

(18) Preventing Solidification Defects in Large Superalloy Castings Used in Advance Electric power Systems

This two-year effort will study macrosegregation in superalloy remelting processes. Weaknesses in existing models, particularly the inability to accurately predict partition coefficients of key elements under real operating conditions, will be addressed. Compositional effects of individual alloying elements in different alloys will be characterized so that a comprehensive database will be available in a useable format. A predictive methodology incorporating advanced computation technologies will be developed. Alloy index of freckle and center segregation formation can be determined for complex alloy compositions with efficient computational and laboratory analysis. The ultimate goal is to develop a predictive technology that can be applied commercially to prevent solidification defects for large superalloy castings used in advance electric power systems.

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Project Participants: GE Energy; Special metals Corp.; Pennsylvania State University

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Presentations/Publications

None.

Patents

None.

Progress in Past Quarter and Current Status

Task 1: Solidification Modeling

Task 1.1 Thermodynamics Database and Partition Coefficients

Sub-task 1.1.1 Thermodynamic Modeling (Liu/PSU)

The goal for this subtask is to obtain the partition coefficient of the model systems Ni-Cr-Nb, Ni-Cr-Fe, Ni-Cr-Ti, Ni-Cr-Fe-Nb, and Ni-base superalloys IN 706 & IN 718, through first-principle calculation and thermodynamic modeling. During the first quarter, the research efforts were focused on conducting literature review and comparing current partition coefficients measurement and thermodynamic calculations.

The Fe-Ni-base superalloys, such as IN706 and IN718, are used in the latest generation of gas turbines. Due to the presence of convection, solidification of large ingot usually occurs with segregation [1,2]. As a consequence the solidification defects (e.g., freckle) occur. To understand and prevent the solidification defects, the partition coefficients for each solute element (i.e., the ratio of solid composition over liquid composition at the solidification front) are needed, as the main reason for segregation is due to the nonuniform distribution of alloying elements between liquid and solid during solidification. The partition coefficients can be determined either from the measurements or from the available thermodynamic (and kinetic) databases.

Thermodynamic analysis in combination with experimentation, Long et al. [3] assessed the changing of liquid composition (partition coefficient) during solidification and its effect on freckle formation in superalloys. Using the Scheil model together with Thermo-Calc software and the commercial Ni-base

database, the predicted freckle formation tendencies (represented by the Rayleigh number in which the partition behavior was included) for superalloys IN706 and IN625 were in good agreement with those of semi-experimental analysis (where the liquid composition at different liquid fractions were measured), but there was an obvious difference (>30%) for superalloy IN718. Long et al.[3] attributed this discrepancy to the less accurate database. Using the commercial Ni-base thermodynamic database, the predicted partition coefficient of Nb in IN718 by Thermo-Calc software is 0.36, whereas its experimental value is around 0.5 [4]. Therefore the improvement of the present Ni-base database is necessary, which can be achieved from the recently evaluations of systems: Nb-Ni [5], Fe-Ni [6], Cr-Nb-Ni [7], and Fe-Cr-Ni [8].

In terms of thermodynamic and kinetic databases, Hillert et al. [9] simulated the Cr segregation ratio (partition coefficient) during solidification of Fe-Cr-C alloys by using the software package DICTRA. They concluded that the microsegregation behavior of Cr as a function of the C content can satisfactorily be described with the planar model and by approximating the rates of diffusion of C and Cr in the liquid and C in the solid phase as infinitely fast. They also emphasized that diffusion in the solid phase during cooling after solidification should not be neglected.

In summary, the measured partition coefficients are greatly affected by the conditions of measurement and sorting method. The predicted partition coefficients in IN718 alloy are not accurate. The influences of kinetic factors (e.g., back diffusion and cooling rate) upon the experimental partition coefficients can be satisfactorily simulated by DICTRA code together with the corresponding thermodynamic and kinetic databases.

Sub-task 1.1.2 Experimental Investigation on Partition Coefficients (Liu/WVU)

The objective of this subtask is to experimentally study the solidification characteristics of Inconel 706, Inconel 718, and several model Nb-, or Ti-content alloys. The solidification characteristics include partition coefficient, liquid composition, core dendrite composition, and liquid fraction as the function of temperature. The experimental outcome will be used to calibrate thermodynamic calculations. During the first quarter of the project, the research efforts were focused on literature review and setting up experimental apparatus.

