	46.05	1.05	18.3	27.56	0	0.1	1.22	0.02	1.80	3.8	0	0.04

Task 1.2. As-cast Microstructure: Sections obtained from one casting of each of the alloys Haynes \$230\$, Haynes\$282\$, IN\$740H\$ were prepared from optical metallography and Scanning Electron Microscopy. Figure 1.2 shows an optical image of the as-cast microstructure in Haynes \$230\$. Figure 1.3 is a scanning electron microscope image and an elemental X-ray map of the interdendritic region. The X-ray map suggests precipitation of Cr and Mo rich carbides in the interdendritic region in Haynes \$230\$. Figure 1.4 shows the as-cast microstructure in Haynes \$282\$. The scanning electron microscope image and the elemental X-ray map of the interdendritic region in Figure 1.5 shows the presence of Ti and Mo-rich carbides in this alloy. Figure 1.6 shows the as-cast microstructure in IN\$740H\$. The scanning electron microscope image and the elemental X-ray map of the interdendritic region in Figure 1.7 shows the presence of Ti and Nb-rich carbides in this alloy.

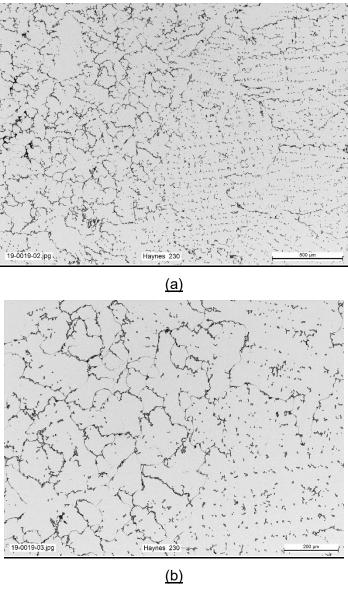
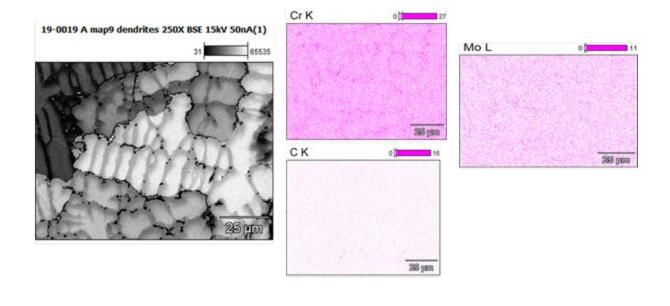


Figure 1.2. (a) Lower magnification and (b) higher magnification optical image from Haynes®230® showing the dendritic structure in the as-cast condition.



(a)

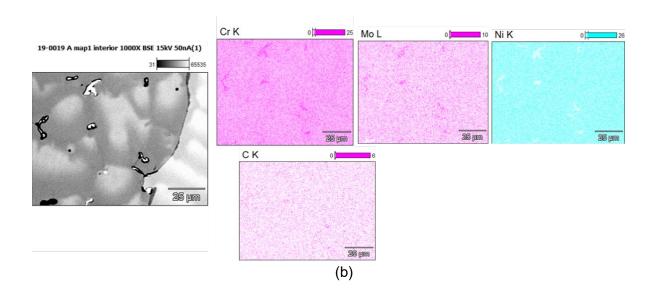


Figure 1.3. (a) Lower magnification and (b) higher magnification scanning electron image and EDX maps from Haynes®230® showing the formation of Cr and Mo-rich interdendritic precipitates.

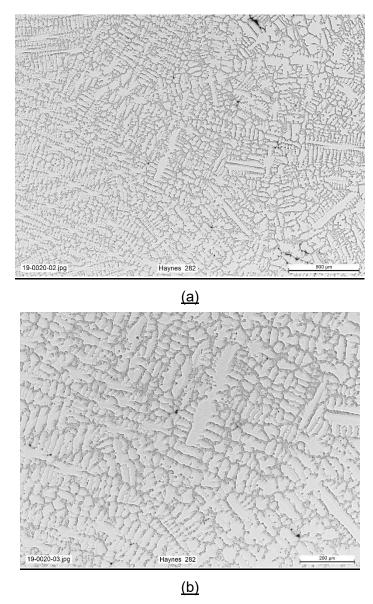


Figure 1.4. (a) Lower magnification and (b) higher magnification optical image from Haynes®282® showing the dendritic structure in the as-cast condition.

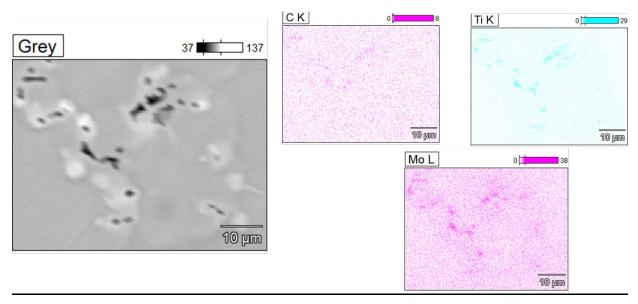


Figure 1.5. Scanning Electron Microscopy and elemental X-ray mapping shows the presence of Ti and Mo rich precipitates in the interdendritic region in Haynes[®]28[®]2 in the as-cast condition

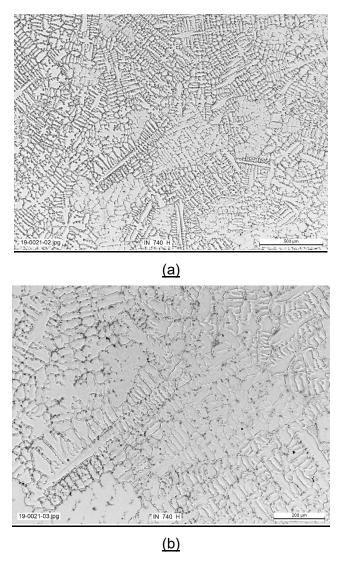


Figure 1.6. (a) Lower magnification and (b) higher magnification optical image from $IN^{8}740H^{8}$ showing the dendritic structure in the as-cast condition.

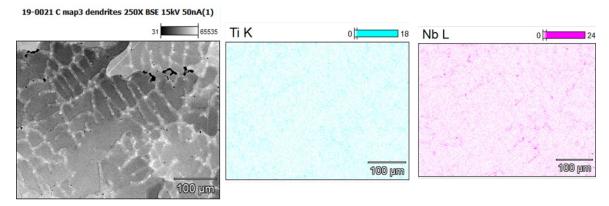


Figure 1.7. (a) Scanning Electron Microscopy and X-ray Elemental Mapping Shows the Presence of Ti and Nb rich carbides in the interdendritic region in IN®740H®.

Figure 1.8 (a), (b) and (c) show a comparison of higher magnification optical images from Haynes®230®, Haynes®282®, IN®740H®, respectively. Note that the as-cast microstructure, non-equilibrium phase precipitation, and segregation varies with the alloy composition. Hence a customized homogenization/ solution anneal or a homogenization/solution anneal/aging treatment was used for each laboratory scale alloy as shown in Table 1.4.

Table 1.4. Summary of initial homogenization, solution-annealing, and aging treatments used In this study.

Alloy	Heat-treatment
Haynes [®] 230 [®]	Homogenization: Ramp to 1093°C, hold for 8 hours, increase temperature to 1204°C, hold for 24 hours Solution anneal: 1177°C for 0.5 hour, followed by brine quench
Haynes [®] 282 [®]	Homogenization: Ramp to 1093°C, hold for 8 hours, increase temperature to 1204°C, hold for 24 hours Solution anneal: 1121°C for 1 hours, water quench Aging: 1010°C for 2 hours in inert atmosphere, air cool 788°C for 8 hours, air cool
IN®740H®	Homogenization: Ramp to 1093°C, hold for 8 hours, increase temperature to 1204°C, hold for 24 hours Solution anneal: 1121°C for 1 hours, water quench Aging: 800°C for 5 hours in inert atmosphere and air cool

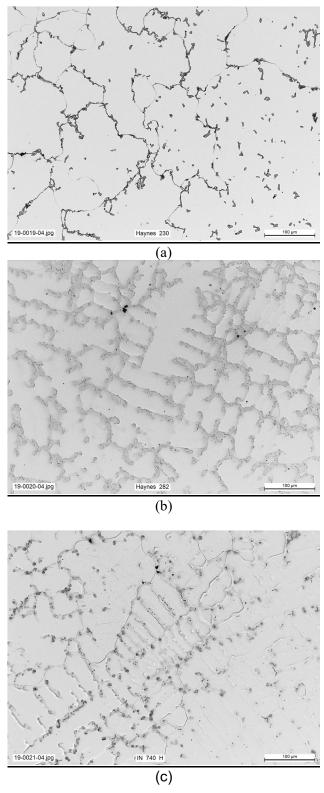


Figure 1.8. Higher magnification optical image showing the presence of interdendritic precipitates in (a) Haynes $^8230^8$, (b) Haynes $^8282^8$, and (c) IN $^8740H^8$.

