



Precipitation Model Validation in 3rd Generation Aeroturbine Disc Alloys

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NASA GRC Project



Project title: Microstructure Modeling of 3rd Generation Disk Alloys Dec'06 – Dec'09

Objectives:

- 1. Successful calibration and validation of databases and physics-based precipitation models in order to predict microstructure evolution in disk alloys (ME3, Alloy10, LHSR) with high degree of accuracy :
 - Intra-granular γ' precipitation
 - GB + intra-granular γ' precipitation
 - MC precipitation
 - TCP precipitation

Multi-phase precipitation

2. Support of on-going disk alloy activities at NASA GRC

Support from Rolls-Royce, AFRL

Extensive use of NIST Ni mobility database¹ in this project.

[1] C.E. Campbell, W.J. Boettinger, U.R. Kattner, Acta Materialia, vol. 50 (2002) pp. 775-792.



Nominal compositions (balance Ni) of four 3rd generation disc alloys studied under this program

		Cr	Со	Мо	W	Al	Ti	Nb	Та	Hf	С	В	Zr
MES	wt%	13.1	20.0	3.8	1.9	3.5	3.6	1.1	2.3	-	0.04	0.03	0.05
IVIES	at%	14.5	19.5	2.3	0.6	7.5	4.3	0.7	0.7	-	0.19	0.16	0.03
гспр	wt%	13.0	21.0	2.7	4.3	3.5	3.5	1.5	1.6	-	0.03	0.03	0.05
LOHK	at%	14.5	20.7	1.6	1.4	7.5	4.2	0.9	0.5	-	0.15	0.16	0.03
	wt%	10.2	14.9	2.7	6.2	3.7	3.9	1.9	0.9	-	0.03	0.03	0.10
Alloy IU	at%	11.5	14.8	1.6	2.0	8.0	4.8	1.2	0.3	-	0.15	0.16	0.07
DD1000	wt%	15.0	18.5	5.0	-	3.0	3.6	-	2.0	0.5	0.03	0.015	0.06
KK1000	at%	16.5	17.9	3.0	-	6.4	4.3	-	0.6	0.2	0.13	0.080	0.04

- ME3 (also called René104)
- LSHR (Low-Solvus, High-Refractory alloy developed by NASA)
- Alloy 10 (developed by Honeywell)
- RR1000 (developed by Rolls-Royce)



Database and Model Parameters of <u> PrecipiCalcTM</u>



CALPHAD fundamental database and tuning parameters

- Thermo-Calc® thermodynamic databases
- $-\Delta E$: phase free energy shift in Thermo-Calc
- DICTRA compatible mobility database
- D_{scale}: diffusivity correction factor used in PrecipiCalc
- Molar Volume: multicomponent molar volume models developed for both γ and γ' under the AIM program

• Material kinetic model parameters

- σ_{coh} : coherent surface energy
- G_{el} : elastic coherency (misfit) energy
- σ_{incoh} : incoherent surface energy
- M_o: prefactor for interfacial mobility term
- $R_{coh \rightarrow incoh}$: particle size for coherency transition



Validation and Calibration Procedure



Experiments	CALPHAD Fundamental Databases	Material Kinetic Model Parameters
Equilibrium Age + APT (literature data, QuesTek) + EDS (NASA)	Thermodynamics ∆E	
Diffusion Couple + Microanalysis (NASA)	Mobility D _{scale}	
SSDTA (OSU) + APT (QuesTek)		σ_{coh} , G_{el} (est.)
Coarsening Age+SEM/TEM for γ' size and fraction (NASA)		$\sigma_{\text{incoh}}, M_O$
XRD, TEM for misfit (NASA)	Molar volume	$G_{el}, R_{coh \rightarrow incoh}$





Experiments	CALPHAD Fundamental Databases	Material Kinetic Model Parameters
Equilibrium Age + APT (literature data, QuesTek) + EDS (NASA)	Thermodynamics ∆E	
Diffusion Couple + Microanalysis (NASA)	Mobility D _{scale}	
SSDTA (OSU) + APT (QuesTek)	Tar and	$\sigma_{coh}, {\sf G}_{\sf el} ({\sf est.})$
Coarsening Age+SEM/TEM for γ' size and fraction (NASA)		$\sigma_{\text{incoh}},M_{O}$
XRD, TEM for misfit (NASA)	Molar volume	$G_{el},R_{coh\toincoh}$

Thermodynamics calibration and validation

1. Atom Probe Tomography

- Prior literature data
- Current program data

2. SEM + EDS



Ni-Al-Cr Ternary: APT Experiments (Literature Data)



Experimental data from: C.K. Sudbrack, R. D. Noebe, and D. N. Seidman. 2007. Compositional pathways and capillary effects during isothermal precipitation in a nondilute Ni-Al-Cr alloy. *Acta Materialia*. 55: 119-130.



