Development of High Performance Ni-base Alloys for Gas Turbine Wheels Using a Co-precipitation Approach

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(ge)

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Outline

- Background
- Co-precipitation Concept
- Present Status:
 - Summary of Round-2 mechanical testing results
 - Round-3 alloy design strategies and modeling
- Progress against milestones
- Summary



Path to Higher Efficiency Gas Turbine

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Next generation heavy duty gas turbine wheels must operate at higher temperatures to enable combined cycle efficiency improvements

3

Designing a Higher Temperature Capable Wheel

Use steel and cool to lower the effective temperature



Invent a better γ" (Ni₃Nb) strengthened alloy



 γ " strengthening phase is unstable at temperatures >1200°F. Use an Aviation disk alloy strengthened with γ' (Ni₃Al)



Precipitation kinetics result in severe over aging of γ' , yielding poor properties.





A new approach to alloy design is required to enable high temperature wheels

Coprecipitation Concept

- Leverage the coprecipitation of γ' and γ" to restrict γ' coarsening during slow cooling of thick section components.
- Desire "compact" coprecipitate structure first identified by *Cozar and Pineau (1973)*
- In previous NETL program (DEFE0026299):
- Developed successful sub-scale billetizing/forging procedure
- Screened tensile and hold time fatigue crack growth properties (*no creep testing or evaluation*)







Program Structure

Program Timeline

Mo. 1: **Program Initiation**

Mos. 2-8: Round 1 Alloys (2)

Mos. 9-20: Round 2 Alloys (4)

Mos. 21-32: Round 3 Alloys (2)

Mos. 33-34: Program Wrap-Up

Mo. 35-36: Documentation

1.0 Project Management [OSU]

2.0 Alloy Processing and Evaluation

2.1 Alloy Selection and Processing [GRC]

2.2 Heat Treatment and Microstructure **Characterization** [GRC]

2.3 Aging Informed by High Temperature Indentation [OSU]

2.4 Phase Field Modeling and **Characterization of Coprecipitation [OSU]**

2.5 Mechanical Testing [OSU/Metals Tech]

3.0 Property Model Development

3.1 Deformation Mechanism Characterization [OSU]

- 3.2 Yield Strength Model [OSU]
- 3.3 Creep Model [OSU]

The Ohio State University

Professor *Michael Mills* (PI) overall program coordination and lead on mechanical testing and deformation microstructure characterization Professor Yunzhi Wang (co-PI) will lead the modeling efforts

GE Global Research

Rich DiDomizio and Andy Detor lead alloy preparation and processing Reza Sharqi-Moshtaqhin has performed preliminary TEM microstructure characterization of as-processed alloys

Metals Technology, Inc

Robert Hayes will perform the creep testing as cost-share to the project



Pls: **Michael Mills** Yunzhi Wang

Collaborators: Bob Hayes (MTI) Rich DiDomizio (GER) Andy Detor (GER)

Experiments: Semanti Mukhopadhyay **Christopher Zenk**

Modeling: Kamalnath Kadrivel Hariharan Sriram





















Updates on Experimental Efforts

 Microstructural Characterization of Proposed Round 3 Alloys

Three different design strategies were introduced in the previous meeting. GE has produced alloys based on these design strategies in as-cooled condition. OSU has started microstructural characterization of these alloys.

• Microstructural Characterization of deformed alloys

OSU has been carrying out ECCI and STEM-HAADF to characterize and study deformation in Round1 and Round 2 alloys in as-crept condition. Two alloys (IN718-27 stabilized & IN718-50) crept at 1200°F (650°C) have been studied.

Results are compared with deformation studies carried out on In-Situ tensile tested IN718-50.