Task 1.2. Post-Heat treatment Microstructure

Figure 1.9 shows the effect of homogenization and solution annealing treatment on the microstructures of cast Haynes [®]230[®], while figures 1.10 and 1.11 show the effect of homogenization and solution annealing treatment followed by dual step aging process on the microstructures of Haynes[®]282[®], and IN[®]740H[®] respectively. Haynes[®]230[®] shows the presence of interdendritic precipitation (likely primary carbides) even after the completion of heattreatments. This is related to the fact that Haynes®230® has the highest levels of carbon (0.1 wt. % C) amongst the three alloys (See Table 1.1). This is also evident from figure 1.9(a) which shows a higher magnification image of Haynes[®]230[®]. Figures 1.10 and 1.11 show the complete elimination of the dendritic structure found in the as-cast microstructure in Haynes[®]282[®], and IN®740H®. Some primary carbides are observed to be present in Haynes®282® are consistent with its carbon content (0.06 wt. %). It can also be observed that the grain size is relatively large due to the processing and heat-treatment steps - casting followed by the high temperature homogenization and solution annealing. IN®740H® shows the smallest amount of carbides and a very large grain size due to the lowest carbon levels (0.02 wt. % carbon). This is also probably responsible for the very large grain size observed in figures 1.11 and 1.12. A modified solution annealing treatment will be required to achieve a finer grain size.

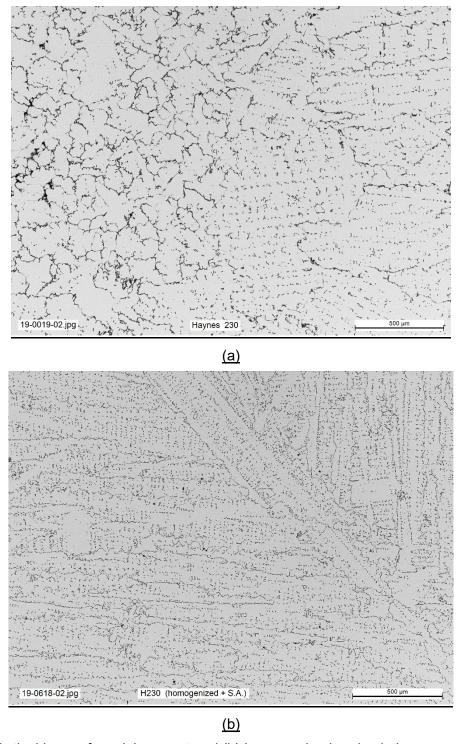


Figure 1.9. Optical image from (a) as-cast and (b) homogenized and solution-annealed Haynes®230® showing the effect of heat-treatment on the as-cast microstructure.

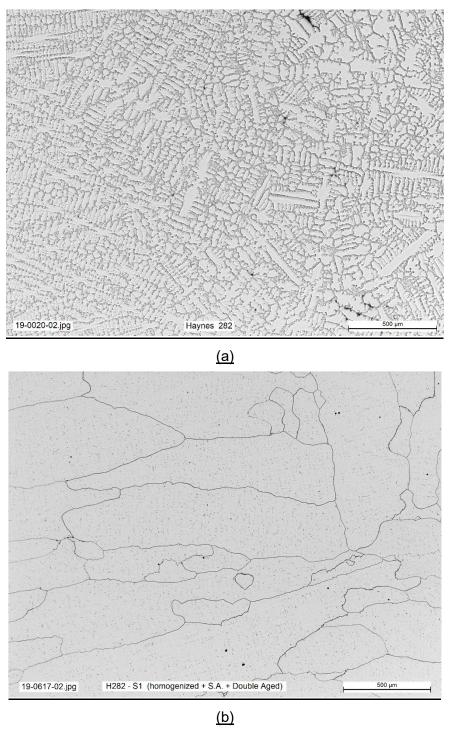


Figure 1.10. Optical image from (a) as-cast and (b) homogenized, solution-annealed, and double aged Haynes[®]282[®] showing the effect of heat-treatment on the as-cast microstructure.

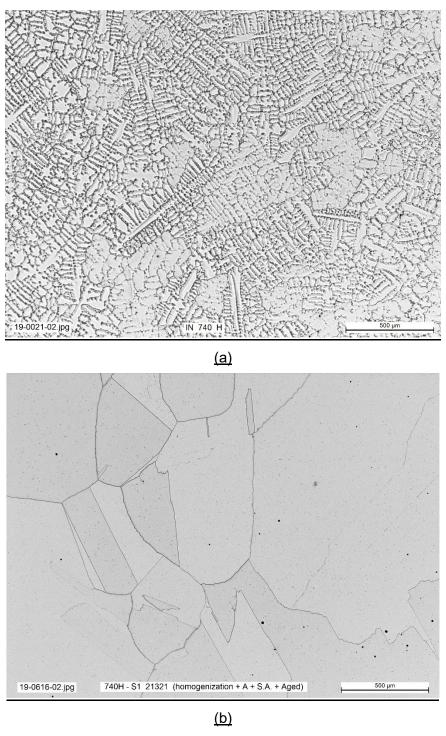


Figure 1.11. Optical image from (a) as-cast and (b) homogenized, solution-annealed, and double aged $IN^{\circ}740H^{\circ}$ showing the effect of heat-treatment on the as-cast microstructure.

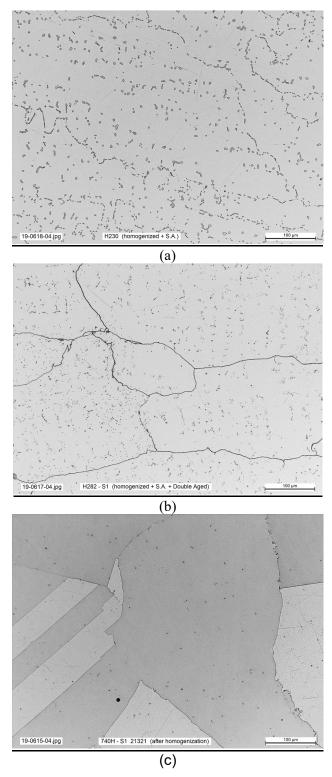
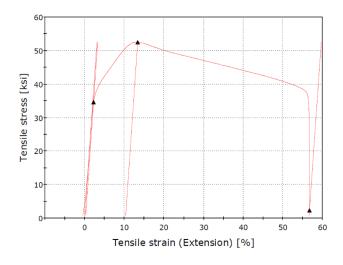
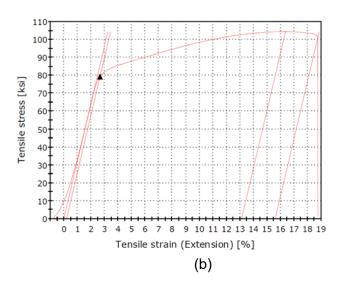


Figure 1.12. Higher magnification optical image showing the presence of interdendritic precipitates in (a) Haynes[®]230[®], (b) Haynes[®]282[®], and to a much lesser extent in (c) IN[®]740H[®].

Task 1.3. Tensile property screening: Tensile tests were conducted using sub-sized round specimens with a dimension of ¼" at the grips and 0.16" in the gage section. These dimensions were used due to the limited sized material available. It is anticipated that the large grain size may affect the ductilities achieved during the tests and hence the results must only be used for screening purposes. Two tests were conducted at room temperature while three tests were conducted each at 750°C and 800°C. Additional tests will be performed with larger sized heats obtained in industrial scale castings. Figure 1.13 shows typical tensile curves from (a) Haynes®230®, (b) Haynes®282®, and (c) IN®740H® obtained at 750°C. Note that the yield strength of Haynes®230® is lower than that of Haynes®282® and IN®740H®.



(a)



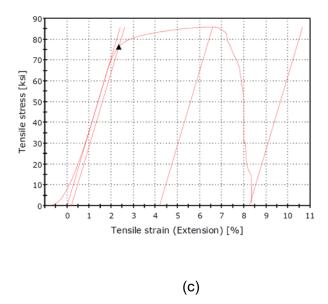


Figure 1.13. Typical tensile curves at 750°C from (a) Haynes[®]230[®], (b) Haynes[®]282[®], and (c) IN[®]740H[®].