APT image for Ni-5.2Al-14.2Cr (at%) after aging for 4hrs at 600°C



Database Comparison : Ni-Al-Cr Ternary Equilibrium and Nucleation



Overall : Ni-5.2Al-14.2Cr (at%)

- 10/

		60092			at	%		APB	600°C	%γ
A Calling to 18 Sec.		600 C		Ni	AI	Cr	RMS	(J/m ²)	misfit %	fraction
			APT	81.26	3.13	15.61				
a man think a hard		Equilibrium	2σ	0.18	0.08	0.18	0.14			
H WALL P			Ni 5,6	81.40	3.64	14.96	0.58			
and a start of	γ	composition	Ni 7	81.34	3.62	15.04	0.53			
and the second second		composition	TCNI1	81.17	3.93	14.90	0.76			
A starting of the			Ni-NIST	80.60	5.20	14.20	1.77			
11 37 Stores			APT	76.53	16.69	6.77		0.19	-0.72	15.4
		γ' equilibrium composition	2σ	0.50	0.44	0.30	0.38	0.01		0.4
			Ni 5,6	74.98	16.23	8.80	1.47	0.19	-0.6	12.4
19 Y			Ni 7	74.97	16.21	8.82	1.49	0.19	-0.6	12.4
			TCNI1	75.59	14.55	9.57	2.49	0.18	-0.8	12.3
where imported data			Ni-NIST	75.96	12.25	11.79	4.74	0.15	-0.9	0.0
om: C.K. Sudbrack.	γ		APT	72.40	18.30	9.30		0.16	-0.2	
. D. Noebe, and D. . Seidman. 2007.		Critical w?	2σ	2.20	1.80	1.40	1.61	0.03		
		oniicaiγ-	Ni 5,6	74.91	17.37	7.71	1.30	0.20	-0.5	
cta Materialia. 55:		composition	Ni 7	74.91	17.40	7.69	1.30	0.20	-0.5	
19-130.		composition	TCNI1	75.36	16.43	8.21	1.53	0.20	-0.6	
			Ni-NIST	No pi	recipita	tion				

Ni5, Ni6, Ni7: Saunders, N. Ni-DATA. ThermoTech Ltd., Surrey, U.K. TCNI1: Dupin N, Ansara I, Sundman B. Calphad 2001;25:279

Ni-NIST: Kattner UR. In: Turchi PEA, Gonis A, Shull RD, editors.CALPHAD and Alloy Thermodynamics. Warrendale (PA):TMS; 2002.



Database Comparison: Ni-Al-Cr-Re/W Quaternary Equilibrium



Ni-10Al-8.5Cr-2Re (at%)

Experimental data from: K.E. Yoon, R. D. Noebe, and D. N. Seidman. 2007., *Acta Materialia*. 55: 1145-1157.

Ni-10Al-8.5Cr-2W (at%)

Experimental data from: Sudbrack, C.K., D. Isheim, R. D. Noebe, N.S. Jacobson, and D. N. Seidman. 2007., *Microscopy and Microanalysis*. 10: 355-365.

Ni 81.07	Cr	AI			0.		
81.07			Re	RMS	(J/m²)	misfit %	fraction
01.07	10.04	6.74	2.15				
0.16	0.04	0.08	0.02	0.05			
80.55	8.94	8.35	2.16	1.13			
80.22	8.79	8.87	2.12	1.42			
76.17	4.97	18.05	0.81		0.19	-0.71	24.8
0.26	0.10	0.18	0.04	0.12	0.00		0.2
75.29	5.68	18.80	0.23	0.68	0.20	-0.68	15.9
75.82	6.13	17.65	0.39	0.75		-0.77	7.2
76.33	5.46	16.92	1.29		0.17		20.3
0.30	0.16	0.26	0.08	0.18	0.00		0.2
75.22	5.56	18.99	0.23	1.35	0.20		12.8
	81.07 0.16 80.22 76.17 0.26 75.29 75.82 76.33 0.30 75.22	81.0710.040.160.0480.558.9480.228.7976.174.970.260.1075.295.6875.826.1376.335.460.300.1675.225.56	81.0710.046.740.160.040.0880.558.948.3580.228.798.8776.174.9718.050.260.100.1875.295.6818.8075.826.1317.6576.335.4616.920.300.160.2675.225.5618.99	81.0710.046.742.150.160.040.080.0280.558.948.352.1680.228.798.872.1276.174.9718.050.810.260.100.180.0475.295.6818.800.2375.826.1317.650.3976.335.4616.921.290.300.160.260.0875.225.5618.990.23	81.0710.046.742.150.160.040.080.020.0580.558.948.352.161.1380.228.798.872.121.4276.174.9718.050.811.420.260.100.180.040.1275.295.6818.800.230.68875.826.1317.650.390.7576.335.4616.921.290.180.300.160.260.080.18	81.0710.046.742.15I0.160.040.080.020.0580.558.948.352.161.1380.228.798.872.121.4276.174.9718.050.810.190.260.100.180.040.120.0075.295.6818.800.230.680.2076.335.4616.921.290.170.170.300.160.260.080.180.00	81.07 10.04 6.74 2.15 I.I I.II 0.16 0.04 0.08 0.02 0.05 III 80.55 8.94 8.35 2.16 1.13 IIII 80.22 8.79 8.87 2.12 1.42 IIIII 76.17 4.97 18.05 0.81 0.19 -0.71 0.26 0.10 0.18 0.04 0.12 0.00 IIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIIII

800℃				at%			APB	300℃	%γ
		Ni	Cr	AI	W	RMS	(J/m ²)	misfit %	fraction
	APT	81.31	5.83	11.52	1.34				
γ equilibrium	2σ	0.07	0.04	0.05	0.02	0.04			
mainx	Ni 7	81.44	6.22	10.53	1.80	0.67			
composition	Ni-NIST	80.48	8.57	8.84	2.11	2.26			
or oquilibrium	APT	76.30	17.00	3.91	2.80		0.21	-0.49	37.9
γ equilibrium precipitate composition	2σ	0.08	0.07	0.04	0.03	0.05	0.00		0.0
	Ni7	75.39	18.00	4.20	2.42	0.64	0.22	-0.36	32.1
	Ni-NIST	75.87	16.61	6.42	1.09	1.77		-0.61	18.7

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Long-Term Aging Experiments









ME3 1093°C/1000hrs : APT + EDS

		γm	atrix	(at 10)93°(C) co	mp	ositi	on,	at%		%γ'
	Ni	Al	Cr	Со	Ti	Мо	W	Nb	Та	С	RMS	fraction
APT	44.9	6.7	17.5	21.7	3.5	3.4	1.0	0.5	0.5	0.04		24.8
2σ	0.04	0.02	0.03	0.03	0.01	0.01	0.01	0.01	0.01	0.002		1.5
EDS	46.1	6.6	16.6	22.7	3.3	3.0	1.3*	0.6	-	-		
Ni 5,6	45.7	5.6	19.7	22.1	2.4	3.1	0.7	0.3	0.3	0.01	0.93	30.7
Ni 7	46.4	6.0	18.5	21.8	2.7	3.0	0.7	0.4	0.5	0.01	0.54	25.5
Pan- Nickel	44.3	5.3	20.3	23.0	2.4	2.9	0.8	0.4	0.5	0.05	1.19	32.3
NIST-Ni	44.7	5.1	19.9	23.8	2.4	3.2	0.8		0.2		1.44	29.4

Alloy 10 1093°C/1000hrs : EDS

			Compositions, at%								
		Ni	Al	Cr	Co	Ti	Мо	W	Та	Nb	RMS
	EDS	53.8	7.0	13.4	17.5	3.2	2.3	1	.6	1.13	
	Ni 5,6	50.8	5.74	17.60	17.76	2.61	2.30	2.54	0.14	0.50	1.75
γ	Ni 7	51.1	5.97	16.97	17.72	2.76	2.21	2.48	0.20	0.63	1.49
	Ni- NIST	51.3	5.44	17.00	18.60	2.58	2.31	2.66	0.10	-	1.68
	EDS	62.3	13.0	4.0	10.9	7.2	0.6	0	.9	1.3	
	Ni 5,6	62.2	12.02	2.82	11.01	8.42	0.24	0.93	0.49	1.89	0.80
γ'	Ni 7	64.1	12.11	2.80	10.53	8.64	0.23	0.91	0.40	1.73	0.83
	Ni- NIST	66.8	13.5	1.35	7.54	9.62	0.13	0.37	0.68	-	1.93

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High Temperature Misfit Estimate



Predictions of misfit model



- Combination of NI7 thermodynamic database and misfit model gives right ranking of relative misfit in alloys
- Elastic energy term to be incorporated in thermodynamic database to account for lattice misfit.