(1) Various Methods on Partition Coefficients Measurement

Experimentally, the partition coefficients are obtained by measuring the quenched solid and liquid composition using methods such as electron probe microzone analysis [10]. Sung and Poirier [11] reviewed the partition coefficients in Ni-base alloys measured mainly by the following three techniques. The first technique involves measurement of the minimum concentration of a solute in a solidified sample. Assuming no diffusion in the solid during solidification, then the minimum concentration divided by the concentration in the melt gives the partition coefficient. However the obtained partition coefficients by this technique are sensitive to the solidification conditions such as cooling rate and convection. The second technique involves planar solidification of the alloy followed by quenching. Microprobe analysis of the solid and quenched liquid at the interface gives the partition coefficients. This technique results in an equilibrium partition coefficient since the growth rates are so slow and there are no effects of interfacial kinetics. In the third technique, the alloy is heated and maintained at a coexisted region of solid and liquid. Then it is quenched and microprobe analyses of the solid and quenched-liquid compositions yield the equilibrium partitions. Due to the different conditions of measurement, the obtained partition coefficients differ from each other for each Ni-base alloy. For example, the measured partition coefficients of Nb in Ni-base superalloys range from 0.24 to 0.50 around the liquidus temperatures [11].

Besides the conditions of measurement, the partition coefficients also rely on the manners in which the measured data are treated [12, 13]. The most widely used method was developed by Flemings et al. [14] to sort randomly sampling data. This method fails to conserve the chemical information pertaining to each measurement, and the variations in the local composition are not reflected in the resulting cumulative profiles [12]. In order to overcome the shortcomings in Flemings's sort, many improved methods have been developed and reviewed by Ganesan et al. [12]. Such as Yang et al.[10] gave a Monte Carlo sampling method. Ganesan et al. [12,13] presented two new alloy-independent data treatment algorithms that are capable of separating the effects of scatter in the data from the underlying segregation trends, assigning each measurement location a unique fraction solid.

(2) Available Partition Coefficients

Because of the difficulty in conducting experiments, there is little experimental data on partition coefficients available currently. During the first quarter of the project, WVU has conducted extensive literature search and collected partition coefficients of available superalloys and Ni-Cr-Nb, Ni-Cr-Ti, & Ni-Cr-Fe-Nb model alloys. The data collected are listed as Tables I-III.

Table I: Partition coefficients measured by DuPont et al. [¹⁵, ¹⁶]

	Alloy Composition (wt%)						Partition Coefficients						
	Fe	Cr	Ta	Nb	Si	C	Fe	Cr	Ta	Nb	Si	Ni	C
Alloy 4	10.72	19.08		1.91	0.4	0.155	0.99	1.02		0.44	0.83	1.02	
Alloy 7	10.7	19.3		4.86	0.52	0.01	0.99	1.06		0.44	0.69	1.01	
Alloy 8	10.8	18.9		4.72	0.52	0.17	1	1.08		0.45	0.71	1.02	
Alloy 8							1	1.08		0.45	0.81	1.01	
Average							1	1.06		0.45	0.76	1.02	
Alloy 15	45.4	19.54		4.88	0.66	0.01	1.06	1.03		0.27	0.58	1	
Alloy 16	44.47	19.45		4.77	0.64	0.216	1.07	1.02		0.25	0.61	1.01	
Alloy 16							1.06	1.01		0.23	0.59	1	
Average							1.06	1.02		0.25	0.58	1	
Inconel 625										0.54	0.57		0.21
Inconel 625							1.02	1.05		0.46		1.04	
Inconel 718							1.04	1.03		0.48	0.67	1	
Inconel 909							1.1	1.1		0.42	0.69	0.97	
ThermoSpan							1.08	0.96		0.33	0.89	0.97	

Table II : Parttion Coefficients measured by Sung and Poirer [¹⁷,¹⁸]
 Alloy Composition (wt%)

