Microstructural Characterization and Modeling of SLM Superalloy 718

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Motivation for Microstructural Modeling

- A number of modeling tools are being developed to support rapid flight certification of SLM 718 components for the SLS engine under NASA's Material Genome Initiative program.
- Post-processing heat treatment of SLM 718 components is required for consolidation and to obtain optimal mechanical properties.

Background

- Commercial software packages based on CALPHAD-based methods have been developed to predict microstructure.
- Accurate microstructural measurements are needed to "tune" these models, i.e. compare, calibrate and then validate model predictions to experimental values.

Objective

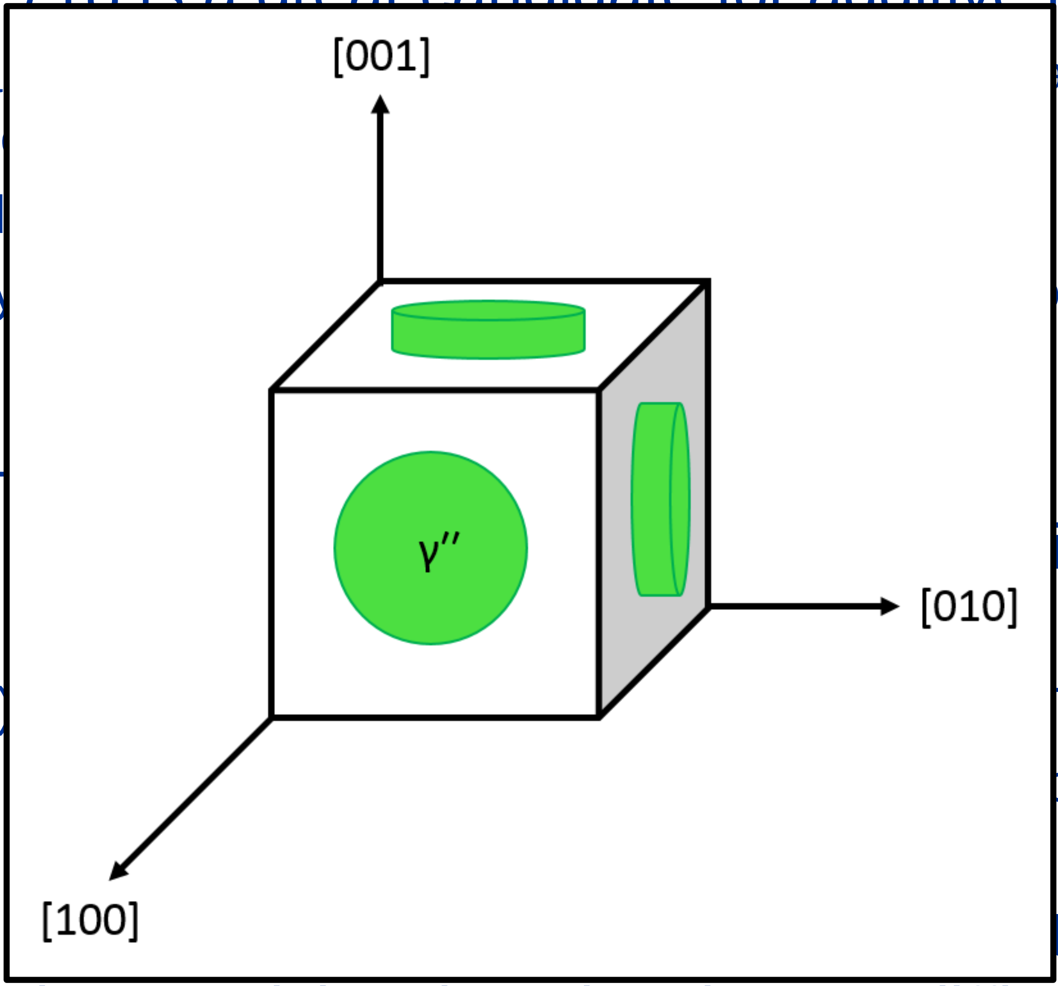
- To obtain accurate microstructural measurements that will enable a model that can predict microstructure well over a range of relevant heat treat conditions

Approach

- $\frac{1}{2}$-inch diameter rods of superalloy 718 were fabricated using SLM on MSFC's M2 Concept Laser.
- All section pieces were stress relieved at high temperature, cut from build plate, then hot isostatic pressed (HIP).
- The thermal history and alloy composition were used as inputs into the Pandat 2013 precipitation models.
- **Detailed microstructural measurements** of the precipitates were performed to verify the model predictions.