Experiments	CALPHAD Fundamental Databases	Material Kinetic Model Parameters
Equilibrium Age + APT (literature data, QuesTek) + EDS (NASA)	Thermodynamics ∆E	
Diffusion Couple + Microanalysis (NASA)	Mobility D _{scale}	
SSDTA (OSU) + APT (QuesTek)	Tar and	$\sigma_{coh}, G_{el} (est.)$
Coarsening Age+SEM/TEM for γ' size and fraction (NASA)		$\sigma_{\text{incoh}},M_{O}$
XRD, TEM for misfit (NASA)	Molar volume	$G_{el},R_{coh\rightarrow incoh}$

Mobility calibration and validation

1. Diffusion Couples + Microanalysis



Focused Experimentation (NASA)



For NIST mobility database validation/calibration



The vacuum/inert gas hot press unit to be used to fabricate the superalloys / Ni couples



Schematic of the typical diffusion couple stacking that will be used for this study

Annealed at 927°C or 1093°C for 100 or 300 hrs



ME3/Ni diffusion couple held at 1093°C/100 hrs



 $\gamma + \gamma' + MC$ I < γ



In order to compare experimental diffusion couple results with DICTRA simulations, the average Matano interface (defined as the interface across which equal number of atoms have crossed in both directions) is equated to the zero-point grid position of the calculated profiles.

The adjoining figure shows the determined average Matano interface and its position with respect to the original interface in the diffusion couple. The Matano interface is now taken as the zeropoint for comparison with calculated profiles



Comparison with DICTRA results: ME3/Ni 1093°C for 100 hrs



Use measured compositions (average over first 50-100 μm of superalloy side)
No diffusion through γ'; acts only as sink or source of solute for diffusion.



Solid lines: DICTRA results Solid circles: Measured data

- Nb, Al, and Ti diffusion is less than predicted
- Cr diffusion is more than predicted





The mobility terms in the NIST mobility database were changed slightly for Cr, Nb, and Ti for better agreement between the DICTRA simulations and the experimentally measured diffusion couples.

MQ(Cr in Ni)	<u>Original Value</u> -287000+R*T*LN(4.3E-4)	Modified Value -287000+R*T*LN(6E-4)
MQ(Nb in Ni)	-255333+R*T*LN(7.6E-5)	-255333+R*T*LN(4E-5)
MQ(Ti in Ni)	-256900+R*T*LN(8.6E-5)	-256900+R*T*LN(6E-5)

The comparison between the simulations using the original database and the modified database is shown in the next slides for some conditions. The agreement with experiment is much better for the simulations using the modified database.



Comparison with DICTRA results: ME3/Ni 1093°C for 100 hrs



Solid lines: DICTRA results Solid circles: Measured data



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Comparison with DICTRA results: ME3/Ni 1093°C for 300 hrs



Solid lines: DICTRA results Solid circles: Measured data



Modified database



Comparison of measured phase boundaries with DICTRA simulations





DICTRA simulations of ME3/Ni 1093°C for 100 hrs:

- Use measured compositions
- No diffusion through γ '; acts only as sink or source of solute for diffusion.
- Predicted phase γ+ γ'+MC / γ phase boundary is in excellent agreement with experimental measurement.





Experiments	CALPHAD Fundamental Databases	Material Kinetic Model Parameters
Equilibrium Age + APT (literature data, QuesTek) + EDS (NASA)	Thermodynamics ∆E	
Diffusion Couple + Microanalysis (NASA)	Mobility D _{scale}	
SSDTA (OSU) + APT (QuesTek)		σ _{coh} , G _{el} (est.)
Coarsening Age+SEM/TEM for γ' size and fraction (NASA)		σ _{incoh} , M _O
XRD, TEM for misfit (NASA)	Molar volume	$G_{el},R_{coh\toincoh}$

Non-isothermal Precipitation

calibration and validation

- 1. SSDTA (onset of nucleation)
- 2. APT (non-isothermal microstructures)



Single Sensor Differential Thermal Analysis: Calibration of Non-Isothermal Nucleation



Single Sensor Differential Thermal Analysis (SSDTA) is a novel technique developed at OSU for studying phase transformations in materials.

Procedure:

- Process SSDTA measured temperature profiles to obtain nucleation onset temperature (T_{onset}).
- Calculate material compositions relevant to the γ' precipitation from the matrix γ phase - remove high temperature phases (borides, carbides and undissolved γ')
- Perform PrecipiCalc simulations using NI7 and NIST mob database, with estimated surface energy.
- Compute time (or temperature) evolution of molar enthalpy.
- Compute dH/dT.
- Determine onset temperature where dH/dT changes by more than 10%.