Summary of Round 1 & Round 2 Alloys

- Alloys and Processing



Summary of Round 1 & Round 2 Alloys ROUND 1 (alloy 27) ROUND 2 (alloy 50)



Bimodal coprecipitates Secondary size – 150-200nm Tertiary - 5-20nm

Summary of Round 2 Alloys (alloy 50)

FEI Thermo Fischer Quattro ESEM

- Intergranular Failure







Heterogeneous Precipitation at Annealing Twin Boundaries Alloy 27





Not previously reported in superalloys

• Mechanism under investigation



Summary of Round 1 & Round 2 Alloys

- Overall Mechanical Properties



13

6°C/min

Room

Temp

Is Situ SEM Tensile Testing

Tests at Room Temperature and 580°C





Intergranular Crack Initiation



Intergranular cracks initiate frequently at the intersection of twin boundaries and grain boundaries



Summary of Round 2 Alloys

Localized and intense slip parallel to annealing twin boundaries





Characterization and Analysis of Slip



Loading axis direction	$\overrightarrow{L_A}$ = [4 $\overline{3}$ 30]					
Foil plane direction	$\overrightarrow{F_P}$ = [$\overline{4}$ 9 15]					
	$\overrightarrow{B_1}$ = [$\overline{1}23$] ZA $\overrightarrow{g_{B1}}$ = [$1\overline{1}1$]					
Beam directions and corresponding diffraction conditions	$\overrightarrow{B_2}$ = [011] ZA					





Step

Step

Characterization and Analysis of Slip

G1 / SIII	In the second	1 1111100	Schmid factor	Plane	Direction \vec{b}
T / F-1011c		GZ-	0.356	(111)	[101]
		1 1 1 1 1 Ind have the	0.096	(111)	[110]
// / [011]9 [1	110]p		0.452	(111)	[011]
	1 1 1 1				-
[1-11]p		1 HILL H	0.346	(111)	[011]
			0.090	(111)	[110]
			0.435	(11 1̄)	[101]
			0.345	(111)	[101]
			0.335	(111)	[01 1]
		HHH H H	0.010	(1 11)	[110]
× 7 – 1					
▲ <i>∠</i> − [0.441	(111)	[011]
		0.000	0.425	(111)	[101]
	View direction along		0.016	(111)	[110]
	[1 2 3] ZA				
	 close (4°) to [$\overline{4}$ 9 15]	"step"- like sur	face relief a	t (111) TB	is formed
(îoı)		by activity of tw	o slip syste	ms:	
·E [011]		.,,			
[110]	_	•			
	→ F _P = [4 9 15]	• SF {(111)[01]	1]} = 0.4	41
				411 0.4	
		• 5F {(111)[10	1]} = 0.4	25
48.5°	\backslash				
L _A	= [4 3 30]				
Y = [7 12 9]					

Summary of Round 2 Alloys

- High Resolution HAADF-STEM



Localized and intense slip parallel to annealing twin boundaries





IN718-50 (HT-2)

Tension Creep at 1200°F (650°C) and 100 ksi (689 MPa)

This sample reached 0.5% strain at 8.5 hours. Sample ruptured at 10 hours & 0.6% strain

20

Ni Nb 50 nm













STEM BF Image



Slip parallel to Annealing twin boundary, similar to what was observed in insitu tensile tested sample

BF

5 nm

G2

Microtwinning!

50 nm







IN718-27 (HT-3)

Tension Creep at 1200°F (650°C) and 75 ksi (517 MPa)

This sample reached 0.5% strain at ~500 hours.



Faults parallel to annealing twin boundary











HAADF STEM

[110]







HAADF STEM



Round 1 and 2 Alloys: Key Issues





Difference in between **718-27** and **718-27 Stab** : Latter has tertiary coprecipitates **Round 2 alloy (IN718-50) does not have tertiaries**



Round 2 alloy (IN718-50) shows intergranular failure at room temperature and high temperature





Even though IN718-50 performs better in compression than tension (due to grain boundaries), conventional IN718 outperforms both cases