Superalloy 718

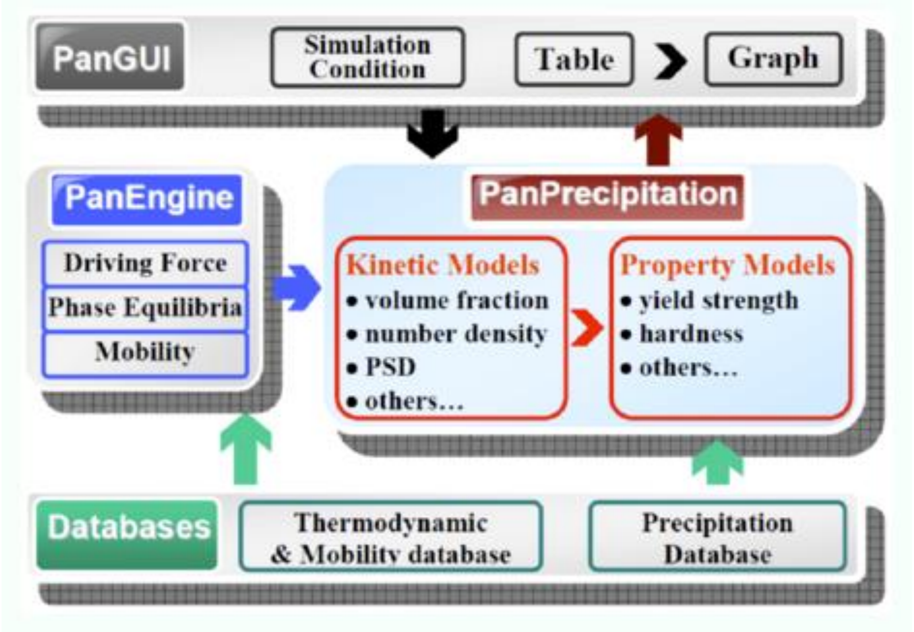
- Superalloy 718 is a great candidate for additive manufacturing
  - Used in a ... ace applications for decad...
  - Has good ...
  - Thermody... ped
- Superalloy ... 19Cr-5Nb-3Mo-1Co-1...
- Superalloy ... itation phases.
  - γ' (Ni_3(Al, ...
  - γ'' (Ni_3Nb) ... Three variants
  - δ (Ni_3Nb) ... cipitates along GB's
- Due to the ... tates, accurately characterizing them has been a difficult endeavor.



Computherm Pandat Modeling

wt.%	Ni	Al	Co	Cr	Fe	Mo	Nb	Ti	W
SLM 718	53.19	0.5	0.09	18.1	18.9	3.1	5.1	1.0	0.02

Computherm Pandat 2013 PanPrecipitation Module



- First precipitation package to allow users to apply thermal history to an initial microstructure, as well as standard homogenized alloy chemistry
- Computherm has worked closely with the Air Force Research Laboratory (AFRL) on superalloy 718 database development: PanNi_MB_2013 is their combined thermodynamic / kinetic databases.

Sample Preparation of SLM Superalloy 718

- Superalloy 718 specimens were fabricated by SLM on MSFC Concept Laser tool.

Thermal post-processing steps – ASTM standard

Reduces thermal stresses from SLM	Removes porosity	Promotes macro chemical uniformity	Promotes micro chemical uniformity	Precipitation hardening
Stress relief (1)	HIP (2)	Homogenization (optional) (3)	Solution heat treatment (4)	Aging (5)
Under argon: 1950°F / 1.5hr + gas quench		Under vacuum: 2125°F / 1hr + gas quench	Under vacuum: **1950 or 1850 or 1700 °F** 1hr + gas quench	Two Step Age 1 or Age 2

Age 1: 1325°F/10hr + FC to 1150°F + 1150°F/≈6hr (until total time is 18hr)
Age 2: 1400°F/10hr + FC to 1200°F + 1200°F/≈8hr (until total time is 20hr)

Set 1 - Homogenized

Z41: SR + HIP + Homo + Sol 1950 + Age 1
Z18: SR + HIP + Homo + Sol 1850 + Age 1
Z1: SR + HIP + Homo + Sol 1850 + Age 2
Z25: SR + HIP + Homo + Sol 1700 + Age 1

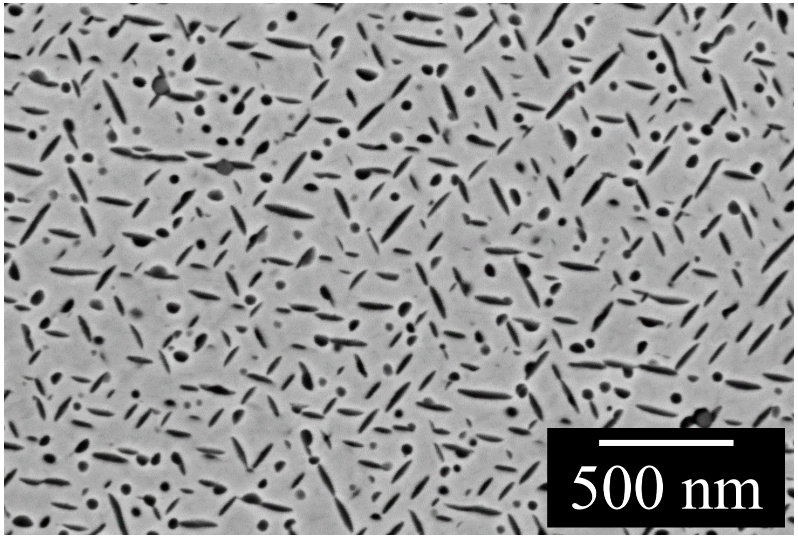
Set 2 – Not homogenized

Z8: SR + HIP + Sol 1850 + Age 1
Z3: SR + HIP + Sol 1850 + Age 2
Z27: SR + HIP + Sol 1700 + Age 1

Microstructural Characterization – Precipitates New Technique: HR-SEM

- New high resolution SEMs allow for direct imaging of γ'/γ'' precipitates when preferentially etched.
- Imaging at 3kV using a secondary electron detector eliminates sample thickness/overlap problems.
- Using precipitate morphology (Aspect ratio), γ' precipitates can be separated from γ''. (Orientation dependent).