	Alloy Composition (wt%)												Partition Coefficients											
	Co	Ti	Al	Fe	Cr	Ta	Nb	Mo	W	Hf	Re	Si	Co	Ti	Al	Fe	Cr	Ta	Nb	Mo	W	Hf	Re	Si
Ni-9.2Al-5.9Ta			9.20			5.90									0.66			0.61						
Ni-6Al-15.2Ta			6.00			15.20									0.66			0.59						
Ni-5.8Al-15.2Ta			5.80			15.20									0.54			0.48						
Inconel 738LC	8.30	3.50	3.40		15.70	1.90	0.90	1.90	2.90				1.10	0.60	1.20		1.05	0.70	0.40	0.85				
ATS 381-G	10.00	1.90	5.30		6.30	2.00		2.50	9.90				1.05	0.70			0.95	0.80		0.90				
Waspaloy IN-100	13.50	3.00	1.50		19.50			4.30					1.10	0.80	0.80		1.00			0.90				
Rene'77	15.00	4.70	5.50		10.00			3.00							0.91									
Mar-M200	18.50	3.50	4.25		15.00			5.20							0.85									
Inconel X750	10.50	2.00	4.90		9.70				12.60				0.60	0.90			0.93							
Inconel 617		2.40	0.70	7.30	15.60	0.70	0.20						0.54	1.18	1.09	0.99	0.59	0.36						
Hastelloy XR	12.30	0.50	0.70	1.60	20.80			11.80					1.06		1.36	1.06	0.99			0.79				
Inconel 706				17.90	20.70			11.30	1.40							1.10	0.97			0.71				
Inconel 700	0.80	0.60		38.00	19.00	1.10	1.30	1.00								1.10	0.99	0.49	0.24	0.70				
Alloy F	27.40	1.50	3.00		13.20	1.40	1.80	3.80					1.08	0.51	0.96		1.05	0.60	0.34	0.90				
Alloy G		0.10		2.70	18.00	2.40	1.90	17.00								1.12	1.05	0.75	0.40	0.81				
Alloy H	17.80	0.40			19.40	1.20	1.20	5.90					1.10				0.99	0.68	0.30	0.79				
Alloy I		0.40		18.20	19.80	1.50	1.40	5.80								1.11	0.99	0.60	0.31	0.85				
Alloy J	16.00	0.90			16.70	1.10	1.80	15.10					1.09				1.02	0.66	0.37	0.83				
Ni-7.2Al-6.4Ta		0.60		16.00	16.70	2.00	1.50	15.80								1.13	1.01	0.68	0.34	0.77				
PWA			7.20			6.40									0.90			0.75						
PWA	5.00	1.40	5.00		10.40	12.00			4.00				1.13	0.43	0.89			1.21						

1480																			
Ni-21Fe-			21.00			30.20					1.21			0.67					
30.2Mo																			
Alloy 77	10.00	2.50	2.00	16.00	2.90	2.10	2.00			1.03	0.62	1.08	1.02	0.80	0.89				
Mar-	10.00	0.90	5.20	8.60	3.30	0.70	9.20	1.30		1.06	0.73	0.93	1.00	0.77	1.00		0.12		
M247																			
CMSX-4	9.00	1.00	5.60	6.50	6.50	0.70	6.00	3.00		1.06	0.60	0.82	0.97	0.68				1.55	
Ni-5Si-Al			2.84							5.10		1.34							0.47
Ni-5Si-	3.28									5.12	1.18								0.49
Co																			
Ni-5Si-Cr				3.20						4.91			1.13						0.59
Ni-5Si-Fe			3.06							5.09		1.18							0.51
Ni-5Si-						3.22				4.97				0.76					0.53
Mo																			
Ni-5Si-W							3.34			5.30									0.49
Ni-Cr-Al		6.8		28.8								0.78	0.97						
Ni-Cr-Al-		6.6		8.9	7.8							0.92	0.94	0.77					
Ta																			
Ni-Cr-Al-		6		8	6							0.92	0.95	0.77					
Ta																			
Ni-Cr-Al-		6		8	6							0.91	0.93	0.77					
Ta																			
Ni-Cr-Al-		2.69		6.92			9.3					0.72	0.89					1.78	
Re																			
Ni-Cr-Al-		2.64		6.79			12					0.85	0.87						
W																			

Table III : Partition Coefficients measured by WVU [¹⁹]

	Alloy Composition (wt%)										Partition Coefficients								
	Ni	Co	Cr	Fe	Nb	Mo	Ti	Al	C	Si	Cr	Fe	Nb	Mo	Ti	Al	Co	Si	
HV8549	50.43	0.02	19.87	18.19	5.31	0.02	5.53	0.14	0.36	0.1	1.11	1.15	0.45		0.62				
HV8520	49.53	0	19.36	18.06	7.32	0.06	5.09	0.29	0.11	0.12	1.14	1.17	0.44		0.64				
HV8551	49.56	0	19.69	18.21	5.37	0.02	5.59	1.14	0.04	0.09	1.14	1.16	0.44		0.65	1.05			
HV8524	32.69	0	20.02	36.04	5.39	0.01	5.43	0.15	0.05	0.11	1.11	1.15	0.31		0.53				
HV8586	41.81	0.35	16.06	35.37	3.23	0	2.94	0.15	0.03	0.01	1.05	1.08	0.39		0.5				