Figure 1.14 shows the yield strengths of cast and heat-treated laboratory scale Haynes[®]282[®] as a function of temperature (blue). Also shown in orange for comparison is the yield strength of wrought Haynes[®]282[®] as a function of temperature. As can be observed in the figure, the yield strengths of the cast and heat-treated Haynes[®]282[®] is lower than that of wrought Haynes[®]282[®] [1]. Figure 1.15 shows an equivalent plot for IN[®]740H[®].

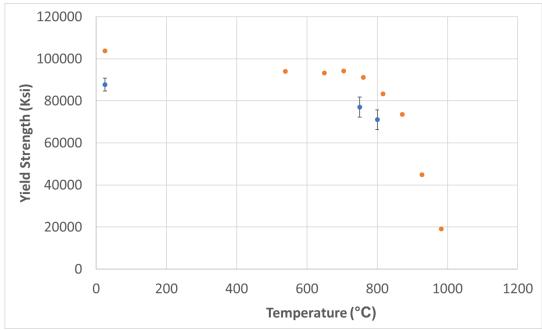


Figure 1.14. Yield Strengths of cast and heat-treated laboratory scale Haynes[®]282[®] as a function of temperature (blue) and wrought Haynes[®]282[®] (orange).

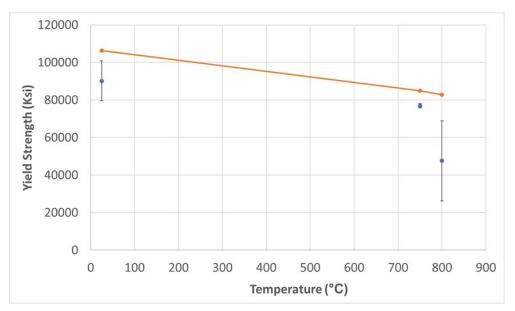


Figure 1.15. Yield Strengths of cast and heat-treated laboratory scale IN®740H® as a function of temperature (blue) and wrought IN®740H® [2].

Table 1.5 shows a summary of yield strength data obtained from the cast and heat-treated alloys and wrought Haynes[®]282[®].

Both Haynes[®]282[®] and IN[®]740H satisfy the milestone criterion for M1.2:

Highest yield strength between 750°C and 800°C ≥80% of the lowest yield strength of the wrought alloy Haynes®282® between 750°C and 800°C obtained from the literature.

Based upon the satisfactory completion of milestone of M1.2, industrial scale castings of both Haynes[®]282[®] and IN[®]740H[®] were now targeted along with the industrial scale casting of Haynes[®]230[®] as the baseline alloy

Table 1.5. Summary of yield strength data obtained from laboratory-scale cast + heat-treated alloys. 95% confidence interval is shown

Alloy	Y. S. at 750°C (Ksi)	Y. S. at 800°C (Ksi)			
Wrought Haynes®282®	91.7	85.6			
80% of Yield strength of wrought Haynes®282®	73.3	68.5			
Cast + Heat-treated Haynes [®] 230 [®]	33.5 ± 3.6	36 ± 5.5			
Cast + Heat-treated Haynes [®] 282 [®]	76.9 ± 4.8	71±4.7			
Cast + Heat-treated IN®740H®	76.9 ±1.1	59.7±21.2			

Task 2.1. Fabrication of static industrial scale castings: Based upon the milestone criterion satisfied as explained above, it was determined that Haynes®282® and IN®740H® along with baseline alloy Haynes®230® would be cast in industrial cast heats. Figure 2.1 shows typical investment casting of plates and test specimens of a heat of an alloy cast at MetalTek International. Figure 2.2 shows the image of Haynes ®282® plate cast at MetalTek and shipped to ORNL. IN®740H® could not be cast at MetalTek yet since a licensing arrangement between Special Metals and MetalTek to cast this alloy has not established. Table 2.1 shows the composition of the industrial scale heat of Haynes ®282® plate. One plate of Haynes®282® was subject to the heat-treatment shown in Table 3.



Figure 2.1. Investment casting of plates and test specimens.



Figure 2.2 Image of a cast Haynes [®]282[®] plate received at ORNL.

Table 2.1. Comparison between feedstock compositions and compositions of samples from laboratory cast alloy and MetalTek cast alloy.

Alloy	Ni	Co	Cr	Fe	W	Mn	Мо	Nb	Al	Ti	Si	С
Haynes [®] 230 [®] -S1	58.89	0.19	21.98	1.37	14.48	0.65	1.33	0.12	0.35	0	0.37	0.1
Haynes [®] 282 [®] - S1	57.52	10.2	19.06	0.77	0.04	0.08	8.25	0.03	1.83	2.07	0.06	0.06
MetalTek Haynes®282®	58.8	9.29	19.0	0.24	0.4	0.02	8.21	0.28	1.54	1.96	0.06	0.036
IN®740H®-S1	49.32	20.19	24.97	0.2	0	0.29	0.35	1.51	1.58	1.43	0.08	0.02

Task 2.2 Microstructural characterization static industrial scale castings. Figure 2.3 shows a typical micrograph from the as-cast Haynes[®]282[®] plate while Figure 2.4 shows a micrograph from the homogenized + aged condition.

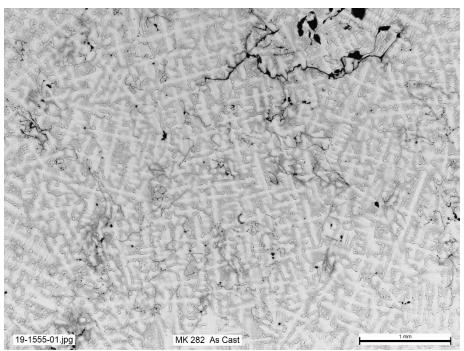


Figure 2.3. Optical micrograph from as-cast Haynes®282® plate.

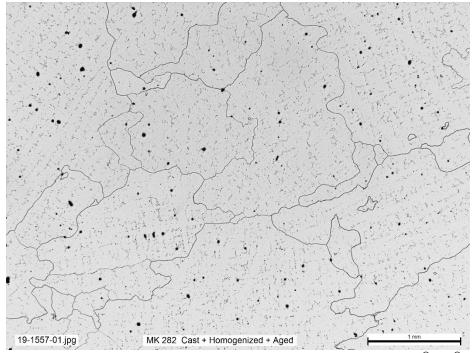


Figure 2.4. Optical micrograph from homogenized and aged Haynes[®]282[®] plate.

Figure 2.5 (a) shows a low magnification image of the as-cast microstructure of industrial scale casting of Haynes[®]230[®] while 2.5 (b) shows a higher magnification image. Note the coarse dendritic microstructure of the as-cast alloy. Figure 2.6 (a) and (b) shows the effect of heat-treatment on the microstructure of the alloy. Note again the coarse grain size obtained after the homogenized and solution annealed condition.

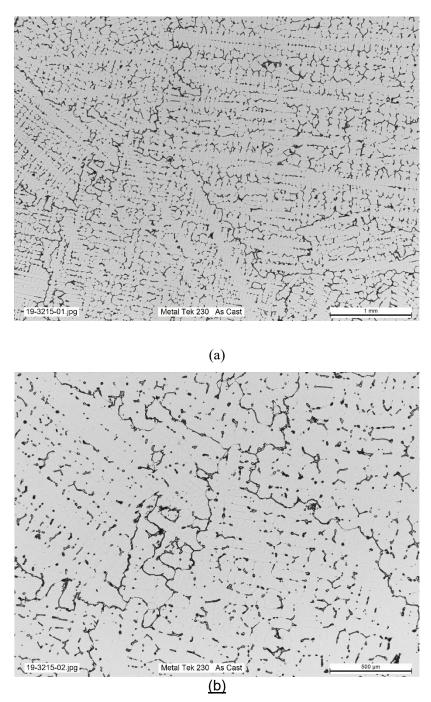


Figure 2.5. (a) Lower Magnification and (b) Higher Magnification Optical image from as-cast industrial scale Haynes $^{\!8}230^{\!8}.$

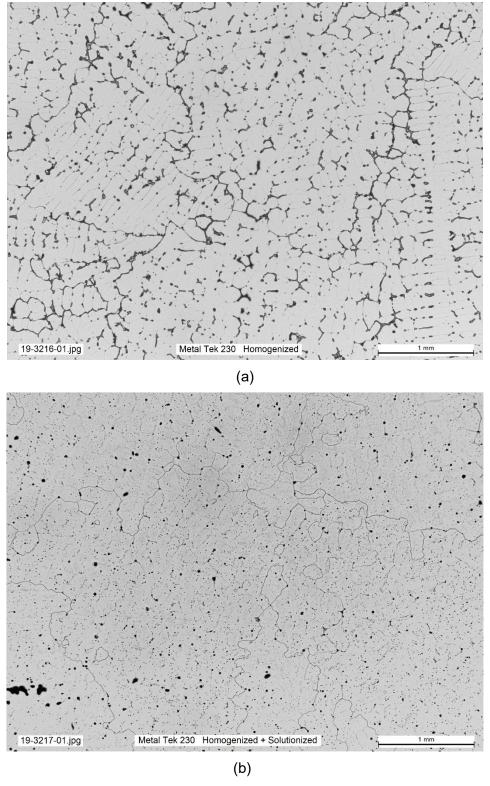


Figure 2.6. Optical Image from (a) Homogenized (b) Homogenized and Solution Annealed industrial scale Haynes $^{\circ}230^{\circ}$.