Etched with a solution of 50mL Lactic Acid 30mL Nitric Acid and 2mL HF

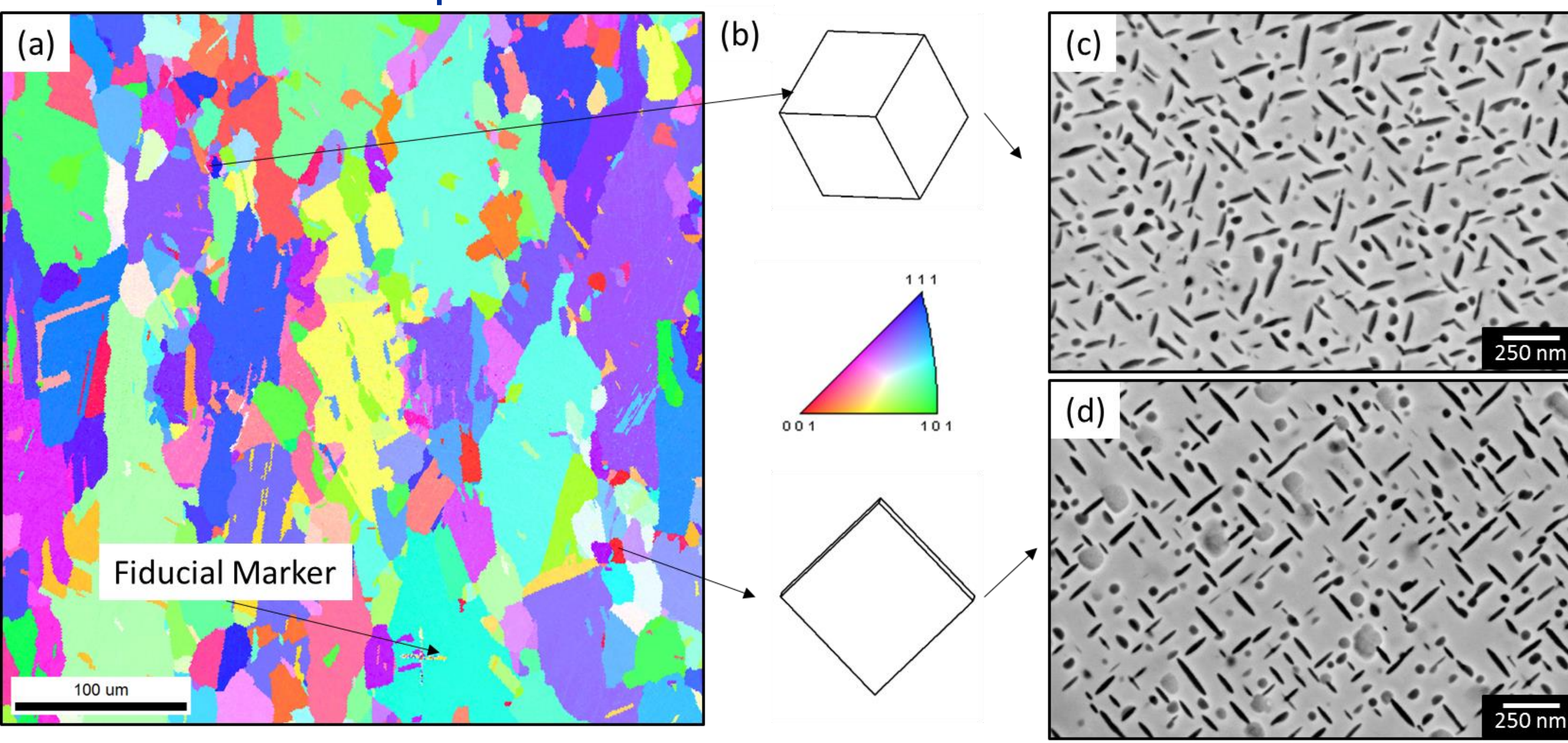


Z1 – Age 2

Microstructural Characterization - EBSD

EBSD Map

SEM - Microstructure



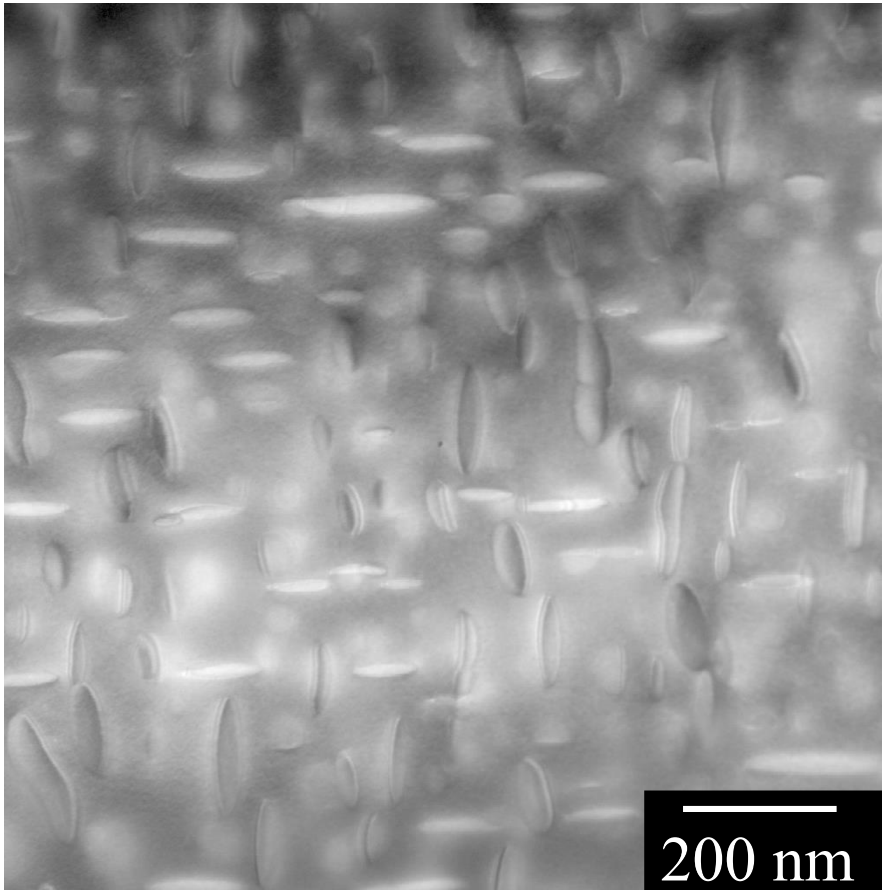
[111] - Volume fraction analysis and γ' size analysis
[001] - γ'' size analysis

Quantifying morphology distinction between precipitates…

Microstructural Characterization – Precipitates (EDS)