HV8582	59.83	0.03	20.16	5.16	14.03	0.02	0.12	0.34	0.03	0.07	1.17	1.22	0.53					
HV8584	57.77	0.02	20.07	5.15	14.28	0.03	0.13	0.35	0.04	1.95	1.31	1.35	0.41					0.51
HV8583	54.79	0.01	19.95	5.15	14.27	5.01	0.16	0.28	0.15	0.09	1.16	1.22	0.52	0.82				
HV8528	24.77	11.91	19.97	35.7	2.98	0.02	2.97	1.4	0.04	0.14	1.04	1.11	0.3		0.47	1.05	1.02	
HV8526	56.6	12	19.98	0.28	5.39	0.01	5.37	0.14	0.06	0.11	1.08	1.19	0.43		0.65		1.08	

(3) Experimental Setup:

Alloy Preparation: WVU will use arc button furnace to make model alloy samples for the partition coefficient measurements and other solidification characteristics investigation. After extensive inventory search and communication with partners, we found one furnace in Prof. Seera's lab in the Physics department at WVU. We are grateful that Prof. Seera allow us to move the furnace to Mechanical & Aerospace Engineering department to carry out research on this project.

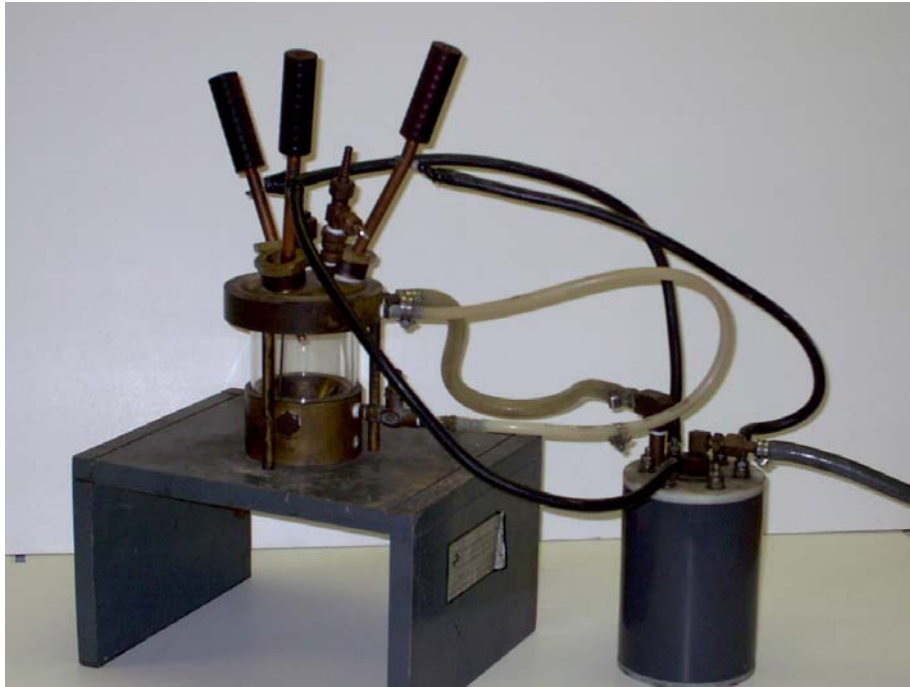


Figure 1: The arc button furnace which will be used to make alloy samples for this research project.

Figure 1 shows the furnace in MAE lab. It should be mentioned that some parts of the furnace are missing since this furnace has not been used for several years. We have contacted the manufacturer and ordered the missing parts, such as the electrode and copper hearth. The furnace will be hooked up and ready for making alloy samples after we have received the order.

Model Alloy Design: Current gas turbine alloy is Ni-base superalloy Inconel 706. To improve the operation efficiency of the turbine systems, the new turbine disk alloy must have higher temperature capability than Inconel 706. The alloys of interests are Inconel 718, Inconel 625, and Inconel 725. The alloy compositions of these alloys are listed as Table IV.

Table IV: Chemical compositions of the alloys of interests (wt%)

	Ni	Co	Cr	Fe	Nb	Mo	Ti	Al	C	Si
Inconel 718	53.62	0	18.19	17.77	5.41	3.01	0.98	0.56	0.04	0.03
Inconel 706	41.39	0.03	16.3	37.03	2.98	0.12	1.7	0.19	0.01	0
Inconel 625	60.11	0.06	22.09	4.4	3.53	9.1	0.29	0.26	0.02	0.03
Inconel 725	57		20.75	10	3.375	8.25	1.35			

Microsegregation in Nb-containing superalloys provides the fundamental knowledge to understand the macrosegregation behavior. During the first quarter, the research team, including WVU (Xingbo Liu), PSU (Zi-Kui Liu), SMC (John J. deBarbadillo), and GE Energy (Joseph J. Jackson), have had one conference call and exchanged emails to discuss the compositions of the first batch of experimental alloys. It has been decided that the first batch of the alloys will be the N-Cr-Fe-Nb alloy system with the compositions listed as Table V.