Summary of Round 3 Alloys







Summary of Round 3 Alloys

- Follow Three Distinct Design Strategies

Alloys produced and heat treatments by GER Problem: Poor Intergranular Strength

Strategy 2

Strengthen grain boundaries through C / B / Zr additions

Characterize microstructures after heat treatments and downselect for mechanical testing

Alloy Name	Ni	Cr	Fe	AI	Ті	Nb	Мо	с	в	Zr
718	52.5	19.0	18.9	0.50	0.90	5.13	3.1	0.02		
718-027	52.9	18.7	18.9	1.07	0.95	4.42	3.1	0.02		
718-071 718-073	55.8 51.9	18.7 18.7	18.9 18.9	1.07 1.07	0.95 0.95	4.42 4.42	0.0 4.0	0.06 0.02	$0.015 \\ 0.015$	0.03 0.03
718-074 718-077	55.7 51.7	18.7 18.7	18.9 18.9	1.07 1.07	0.95 0.95	4.42 4.42	0.0 4.0	0.06 0.02	$0.100 \\ 0.100$	0.10 0.10
718-079	55.8	18.7	18.9	1.07	0.95	4.42	0.0	0.02	0.100	0.03
718-080 718-082	51.8 55.8	18.7 18.7	18.9 18.9	1.07 1.07	0.95 0.95	4.42 4.42	4.0 0.0	0.06 0.02	$0.015 \\ 0.015$	0.10 0.10



Summary of Round 3 Alloys

- Follow Three Distinct Design Strategies

Strategy 3: Increase γ' volume fraction while maintaining co-precipitate potential

Problem: Poor Intragranular Strength

	Nominal Composition (wt%)										
Alloy Name	Ni	Со	Cr	Fe	AI	Ti	Та	Nb	W	Мо	С
718-056	49.3		18.6	18.5	1.37		4.58	4.70		3.0	0.02
718-057	43.4		18.2	18.1	2.23		7.47	7.67		2.9	0.02
718-058	50.1		18.9	18.8	1.39	0.62	2.33	4.78		3.0	0.02

• Ta addition and balance Ta+Ti content

Alloys produced and heat treatments by GER



• Increase coprecipitate volume fraction while also maintaining the "golden ratio" of (Al+Ti+Ta)/(Nb) ~1.

• Ta a strong gamma prime stabilizer known to improve tensile strength of alloys (as in Ta718).

Coprecipitation Modelling Overview



Combining CALPHAD and phase field modeling to develop quantitative prediction of the coprecipitation phenomena





Coprecipitation Concept

• Leverage the coprecipitation of γ' and γ'' to restrict γ' coarsening during slow cooling of thick section components.



He, J., et al. Acta Mater 46 (1998): 215-223.



Desired: form γ' then rapidly nucleate γ''

A. Detor, et al, Metall. Mater. Trans. (2017)

Updates on Modeling Efforts

- Phase field modeling to understand coprecipitation mechanisms
 - We are exploring the roles played by both chemical and mechanical effects, e.g., the concentration and stress fields created by precipitation of γ' phase, on precipitation of γ'' precipitates and the development of compact coprecipitates.
- Design of Round-3 alloys and optimization of heat treatment
 - High throughput calculations have been performed to aid design of Round-3 alloy. These calculations have helped in establishing trends of volume fractions of γ' and γ" as function of Al and Nb contents observed. Effect of Ta in IN 718 system has also been explored.
- Development and calibration of creep model
 - A fast-acting creep model considering dislocation and diffusion creep has been developed and calibrated with creep data from Round 2 alloys.



Phase Field Modeling Microstructural Evolution of Coprecipitates

T=790°C



a)t=0sec

b)t=2.25sec

c)t=15sec

- Using pseudo-ternary database to represent multicomponent system
- For each new alloys, a pseudo-ternary database need to be developed
- Semi-quantitative in nature and diffusion in a ternary system may not be able to represent diffusion in multi-component system





Coprecipitate Formation





[1] . Cozar, R., Pineau, A. Metall Mater Trans B 4, 47–59 (1973).