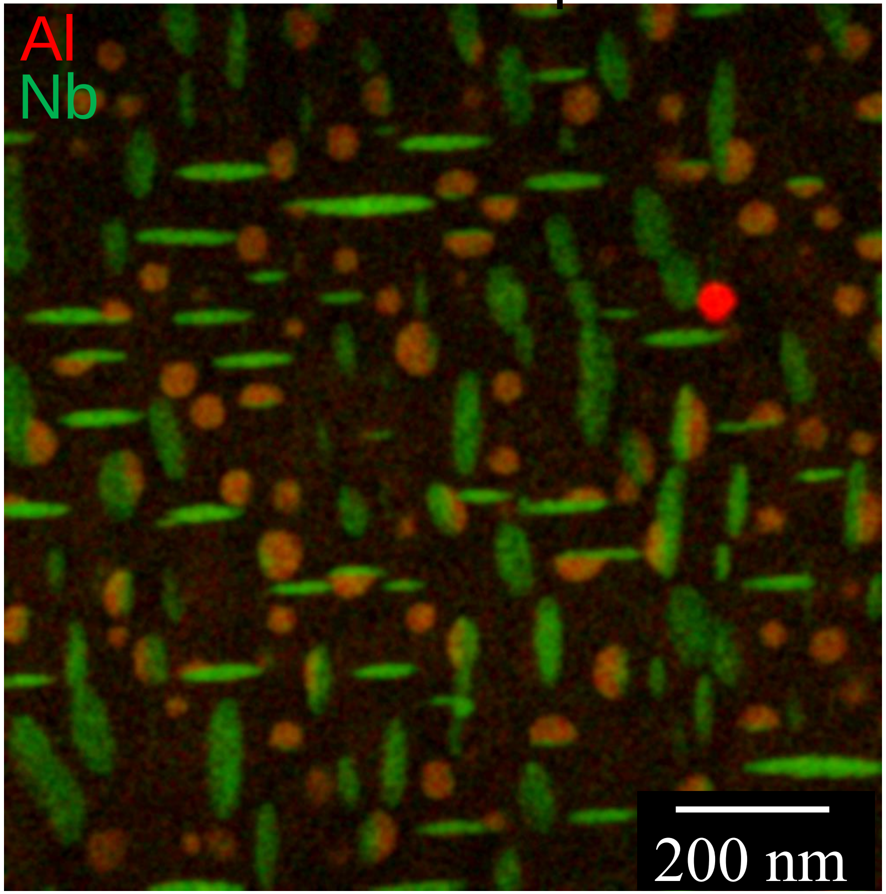
Z1 – Age 2

HAADF



Acquired from a FEI Talos (S)TEM

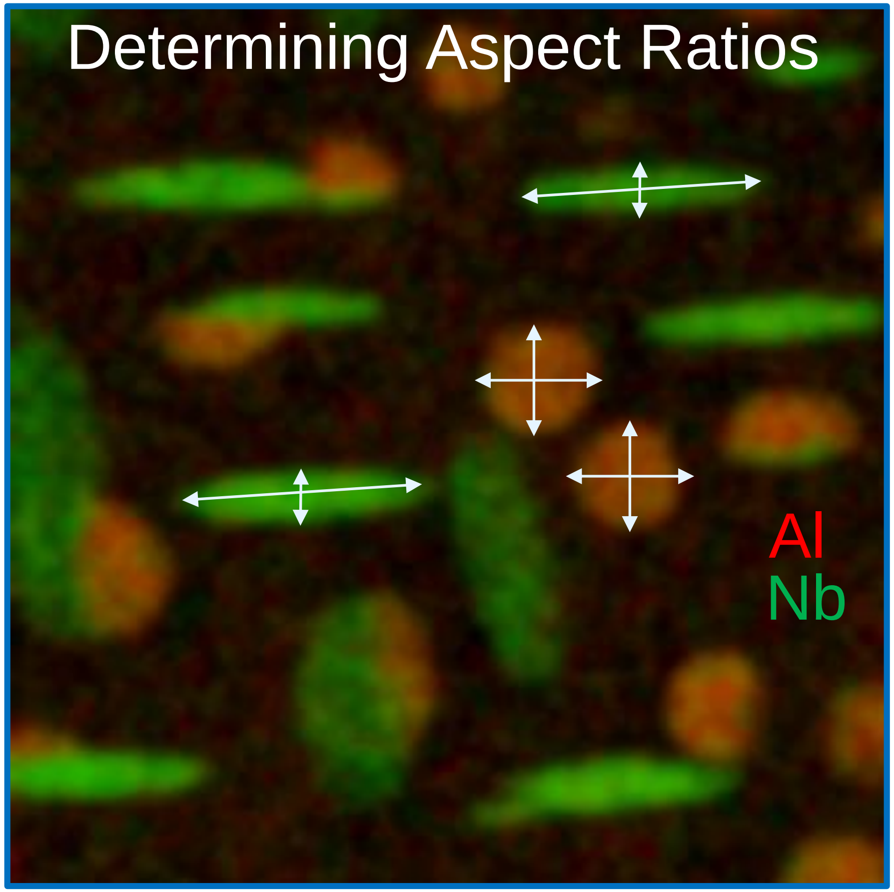
EDS Map



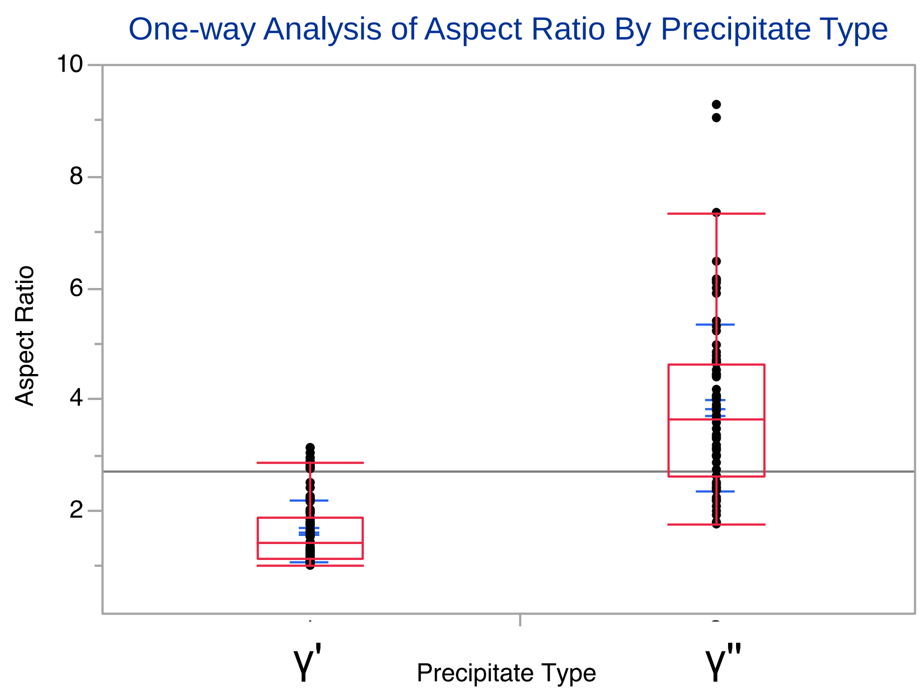
γ' - (Ni_3(Al, Ti)) γ'' - (Ni_3Nb)

Microstructural Characterization – Precipitates (EDS)