Task 2.3. Evaluation of tensile properties of static industrial scale castings: Table 2.3 shows a quantitative comparison of the yield strengths obtained the various alloys heat-treated according to the conditions summarized in Table 2.2. It should be noted that the laboratory cast Haynes®282® and MetalTek Haynes®282® – HT3 meet the tensile strength milestone criteria, while MetalTek Haynes®282® – HT1 did not meet the milestone. Hence an alternate heat-treatment schedule denoted Haynes®282® – HT3 was developed which met the milestone criterion. It is important to understand the effect of heat-treatment on the properties so that the proper heat-treatment can be specified in industrial practice. Cast + Heat-treated industrial scale Haynes®230® shows slightly lower yield properties when compared to laboratory scale cast and heat-treated Haynes®230®.

Table 2.2 Summary of homogenization, solution-annealing, and aging treatments used in this study.

Alloy	<u>Heat-treatment</u>
Haynes [®] 230 [®]	Homogenization: Ramp to 1093°C, hold for 8 hours, increase
	temperature to 1204°C, hold for 24 hours
	Solution anneal: 1177°C for 0.5 hour, followed by brine
	quench
Haynes [®] 282 [®] - HT1	Homogenization : Ramp to 1093°C, hold for 8 hours, increase
	temperature to 1204°C, hold for 24 hours
	Solution anneal: 1121°C for 1 hours, water quench
	Agin g: 1010°C for 2 hours in inert atmosphere, air cool
	788°C for 8 hours, air cool
Haynes [®] 282 [®] - HT3	Homogenization : Ramp to 1093°C, hold for 8 hours, increase
	temperature to 1204°C, hold for 24 hours
	Solution anneal: 1149°C for 1 hours, water quench
	Agin g: 1010°C for 2 hours in inert atmosphere, air cool
	788°C for 8 hours, air cool
IN®740H®	Homogenization : Ramp to 1093°C, hold for 8 hours, increase
	temperature to 1204°C, hold for 24 hours
	Solution anneal: 1121°C for 1 hours, water quench
	Aging: 800°C for 5 hours in inert atmosphere and air cool

Table 2.3. Summary of yield strength data obtained from cast + heat-treated alloys. 95% confidence interval is shown

Alloy	Y. S. at 750°C (Ksi)	Y. S. at 800°C (Ksi)
Wrought Haynes [®] 282 [®]	91.7	85.6
80% of Yield strength of wrought Haynes®282®	73.3	68.5
Cast + Heat-treated laboratory scale Haynes®230®	33.5 ± 3.6	36 ± 5.5
Cast + Heat-treated industrial scale Haynes®230® (MetalTek cast Cast Haynes®230® Plates)	27.9 ± 0.8	29.5 ± 0.7
Cast + Heat-treated IN®740H®	76.9 ±1.1	59.7±21.2*
Cast + Heat-treated laboratory scale Haynes®282®	76.9 ± 4.8	71±4.7
Cast + Heat-treated laboratory scale Haynes [®] 282 [®] (HT-1)	63.3 ± 5.2	61.1±1,4
Cast + Heat-treated industrial scale Haynes®282® (HT-3)	70.7 ± 5.8	68.6±2.9

Industrial-scale casting of Haynes[®]282[®] subject to HT-3 satisfied the milestone criterion for M1.4 and hence was down-selected for centrifugal casting along with the baseline alloy Haynes[®]230[®].

Highest yield strength between 750°C and 800°C ≥80% of the lowest yield strength of the wrought alloy Haynes®282® between 750°C and 800°C obtained from the literature.

Task 2.4. Evaluation of creep properties of laboratory scale and industrial scale static castings:

Figure 2.7 shows a comparison of the Larsen Miller Parameter (LMP) plot comparing the creep properties of wrought Haynes[®]230[®] with that of laboratory scale cast Haynes[®]230[®] and industrial scale casting of MetalTek Haynes[®]230[®]. Note that the creep properties of cast Haynes[®]230[®] seem to compare well with that of the wrought product.

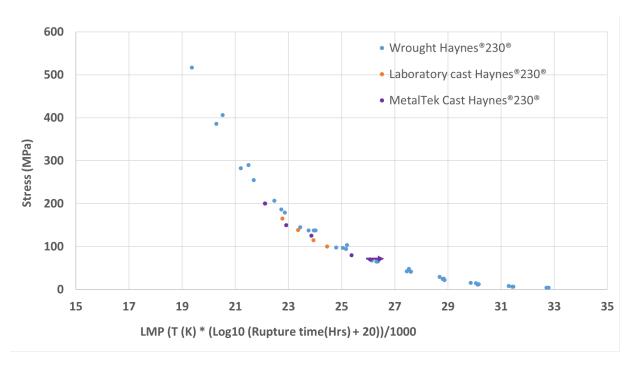


Figure 2.7. Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes®230® [4] with cast and heat-treated laboratory scale Haynes®230® and cast and heat-treated industrial scale Haynes®230® (MetalTek Haynes®230®). Arrow indicates that the test is still running.

Figure 2.8 shows the creep strain as a function of hours for MetalTek Haynes[®]230[®] shown as a test still running in Figure 2.7. At the time of this report, the accumulated creep rupture life for this test is greater than 17544 hours.

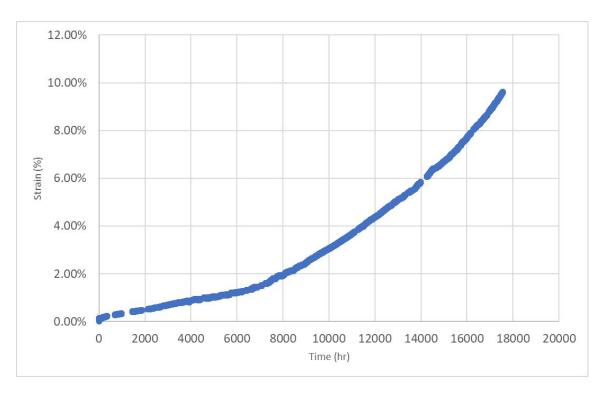


Figure 2.8. Elongation vs time (hours) for Metaltek Cast Haynes®230® at 800°C, 70MPa stress with accumulated creep life of 17544 hours.

Figure 2.9 shows a Larsen Miller Parameter (LMP) summary of the completed and on-going creep tests of the following alloys conducted at 750°C to - 800°C at the end of the project.

- 1. Cast + Heat-treated laboratory scale Haynes[®]230[®]
- 2. Cast + Heat-treated laboratory scale IN®740H® (3 tests)
- 3. Cast + Heat-treated industrial scale laboratory scale Haynes®282® (HT-1, HT-3)

From the LMP it should be noted that:

- 1. Creep properties of the cast IN®740H® and cast Haynes®282® fits within the scatter band of the creep properties of wrought Haynes®282® in the LMP plot satisfying the creep property requirements as per Milestone M1.4.
- 2. It should also be noted that the creep properties of cast Haynes[®]230[®] are clearly lower than that of cast IN[®]740H[®] and Haynes[®]282[®].
- 3. 100 MPa, 750°C rupture life of cast Haynes®282® is at least equal to or better than that of wrought Haynes®282®.

Figure 2.10 shows a creep curve obtained from industrial scale cast Haynes[®]282[®] where the rupture life at 800°C, 150 MPa resulted in the LMP parameter being larger than that for wrought Haynes[®]282[®] at 150 MPa (see Figure 2.9). Hence centrifugal castings of Haynes[®]282[®] were fabricated in this project.