- Influence of nucleation of γ " on a facet of γ' of two different sizes is investigated.
- Nucleation of γ " on a smaller γ' influences the shape evolution of γ' significantly.





Efforts of Concentration and Stress fields





Nucleation of γ ["] on a small γ' results in a large Nb depletion zone (dark blue region in b)). Nucleation of $\gamma^{"}$ on adjacent faces is unlikely because of solute depletion, leading to sandwich coprecipitate.

Nucleation of $\gamma^{"}$ on a larger γ' does not deplete Nb solute on adjacent faces and the larger negative elastic Interaction energy observed on adjacent faces will assist the formation of compact coprecipitate.

Al concentration (at%)



Calculations to quantify the critical γ' size for the formation of compact vs. sandwich coprecipitates are underway.

High throughput calculations to aid Round-3 alloy design



High throughput calculations using full database from CompuTherm are carried out to aid in design of Round 3 alloy. Concentrations of Al and Nb are varied from 1.5 to 3 and 4-7 (wt%), respectively **Design considerations:**

- •
- High γ' volume fraction and satisfying stability ratio $(f_{\gamma'}/f_{\gamma''})$ predicted by phase field study Small difference in solvus temperatures of γ' and γ'' phases to reduce growth and coarsening of • γ' under slow cooling before γ'' precipitates out
- Follow (Ti+Ta+Al)/Nb ratio to form coprecipitates ٠
- Red Box indicates potential Rond-3 alloy compositions •

TTT Diagram Calculation – PanPrecipitation







TTT Diagram Calculation – IN 718-27

- KWN (Kampmann-Wagner Numerical) model for precipitation
- Homogenous Nucleation of both γ' and γ'' for now
- Expanding to heterogeneous nucleation of γ'' on γ' is underway



- Experimentally, compact coprecipitates were observed in IN 718-27
- Separation between γ' and γ'' TTT curves allow γ' core to grow to certain sizes and then be "coated" by γ'' precipitates





Calculating CCT diagram from TTT Diagram



- The rule of additivity discretizes the continuous cooling curve into steps.
 - For each step, time taken to reach transformation of x₀%(say 1%) transformation at that step's temperature is obtained from the TTT curve and the time spent on that step is found from the discretization.
 - The fractions obtained from each step are added until the sum is unity.
 - The temperature and time on the cooling curve where the sum of fractions is unity is the calculated CCT point for that cooling curve.

One can use the rule of additivity [1] is used to convert TTT curves into CCT curves: $\sum_{T_0}^{T_{CCT}} \frac{\Delta t_i}{\tau_i^{TTT}} = 1$ τ_i^{TTT} – Time taken at temperature T_i to reach x_0 % transformation under isothermal condition

TTT to CCT conversion – IN 718-27





Precipitation Calculation – IN 718-27



Bimodal microstructure Secondary coprecipitates: 150-200 nm Tertiary "hamburger" γ'/γ'' : 10-20 nm Secondary area fraction: 10.4% Tertiary area fraction: 17.2%









- Bimodal distribution of γ' is predicted by the simulation.
- The sizes predicted for the secondary and tertiary precipitates are reasonable
- The calibrated PanPrecipitation Module is being used to optimize heat treatment schedules for Round-2 alloys and design new heat treatment schedules for Round 3 alloys

Precipitation Calculation – IN 718-50







Bi-modal distribution obtained utilizing a 3-step sub solvus heat treatment on Alloy 50

Volume fraction predicted: secondary γ' precipitates ~ 11 %, tertiary ~ 18 %

MPF simulations with direct coupling with PanDat

 $\frac{\partial f^{chem}}{\partial c_i}$, $\frac{\partial f^{chem}}{\partial \phi_{\alpha}}$, M^{ij}

- This approach has been tested by simulating phase separation in a four-component system (Ti-Zr-Nb-Ta).
- Compared to classical approach (MPFM↔PE), our new approach (MPFM ↔ PDN↔PE) is 55 times faster.