Z1 – Age 2

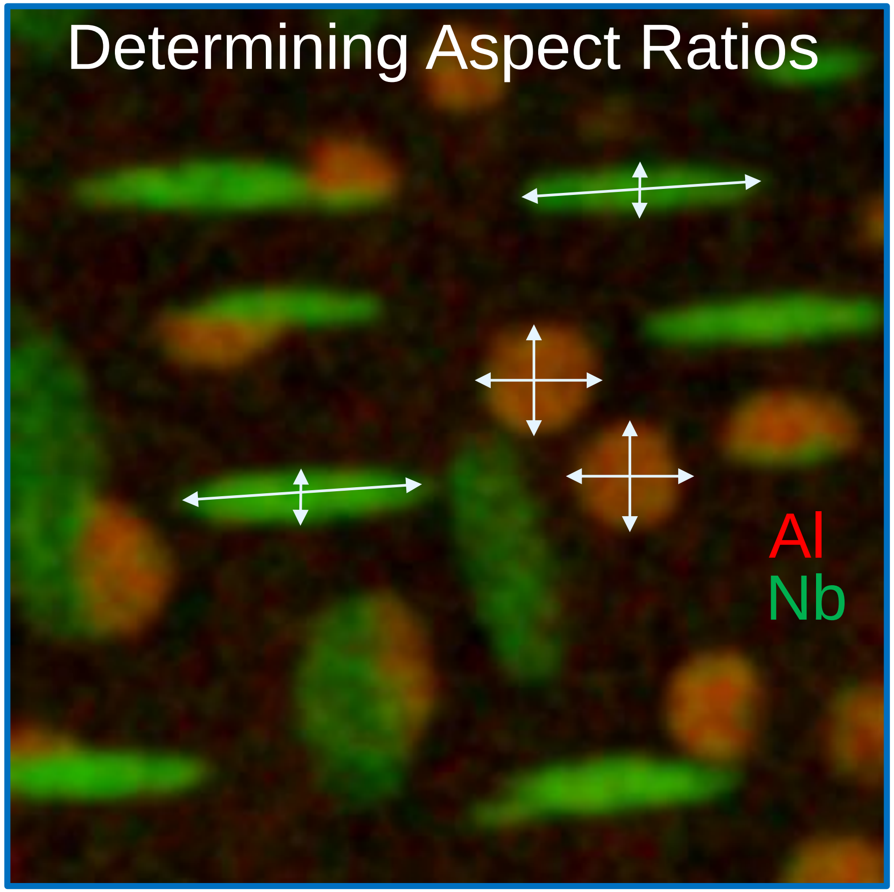


γ' - (Ni_3(Al, Ti)) γ'' - (Ni_3Nb)



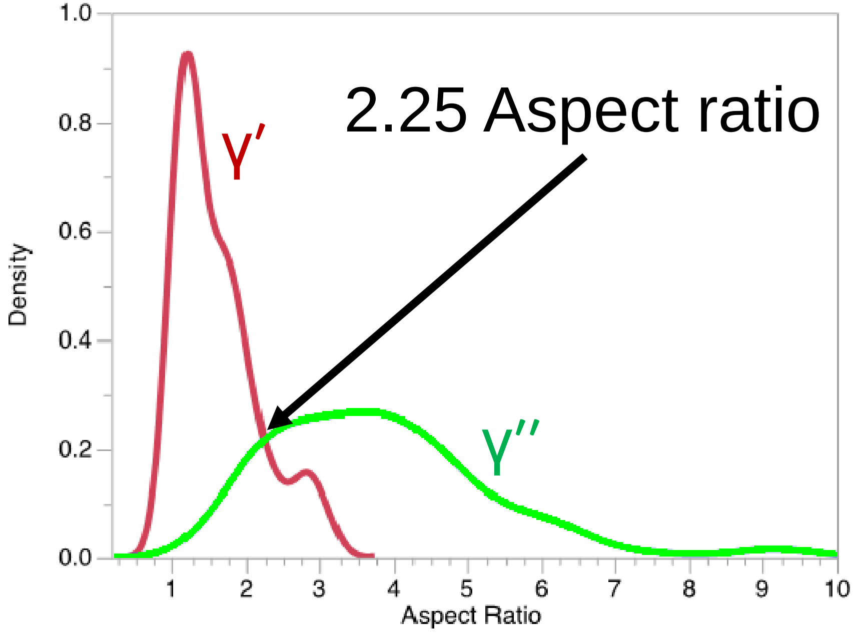
Microstructural Characterization – Precipitates (EDS)

Z1 – Age 2



Note: Presence of composite particles!