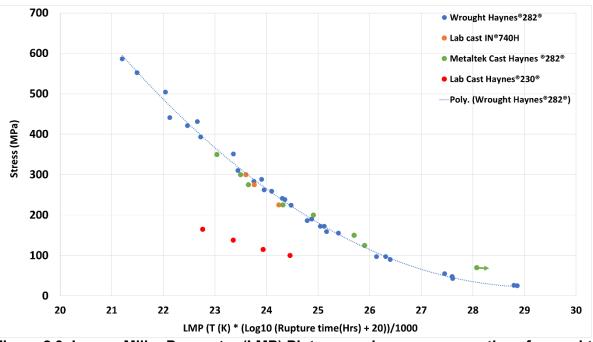


Figure 2.9. Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes®282 [3] with cast and heat-treated laboratory scale IN®740H and cast and heat-treated MetalTek Cast Haynes ®282® - HT1 and Laboratory Cast Haynes ®230®.

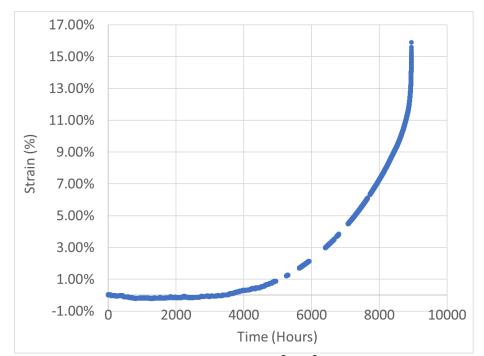


Figure 2.10. Creep curve from MetalTek Cast Haynes $^{\circ}282^{\circ}$ - HT1 at 800 $^{\circ}$ C, 150 MPa. Rupture life was ~8947 hours. The LMP is greater than that of wrought Haynes $^{\circ}282^{\circ}$ in Figure 2.9.

Task 2.5 Fabrication of centrifugal castings:

Centrifugal casting of Haynes®230®: Figure 2.11 (a) shows the centrifugal casting of Haynes®230® received at ORNL and Figure 2.11 (b) shows the cross-section of the centrifugal cast tube.



(a)

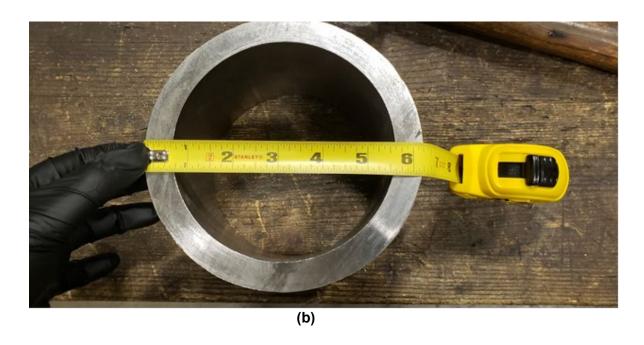


Figure 2.11 (a) Centrifugal casting of Haynes®230®. (b) Cross-section of tube shown in (a).

Non-Destructive Examination of Haynes®230® Centrifugal Cast arc:

The first examination performed on the Haynes [®]230[®] material was radiography. The material is nickel-based so a monel IQI was included to show adequate film sensitivity. The middle area is the material itself. The sides were masked with lead to lessen the burn out on the edges. Figures 2.12(a) and 2.12 (b) show x-ray radiography images from two edges of a section of a cylindrical tube. No obvious voids or gross discontinuities were seen in this examination.

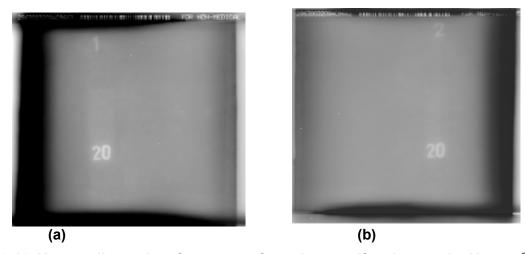


Figure 2.12. X-ray radiography of an arc cut from the centrifugal cast tube Haynes[®]230[®] shown in Figure 3. Conditions: 280kV, 15mA, 3.5min.

A liquid dye penetrant exam on the smooth surfaces was performed to see if there were any blemishes on the surface that were deep and surface-breaking (Figure 2.13 (a) and 2.13 (b). The edges were clear, but the inside of the arc had many small indications, as shown below. These seem to be small imperfections on the inside surface of the tube that are left behind after pull-boring.

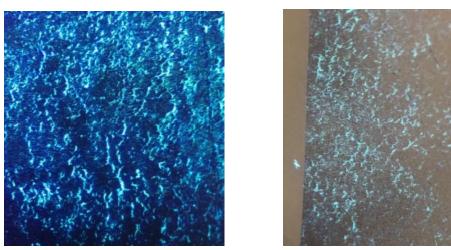


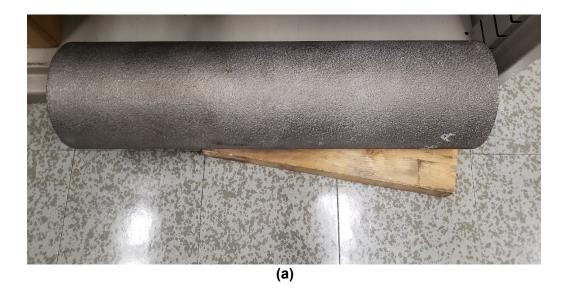
Figure 2.13. (a), and (b). Different views of the inside arc surface, under blacklight and white light conditions

Centrifugal casting of Haynes[®]282: Figure 2.14 shows a centrifugal casting of Haynes [®]282[®]...



Figure 2.14. Centrifugal casting of Haynes[®]282[®]

Figure 2.15 (a) shows a short section of the centrifugal casting and 10 (b) shows a cross-section. The diameter of this pipe is 6" as shown in the figure.



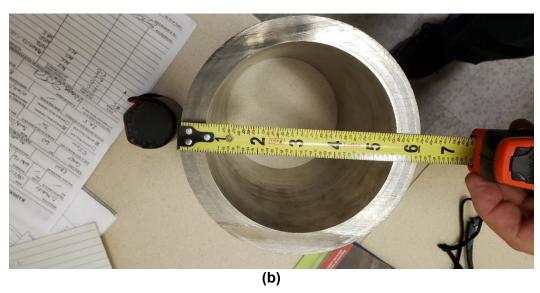


Figure 2.15 (a) A short section of the centrifugal casting and cross-section of the tube shown in figure 2.15 (a).

Since non-destructive testing of MetalTek Haynes[®]230[®] pipe provided very little evidence of voiding or other defects, non-destructive testing of MetalTek cast Haynes[®]282[®] pipe was not performed.

Task 2.6 Microstructural characterization centrifugal castings.

Figure 2.16 shows the presence of Si, Cr, Mo, W rich regions in the as-cast structure.

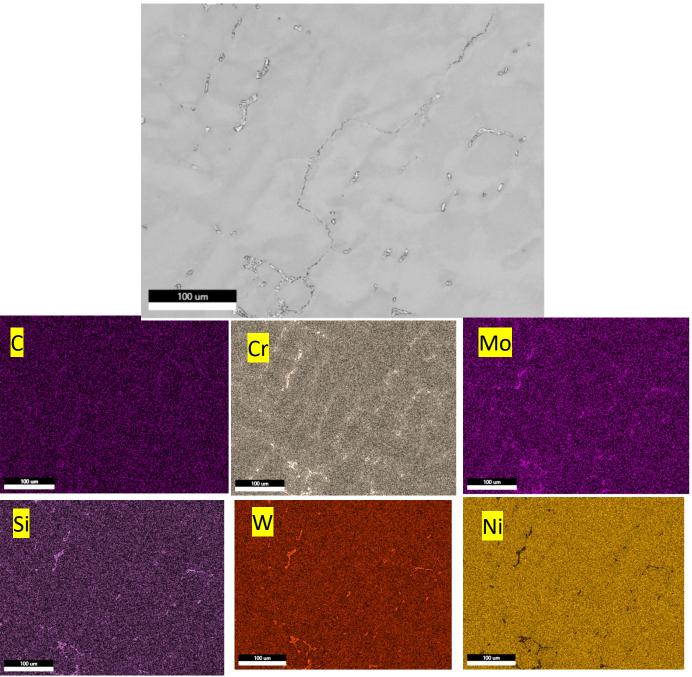


Figure 2.16 (a) SEM image and EDX Maps Showing Cr, Mo, Si, W, rich regions in the ascast structure.