Output:





- Uses interpolation on previous calculations to estimate the data.
- New calculation is performed only when necessary.

 This PanDat + MPF simulations can be used to benchmark the TTT/CCT diagram obtained from PanPrecipitation that considers heterogeneous nucleation

PanEngine (PE)

- Reads thermodynamic and kinetic database (*.pdb)
- Performs equilibrium and quasiequilibrium calculation.

Linking Phase field to PanPrecipitation

- Phase field model + NEB will be used to obtain activation energy barrier and critical nucleus size for heterogeneous nucleation of γ" on γ'.
- The phase field model will be used to compare growth rates between monolith precipitates and that of coprecipitates.
- The nucleation and growth rate equations in PanPrecipitation will be informed these phase field simulation results.

(b)

Growth



Creep Model - Results

- A mean field constitutive creep model considering dislocation creep and diffusion creep has been developed and calibrated
- The use of mean field model is justified as the microstructure is homogenous
- Deformation mechanisms considered are climb bypass and particle shearing
- Diffusion creep in the model accounts for grain boundary effects on damage accumulation.
- A MATLAB code has been developed for the fast-acting model.





Summary of Modeling Efforts

- Phase field model has been used to study coprecipitate formation mechanisms and to determine critical size on achieving compact coprecipitates
- High throughput thermodynamic calculations are carried out to aid experimental design of Round-3 alloy compositions
- Heterogeneous nucleation will be implemented in PanPrecipitation to model heterogeneous nucleation of γ " on γ' . Direct coupling between multi-phase field model and PanDat thermodynamic database has also been explored with great promises
- Creep model framework has been developed and calibrated against creep data of Round 2 alloys.











Equations and Inputs for Creep Model

In γ matrix, plastic shear rate is given as:

$$\dot{\gamma}_{\rm FCC}^{\alpha} = 2\rho_{\rm FCC}b\lambda_{\rm FCC}^{\alpha}exp\left\{-\frac{Q_{\rm act}^{\gamma}}{k_BT}\right\}sinh\left\{\frac{\tau_{\rm eff}V_c}{k_BT}\right\}\\\tau_{\rm eff} = |\tau + \tau_{\rm mis}| - \tau_{\rm pass} - \tau_{\rm fric}$$

- $Q_{\rm act}^{\gamma}$ is an *effective* energy barrier for ratelimiting process in the matrix
- au_{eff} is the effective stress driving dislocation glide and is related to the strength model developed previously.

In γ' (similar for γ'') particles, plastic shear rate is given as

 $\dot{\gamma}^{\alpha}_{L1_2}$

$$= 2\rho_{\mathrm{L1}_2}b\lambda_{\mathrm{L1}_2}^{\alpha}\nu_0\sum_i P_i exp\left\{-\frac{Q_{\mathrm{act}}^i}{k_BT}\right\}sinh\left\{\frac{\tau_{\mathrm{eff}}V_{\mathrm{act}}^i}{k_BT}\right\}$$

 $\tau_{\rm eff} = \tau - \tau_{\rm pass}$

- P_i Percentage of mechanism *i* (obtained via Phase-field or DAD)
- $\rho_{L1_2}^{\alpha}$ dislocation density
- *b* Burgers vector
- $\lambda_{L1_2}^{\alpha}$ channel width
- v_0 Attempt frequency
- Vⁱ_{act} Activation volume for mechanism i
- τ^{α} Resolved shear stress for slip system α
- $\tau^{\alpha}_{\text{pass}}$ Passing stress for slip system
- α (threshold *stress*)





The composite compact and sandwich co-precipitate would be reduced to simplified γ' precipitate.



Representative microstructure used for mean field model

Key Inputs

Volume fraction, γ channel width Active deformation mechanism Experimental creep curve