Density Map

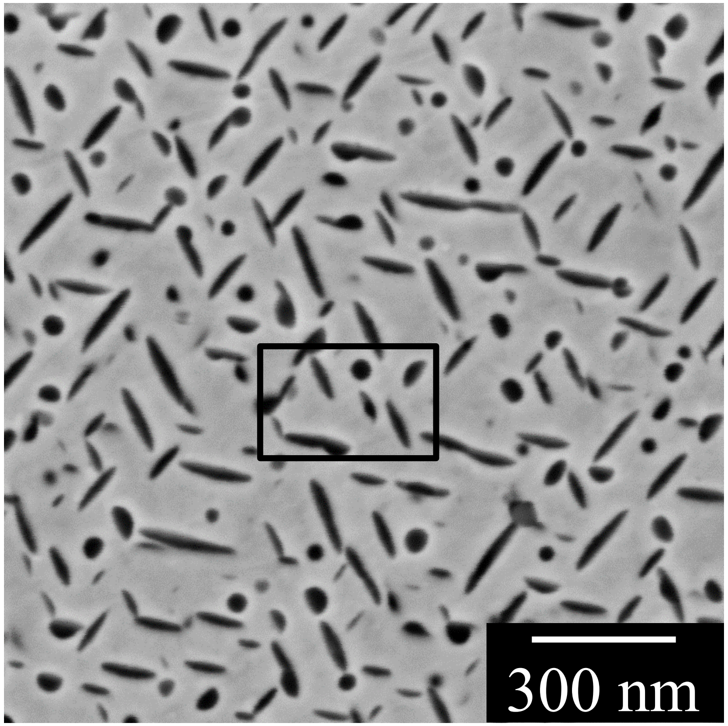


Age 1 cutoff ratio: 1.8
Age 2 cutoff ratio: 2.25

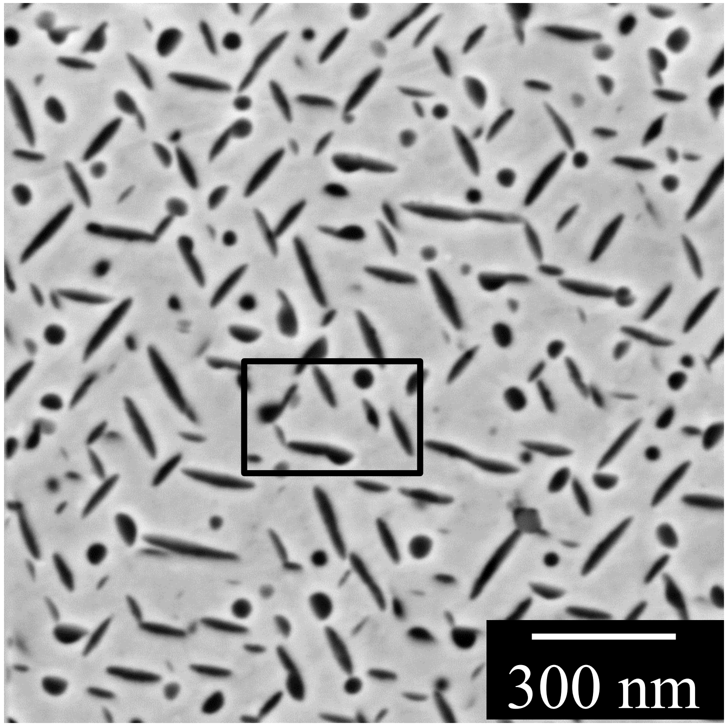
Microstructural Characterization – Precipitates SEM Vibration/Distortion Correction