Partition Coefficients Measurement: The partition coefficients of the alloys will be measured by square-mesh systematic point counting metallographic technique developed by WVU in recent years. Samples will be melted and solidified at certain heating and cooling rates (typically 20°C/min) in TA-1600 Differential Thermal Analyzer cell, then the analysis will be conducted on a Hitachi-4700 FE-SEM with EDAX system. Multi-element standards will be used for the analysis since the bulk compositions of the samples are already known.

During this quarter, WVU performed baseline calibration and temperature calibration of the DTA equipment. In addition, the graduate student Jairo Valdes Ortiz was trained on SEM operation at Oak Ridge National Lab, through ORNL's SHaRE program. The partition coefficients measurement will be carried out in next quarter after the alloy samples are prepared with the above mentioned arc button furnace.

Table V: Chemical compositions of the first batch of experimental alloys (wt%)

	Ni	Cr	Fe	Nb
Ni-Cr-Nb		15		3
		15		4.5
		15		6
		20		3
		20		4.5
Ni-Fe-Nb		20		6
			5	3
			5	5
			10	3
			10	5
			18	3
			18	5
Ni-Cr-Fe-Nb			36	3
			36	5
		18	10	3
		20	18	3
	18	18	5	
	20	10	5	

Task 2 Directional Solidification Verification

The goal of this task is to experimentally study the effects of composition and kinetic parameters during solidification on the formation of macrosegregation defects, especially freckles. A directional solidification (DS) furnace will be built and employed in the investigations.

Task 2.1 Verification of Model Alloys (Liu/WVU)

The objective of this task is to carry out DS of a series of simplified alloys with various eutectic phases, such as Laves phase, δ -Ni₃Nb, or η -Ni₃Ti, to verify and modify phase diagram database and solidification modeling. During the first quarter, the research effort was focused on construct DS furnace for future experiments.

There are two kinds of DS furnaces: horizontal furnace and vertical furnace. Upward vertical directional solidification has been usually used to investigate the macrosegregation mechanism, especially for freckles. However, upward directional solidification is not suitable to explain macrosegregation during Vacuum Arc Remelting (VAR)/Electric Slag Remelting (ESR) processes because of the large difference of solidification conditions. Therefore, during this research, horizontal directional solidification is the better choice to simulate VAR/ESR solidification.

Through extensive inventory search and communicating with industrial partners, we found that there was a DS furnace in SMC, which may be suitable for the research. The furnace has been moved to WVU and the researchers at WVU will build the DS furnace based on it. It should be pointed out the furnace is not functional now because it has not been used for several years. The WVU team is checking the status of the furnace and making the plans to fix and update the furnace. This furnace will be employed for DS verification of both model alloys and commercial available superalloys after it is up and running.



Figure 2: The Directional Solidification (DS) furnace at WVU.

Plan for Next Quarter:

Task 1: Solidification Modeling

Task 1.1 Thermodynamics Database and Partition Coefficients

Sub-task 1.1.1 Thermodynamic Modeling (Liu/PSU)

Ni-Cr-Fe-Nb phase diagram and the associated database will be developed by the first-principle method and the results will be compared with commercially available database.

Interpretation of available partition coefficients measurement by taking back-diffusion into account will also be carried out.

Sub-task 1.1.2 Experimental Investigation on Partition Coefficients (Liu/WVU)

The research effort of the next quarter will be to prepare the alloy samples listed in Table IV and measure the partition coefficients of these alloys. In addition, the research team will determine the compositions of next batch alloys list.

Sub-task 1.1.3 Kinetic Effects

The partition coefficients and other solidification characteristics of the first batch (Ni-Cr-Fe-Nb) of alloys will be investigated under various heating and cooling rates to study the kinetic effects on solidification behaviors of these alloys.

Task 2 Directional Solidification Verification

Task 2.1 Verification of Model Alloys (Liu/WVU)

The research effort of the next quarter will focus on the construction of the horizontal DS furnace.

Task 2.2 Effect of Processing Parameters

The research team will decide the experimental plan on the investigation of processing parameter effect, based on results of tasks 1.1.1 through 1.1.3. The experiments are expected to start from the third quarter of the project.

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