Figure 2.17 (a) and (b) show the microstructure of centrifugally cast Haynes[®]230[®] after homogenization and solution annealing following Table 2.

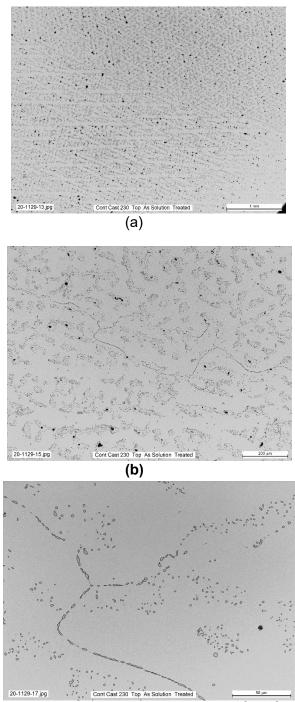


Figure 2.17 (a) Microstructure of centrifugally cast Haynes®230® after homogenization and solution annealing.

Figure 2.18 shows the microstructure of as-centrifugally cast Haynes[®]282[®]. Interdendritic segregation can be clearly observed. Figure 2.19 shows the effect of homogenization, solution annealing and aging on the microstructure. Note that the grain size is > 1 mm.

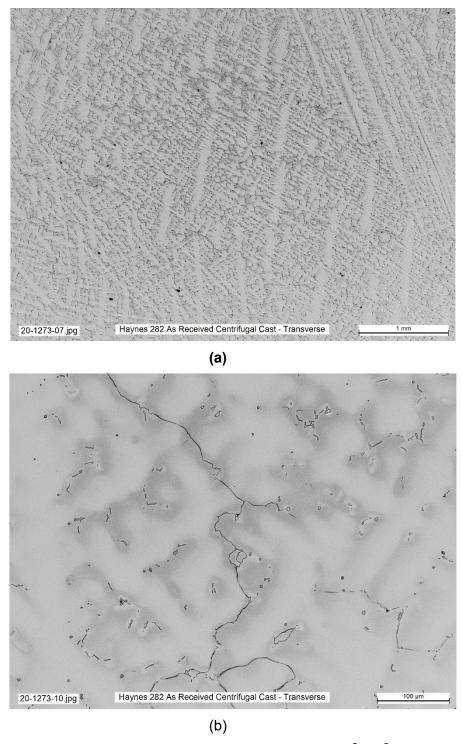


Figure 2.18 (a) Microstructure of as- centrifugally cast Haynes®282®

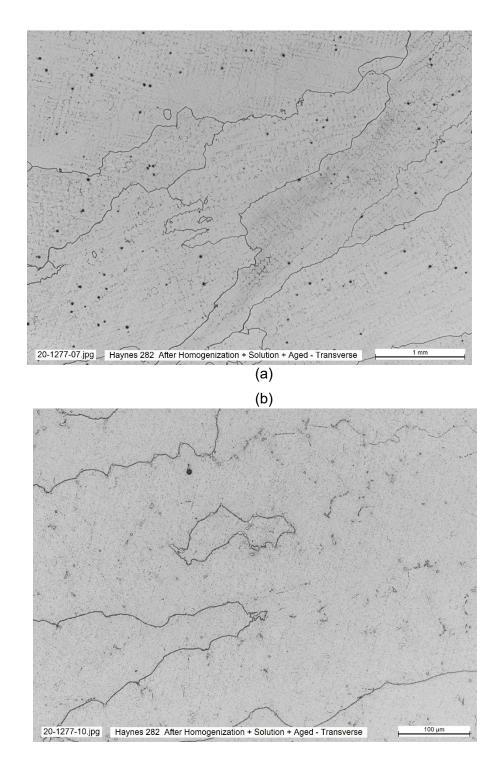


Figure 2.19 (a) Microstructure of homogenized, solutioned + aged centrifugally cast Haynes®282®.

Task 2.7 Evaluation of tensile properties of centrifugal castings: Table 2.4 shows a comparison of the yield strengths of wrought Haynes[®]282[®] at 750°C and 800°C, target value of 80% of this yield strength, yield strengths of laboratory cast, investment cast, and centrifugally cast Haynes[®]230[®] and Haynes[®]282[®].

- 1. Yield strengths of centrifugally cast Haynes[®]230[®] seem to be comparable to the laboratory cast Haynes[®]230[®] which was cast into a copper mold.
- 2. Yield strengths of centrifugally cast Haynes[®]282[®] is good in the as-cast condition but can be improved by heat-treatment.
- **3.** Yield strengths of centrifugally cast Haynes®282®, homogenized, solutioned and aged satisfy the yield strength requirement (> 80% of wrought) and thus satisfy the milestone requirement.

Table 2.4. Summary of yield strength data obtained from cast + heat-treated alloys. 95% confidence interval is shown

Alloy	Y. S. at 750°C (Ksi)	Y. S. at 800°C (Ksi)
Wrought Haynes®282®	91.7	85.6
80% of Yield strength of wrought Haynes®282®	73.3	68.5
Laboratory Cast + Heat- treated Haynes®230®	33.5 ± 3.6	36 ± 5.5
Cast + Heat-treated industrial scale Haynes®230 ® (MetalTek cast Haynes®230® Plates)	27.9 ± 0.8	29.5 ± 0.7
Centrifugal Cast + Heat- treated Haynes [®] 230 [®]	29.1 ± 2.2	36.4.5 ± 6.6
Cast + Heat-treated laboratory scale Haynes®282®	76.9 ± 4.8	71±4.7
Cast + Heat-treated industrial scale Haynes®282® (HT-3)	70.7 ± 5.8	68.6±2.9
Centrifugal As-cast Haynes®282®	68.0 ± 1.1	63.0±2.1
Centrifugal Cast + Heat- treated industrial scale Haynes®282® (HT-3)	75.8 ± 11.6	72.7±18.5
(meets down-select criterion)		

Task 2.8 Evaluation of creep properties of centrifugal castings:

Figure 2.20 is a Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes®230® [4] with cast and heat-treated laboratory scale Haynes®230®, cast and heat-treated industrial scale Haynes®230®, and cast and heat-treated centrifugal cast Haynes®230®. Arrow indicates that the tests are still running. Results show that the creep life of heat-treated centrifugally cast Haynes®230® is comparable to that of wrought Haynes®230®, particularly at the lower stress levels. Further experiments are required to compare behavior at high stresses and longer-term behavior at lower stresses than those shown in the chart.

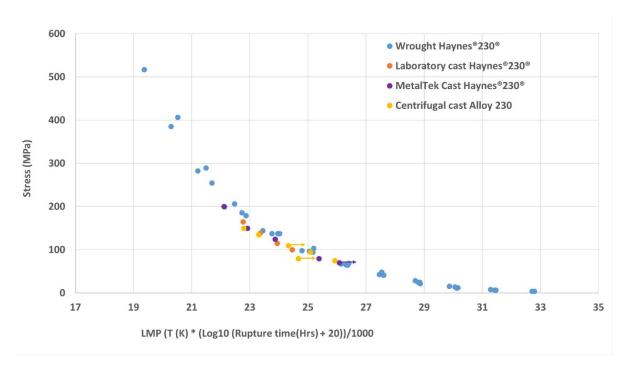


Figure 2.20. Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes®230® [4] with cast and heat-treated laboratory scale Haynes®230® and cast and heat-treated industrial scale Haynes®230®, and cast and heat-treated centrifugal cast Haynes®230®. Arrow indicates that the test is still running.

Figure 2.21 shows a creep curve obtained from centrifugally cast and heat-treated Haynes[®]230[®] at 800°C and 75 MPa with a rupture life of 14591 hours.

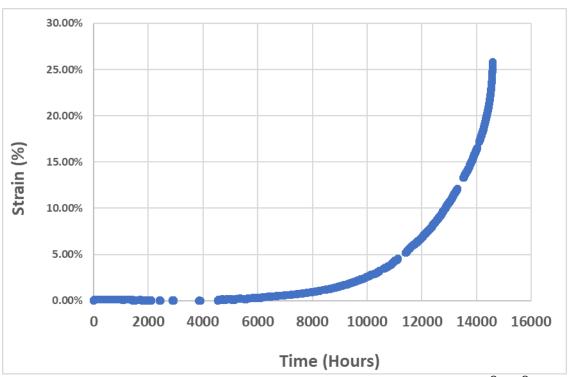


Figure 2.21. Creep strain (%) vs time curve for centrifugal cast Haynes[®]230[®] at 800°C, 75 MPa. Sample failed after 14591 hours.