Z1 – Age 2

No Correction



Scan Corrected

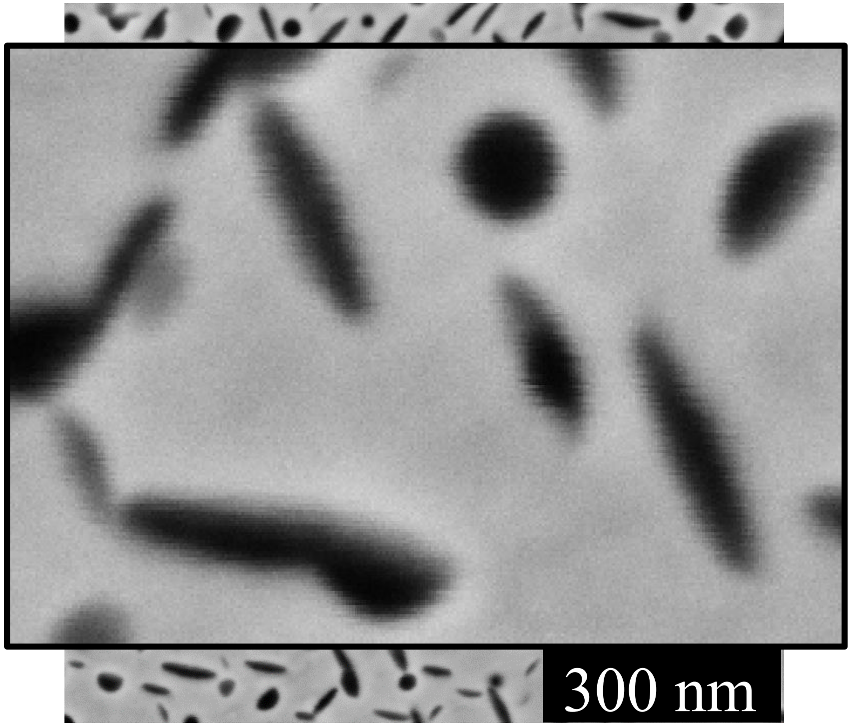


At low magnifications there isn't a noticeable difference…

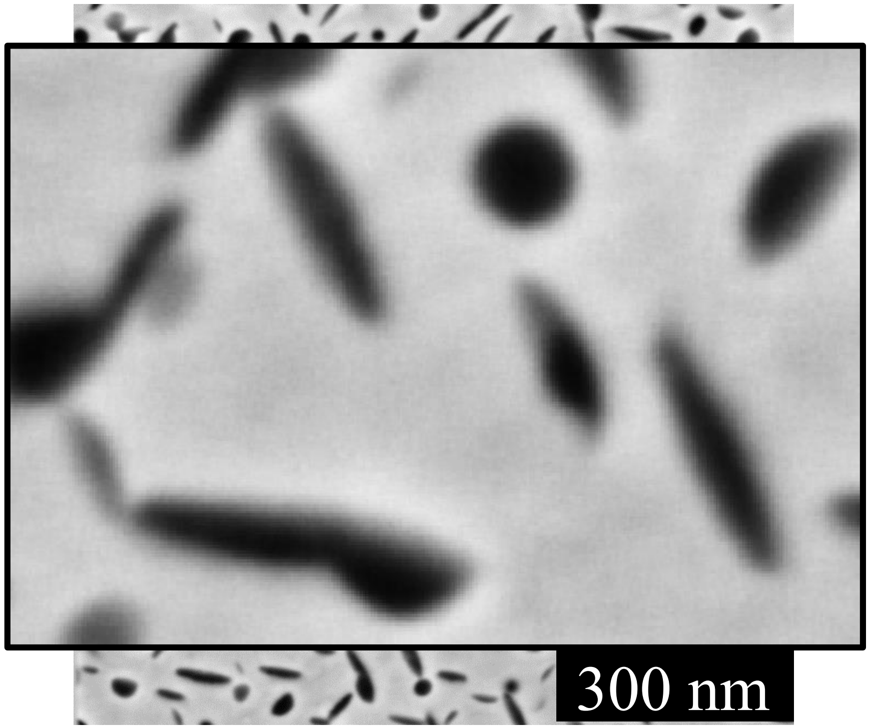
Microstructural Characterization – Precipitates