SSDTA results and PrecipiCalc predictions





- Optimized interfacial energies for ME3, Alloy 10 and RR1000 are similar and agree with the value reported by Sudbrack et. al.¹ (~0.022-0.023 J/m2).

1: C.K. Sudbrack, R. D. Noebe, and D. N. Seidman. 2007. *Acta Materialia*. 55: 119-130.



γ ' microstructure in SSDTA samples

LSHR -2Ar

RR1000-3He



41 nm

45.6 nm

20.5 nm



40nm

 γ ' mean size comparison HR SEM Alloy LEAP APT PrecipiCalc Predictions ME3 -1 Ar 40-60 nm 53.8 nm

50.1 nm

31.8 nm



20-50 nm

Characterization of γ ' microstructure in ME3-1Ar SSDTA sample

Particle size histogram of PrecipiCalc and HR SEM in LSHR-1Ar SSDTA sample



γ ' microstructure in furnace cooled samples





Comparison shows good agreement between experimental SEM (bars) and *PrecipiCalc* simulated (converted to 2D, solid line) and normalized particle size distributions for LSHR furnace cooled sample. 2D coarsening size distribution is shown in a dashed line

γ ' mean size comparison

Alloy	HR SEM	<i>PrecipiCalc</i> Predictions
ME3	430 nm	348 nm
LSHR	408 nm	402 nm
RR1000	376 nm	396 nm

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Experiments	CALPHAD Fundamental Databases	Material Kinetic Model Parameters		
Equilibrium Age + APT (literature data, QuesTek) + EDS (NASA)	Thermodynamics ∆E	18 J		
Diffusion Couple + Microanalysis (NASA)	Mobility D _{scale}			
SSDTA (OSU) + APT (QuesTek)	Card and	$\sigma_{coh}, G_{el} (est.)$		
Coarsening Age+SEM/TEM for γ' size and fraction (NASA)		σ _{incoh} , M _O		
XRD, TEM for misfit (NASA)	Molar volume	$G_{el},R_{coh\toincoh}$		

Coarsening calibration and validation

1. SEM + TEM for γ ' size and fraction

Quester"

Coarsening Experiments



ME3, LSHR and Alloy 10:

- Supersolvus treated at 1193°C for 1hr + WQ
- Aged:
 - 1093°C/20hrs and 1000hrs
 - 927°C/1000hrs
 - 760°C/1000hrs



PrecipiCalc modeling of coherent intragranular γ' is ongoing

Experimental measured γ ' mean equivalent diameters (μ m) of isothermally aged samples.

Aging Exp	ME3		LSHR		Alloy 10		
Conditions	Ξxμ	G	GB	G	GB	G	GB
Before Aging	TEM	0.044	-	0.028	-	0.044	-
1093C-20h	SEM	0.510	0.810	0.504	0.984	0.546	1.084
1093C-1000h	SEM	1.380	2.840	1.420	2.996	0.894	2.618
927C-1000h	SEM	0.584	0.758	0.598	0.954	0.670	1.224
760C-1000h	TEM	0.058	-	0.062	-	0.062	-

SEM image of ME3 super-solvus + aged at 1093°C for 1000hrs. Intragranular and GB γ ' are represented as black and grey.



Particle Size Distribution of Intra-grain y











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Summary



- Focused experiments performed at NASA GRC for calibration of databases and precipitation models.
- Measured phase compositions show best agreement with Thermotech Ni7 thermodynamic database.
- Prediction of high misfit in high-refractory 3rd generation alloys supported by microstructural evidence: XRD for confirmation.
- Comparison of diffusion couple experiments with DICTRA predictions indicate NIST mobility database (in combination with NI7) gives sufficient accuracy to support *PrecipiCalc* simulations.
- SSDTA measurement of critical nucleation under-cooling defines reasonable values of coherent interfacial energies.
- PrecipiCalc simulations of non-isothermal precipitation gives good agreement with observed particles sizes and compositions.

Accuracy of predictions validated so far indicates that available fundamental databases and precipitation models offer sufficient fidelity for effective application of AIM methodology to 3rd generation aeroturbine disc alloys

Фиеб**Т**ек



- John Gayda and Tim Gabb of NASA GRC.
- Jeff Simmons and Chris Woodward of AFRL
- David Furrer, Rob Mitchell and Mark Hardy of Rolls-Royce Corporation
- Carelyn Campbell of NIST
- Boian Alexandrov of Ohio State University
- Eugine Kang of Northwestern University
- David Siedman of Northwestern University