Figure 2.22 is a Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes®282® [1] with cast and heat-treated industrial scale Haynes®282® (HT1 and HT3) and cast centrifugal cast and heat-treated (HT3) Haynes®282®. Results show that except at a relatively high stress levels (> 200 MPa), the cast and heat-treated Haynes®282® has creep properties comparable to or better than that of wrought Haynes®282®. Since the yield strengths of the cast and heat-treated alloy were already found to satisfy the milestone criteria, it is clear that cast and heat-treated Haynes®282® satisfies the strength and creep property required by milestone 2.6. In addition, Figure 2.22 shows LMP for 10,000 hour rupture life at 750°C along with the line representing 134 MPa. Clearly, the LMP plot shows that cast and HT3 treated Haynes®282® has 10,000 rupture life at 750°C at a stress greater than 134 MPa satisfying conditions for Milestone 3.3. Table 2.4 shows a summary of the creep rupture lives obtained in this study.

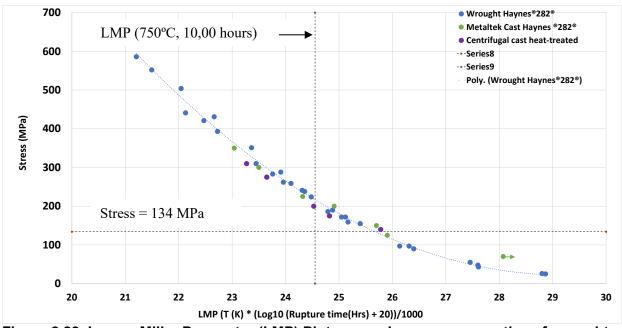


Figure 2.22. Larsen Miller Parameter (LMP) Plot comparing creep properties of wrought Haynes 282 [1] with cast and heat-treated MetalTek Cast Haynes 282 - HT1, HT3 and centrifugal cast Haynes 282 - HT3. The line representing LMP for 10,000 hour rupture life at 750 C is shown along with the line representing 134 MPa.

Table 2.4 Summary of creep tests completed in the study. All materials tested in the heat-treated condition.

Material	Temperature (°C)	Stress (MPa)	Rupture Life (h)
Investment Cast Haynes®230®	700	200	526
	750	125	2131
	750	150	250
	800	80	4487
	800	70	20017
Centrifugal Cast	700	150	2600
Haynes [®] 230 [®]	750	135	597
	800	95	2404
	800	75	14591
Investment Cast Haynes®282®	750	275	1310
	750	300	937
	800	125	13916
	800	150	8947
	800	200	1644
	800	225	465
	800	350	30
Centrifugal cast Haynes [®] 282 [®]	750	175	2420
	750	275	1303
	750	310	556
	800	200	720
	800	175	1356
	800	140	13143

Task 3.1. Weld Process Development for Haynes®282: Figure 3.1 shows a centrifugal casting of Haynes ®282®. Figure 3.2 shows plates that have already been machined for welding process development. Solution treatment was completed according to the heat-treatment schedule shown in Table 3.1 and designated as Haynes®282- HT3.



Figure 3.1. Centrifugal casting of Haynes®282®.



Figure 3.2. Heat-treated plates of Haynes®282® for welding trials (designated as #1 and #2).

Table 3.1. Summary of homogenization, solution-annealing, and aging treatments used In this study.

Alloy	<u>Heat-treatment</u>
Haynes [®] 282 [®] - HT1	Homogenization: Ramp to 1093°C, hold for 8 hours, increase temperature to 1204°C, hold for 24 hours Solution anneal: 1121°C for 1 hours, water quench Aging: 1010°C for 2 hours in inert atmosphere, air cool 788°C for 8 hours, air cool
Haynes [®] 282 [®] - HT3	Homogenization: Ramp to 1093°C, hold for 8 hours, increase temperature to 1204°C, hold for 24 hours Solution anneal: 1149°C for 1 hours, water quench Aging: 1010°C for 2 hours in inert atmosphere, air cool 788°C for 8 hours, air cool
Haynes [®] 282 [®] - HT4	Homogenization/Solution anneal: Vacuum Solution Heat-treated at 1148.92°C (2100°F) / 90 minutes / Ar gas quench Aging: 1010°C (1850°F) for 2 HR, air cooled to room temperature, followed by 787.8°C (1450°F) for 8 hour, air cool to room temperature

The following conditions were used for welding the plates:

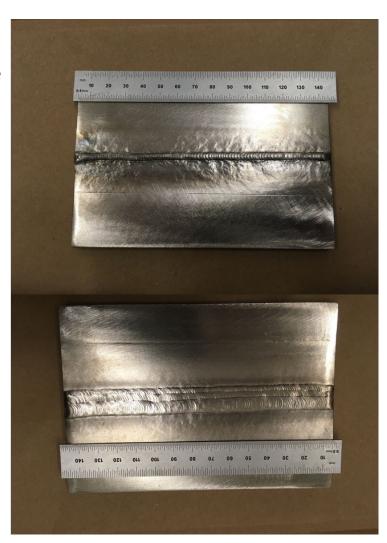
- Plates 1 and 2 (from centrifugal cast pipe)
 - ~ 8.5 x 50 x 100 mm

 - Followed Haynes[®] 282[®] Welding Guide
 V groove weld joint (75 degree included)
 - 11 weld passes
 - Haynes[®] 282[®] weld wire Manual GTAW

 - Preheat: Room Temperature
 - Interpass Temperature: < 93 C (200 F)

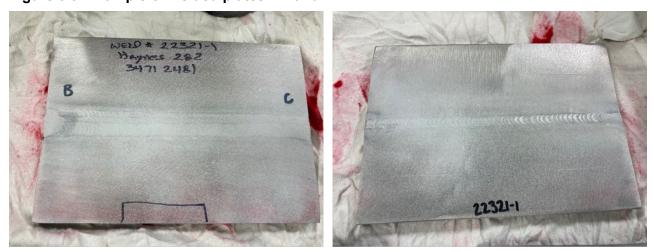
Figure 3.3 shows images of the bottom and top surfaces of the welded plates. There were no visual indications of cracking, and dye penetrant inspection did not show any cracking (Figure 3.4). Figure 3.5 shows a cross-section of the welded joint and no obvious cracking was observed.

Bottom Surface



Top Surface

Figure 3.3. Example of welded plates # 1 and #2.



No Dye Penetrant Indications

Figure 3.4. Dye penetrant examination did not show obvious defects.



Figure 3.5. Cross-section of a welded joint of plates #1 and #2.

Welded pipe Specimens:

Centrifugal cast pipes were also welded by MetalTek International in the solution annealed condition (Indicated by Haynes[®]282[®]- HT4 in Table 4.1).

Vacuum Solution Heat-treated at 1148.92°C (2100°F) / 90 minutes / Ar gas quench

Double J groove 17 passes

1st pass was autogenous root pass

Subsequent passes at 20-30 IPM, using **0.045" Haynes 282 6 filler** at nominally 150 Amps, 9.5 V



Figure 3.6. Welded Centrifugal Cast Haynes[®]282[®] Tube.

This completes requirements for milestone 3.1.

Strips were cut out of the welded pipe and subsequently were post weld heat-treated as follows: 1010°C (1850°F) for 2 hours, air cooled to room temperature, followed by 787.8°C (1450°F) for 8 ours, air cool to room temperature. Figure 3.7 shows the weld profile of the welded centrifugal cast tubes.

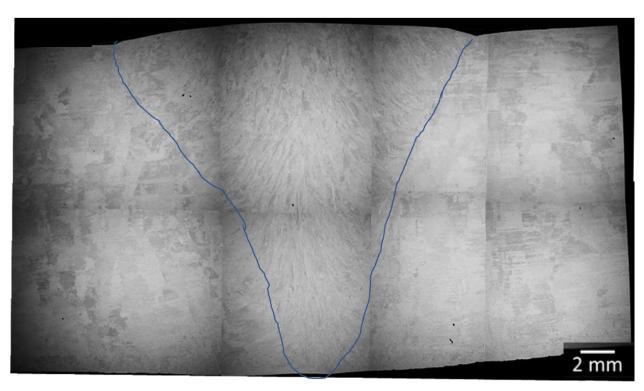


Figure 3.7. SEM image of the weld produced in the centrifugal cast pipe.

Tensile Testing results:

Figure 3.8 shows the schematic of specimens used for cross-weld tensile testing of the welds fabricated from plates # 1 and # 2 and specimens prepared from the welded, centrifugally cast pipes. Cylindrical specimens with 0.188 inch diameter were prepared from the welded plates while specimens with a diameter of 0.35" inch were prepared from the welds made from the centrifugally cast pipe. Figure 3.9 shows results from tensile testing of post-weld heat-treated specimens obtained from plates #1 and #2 fabricated from the centrifugally cast specimens and from welded centrifugally cast pipe. One room temperature test, 2 tests at 750°C, and 2 tests at 800°C were completed on specimens fabricated from plates. Two room temperature tests, 3 tests at 750°C, and 3 tests at 800°C were completed on specimens fabricated from welded pipe.

It should be recalled that the pipe was welded in a modified solution annealed condition and subject to a modified heat-treatment procedure shown as HT4 in table 3.1. Hence to facilitate a direct comparison with the weld properties, properties of the base material were also obtained in the HT4 condition and shown in Figure 3.9.

For the welds fabricated from the plates, the strength at room temperature is lower than that of base heat-treated centrifugally cast tube (HT3) material but the strengths are comparable at high temperatures. The elongation in the post-weld heat-treated condition was lower (between 4

and 7 %) when compared to the cast and heat-treated condition which was in the range of 11-12%. In contrast, the strength of the welded centrifugally cast pipe was greater than the base material subject to the modified heat-treatment HT4 at all temperatures. Specimens fabricated from the welded pipe showed ductility from 6.27% at room temperature to about 15.5% at 800°C due to the larger specimen diameter. This completes requirements for milestone 3.2.

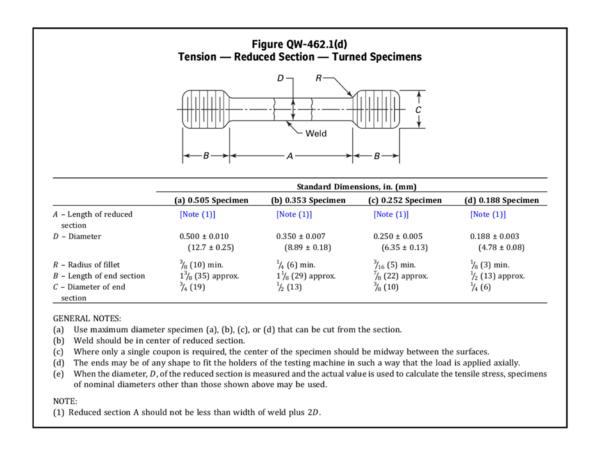


Figure 3.8. Schematic of tensile specimens fabricated from welded material.

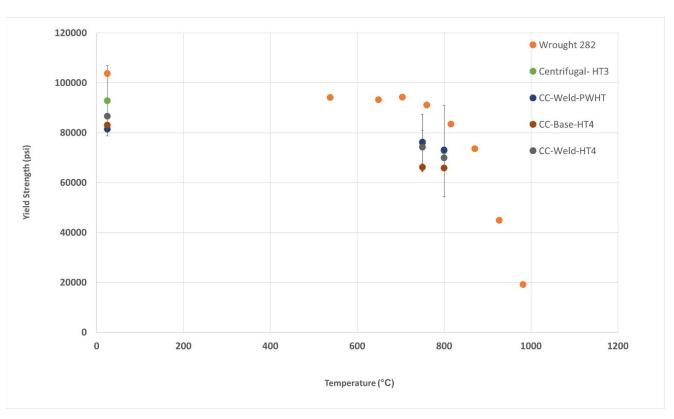


Figure 3.9. Results on weld yield strength after post weld heat-treatment.

4.0 Cost comparison with target: A comparative analysis of materials and process cost was performed in this BP by MetalTek based upon cost estimates for wrought product.

Assumptions used in the calculations are as follows:

- Cost analysis is based on current material prices. Obviously if Co or Ni prices spike or drop significantly so that their relative value to each other changes, then the material portion would increase or decrease over the processing portion
- Cost analysis done on a per lb finished part basis with a single solution heat treat.
 Wrought low volume used as baseline, all others used as a fraction of that cost on a % basis.
- Assumes NDT and other cert costs are identical.
- Centrifugal cast material goes through the following steps:
 - Melting
 - Casting
 - ID Boring and Cut-off
 - Single step heat-treatment
 - NDT

_

- Assuming finished tube parts covering roughly 3-12" diameter and wall thicknesses ranging from 0.5-1.5" in 6-12' lengths. Essentially this size range is typical range for horizontal centrifugal casting. Obviously not all combinations of wall thickness and diameter can be produced by either process.
- Low volume (*i.e.* 1 or 2 pieces) is similar cost whether wrought or cast. Data that Metaltek has for these types of alloys plays this out.
- Wrought large heat assumes a single full heat on the order of 10,000 25,000 lbs. This is similar to 10-100 pieces of cast product in terms of weight.
- For cast ~ >100pcs volume, we can realize more economies of scale in the material and melting process. There are probably further economies in wrought if a dedicated setup for multiple heats is used - however we are talking pushing more to commodity volumes.
- Lead time is a big thing here. Lead time on castings would be much shorter that fully wrought processed materials.
- Figure 4.1 shows a comparison of the cost of wrought and cast products at different volumes. There are cost advantages in cast products at lower volumes. This assessment should be treated as confidential. This should not be interpreted as wrought vs cast competition. There are advantages to both in certain situations.
 - o If one needs the best properties, large volumes = chose wrought,
 - If flexibility and low volumes = cast

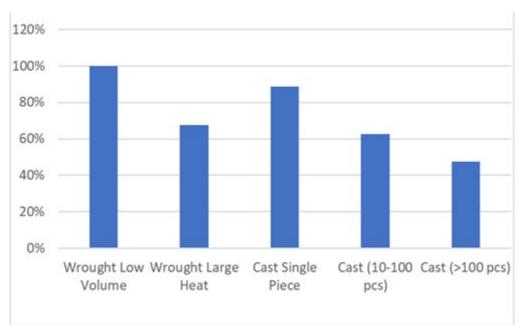


Figure 4.1. A comparative assessment of cost of wrought and cast tubular product Haynes®282®

This completes work towards Milestone 2.5

Summary and Conclusions

This project has shown that

- 1. Haynes®230®, and Haynes®282® can be successfully produced using investment and centrifugal casting techniques
- 2. Yield strengths of up to 85% of wrought Haynes[®]282[®] can be achieved in centrifugally cast Haynes[®]282[®] in the temperature range of 750°C to 800°C with appropriate homogenization, solution annealing and aging heat-treatments.
- 3. Creep properties of homogenized, solution annealed Haynes[®]230[®] are comparable or better than those of wrought Haynes[®]230[®] at intermediate and lower stress levels in the temperature range of 750°C to 800°C.
- 4. Creep properties of homogenized, solution annealed, and aged centrifugally cast Haynes[®]282[®] are comparable to or better than wrought Haynes[®]282[®] in the temperature range of 750°C to 800°C at intermediate to low stress levels.
- 5. Rupture lives of 10,000 hours can be obtained at stress levels greater than 134 MPa at 750°C in homogenized, solution annealed, and aged centrifugally cast Haynes[®]282[®].
- 6. Centrifugally cast Haynes[®]282[®] can be successfully welded and yield strengths of 85% of the base material yield strength in the temperature range of 750°C to 800°C can be achieved with appropriate heat-treatments.

Acknowledgements

The authors would like to acknowledge Cecil Carmichael, Kevin Hanson, Greg Cox, and Ian Stinson for heat-treatments, Shane Hawkins and Kelsey Hedrick for mechanical property evaluation, and Tom Geer, Victoria Cox, and Caitlin Duggan for metallography. The authors would also like to acknowledge Dr. Kamala Raghavan, Dr. Rajagopal Vijaykumar, Shane Powers, and Dr. Avi Shultz for their support.

References:

- 1. Haynes International. Haynes 282 alloy technical data sheet. Technical report, 2017.
- 2. PCC Energy Group, "INCONEL® Alloy 740H® A Superalloy specifically designed for advanced ultra-supercritical power generation.
- 3. R. Viswanathan, J. Hawk, R. Schwant, D. Saha, T. Totemeier, S. Goodstine, M. McNally, D. B. Allen, R. M. Purgert, "Steam Turbine Materials for Ultrasupercritical Coal Power Plants, Final Technical Report DOE Award Number DE-FC26-05NT42442 OAQDA-OCDO Grant Number 05-02(B).