SEM Vibration/Distortion Correction

Z1 – Age 2

No Correction



Scan Corrected

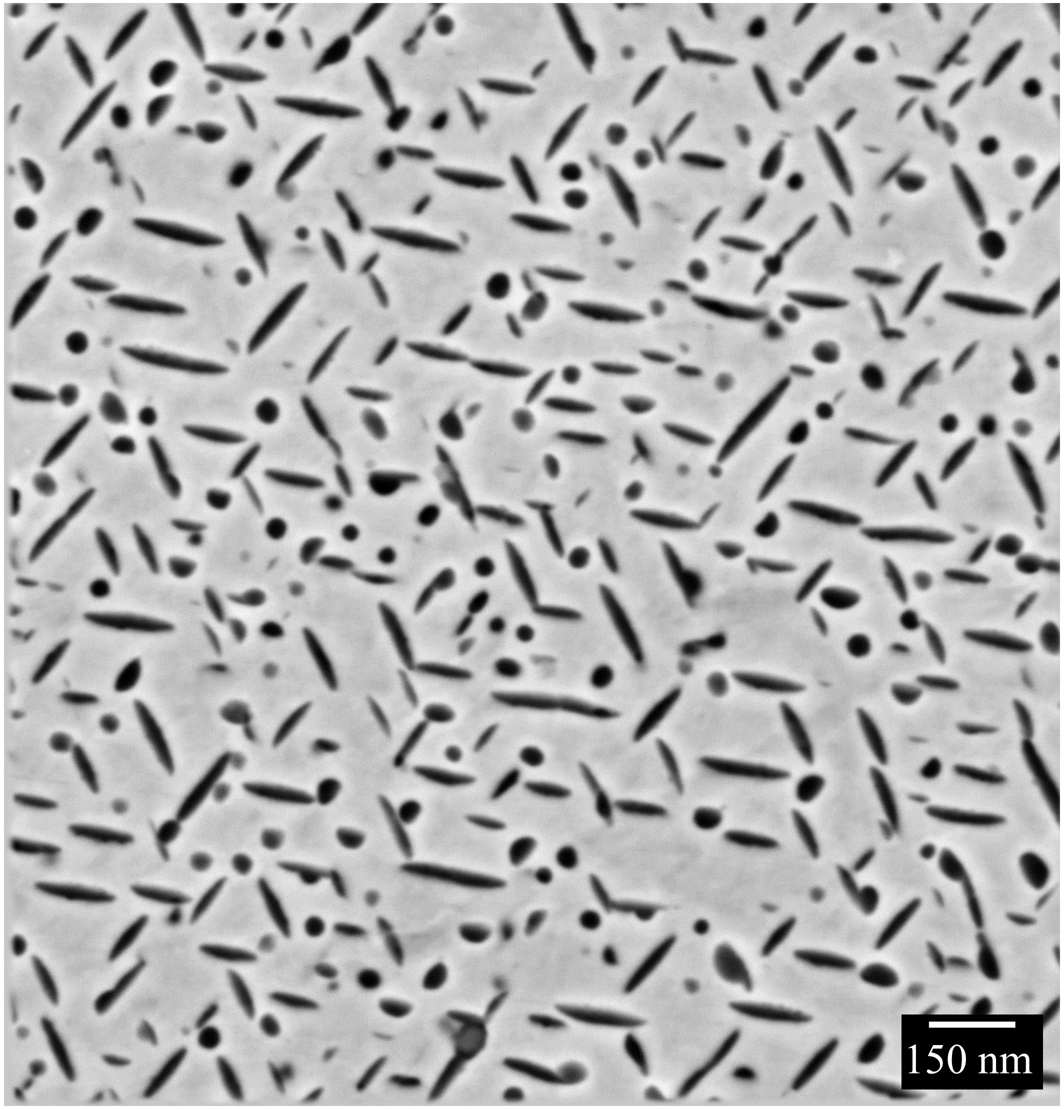


However, at high magnifications it is very noticeable!

* C. Ophus, J. Ciston. Ultramicroscopy 2015

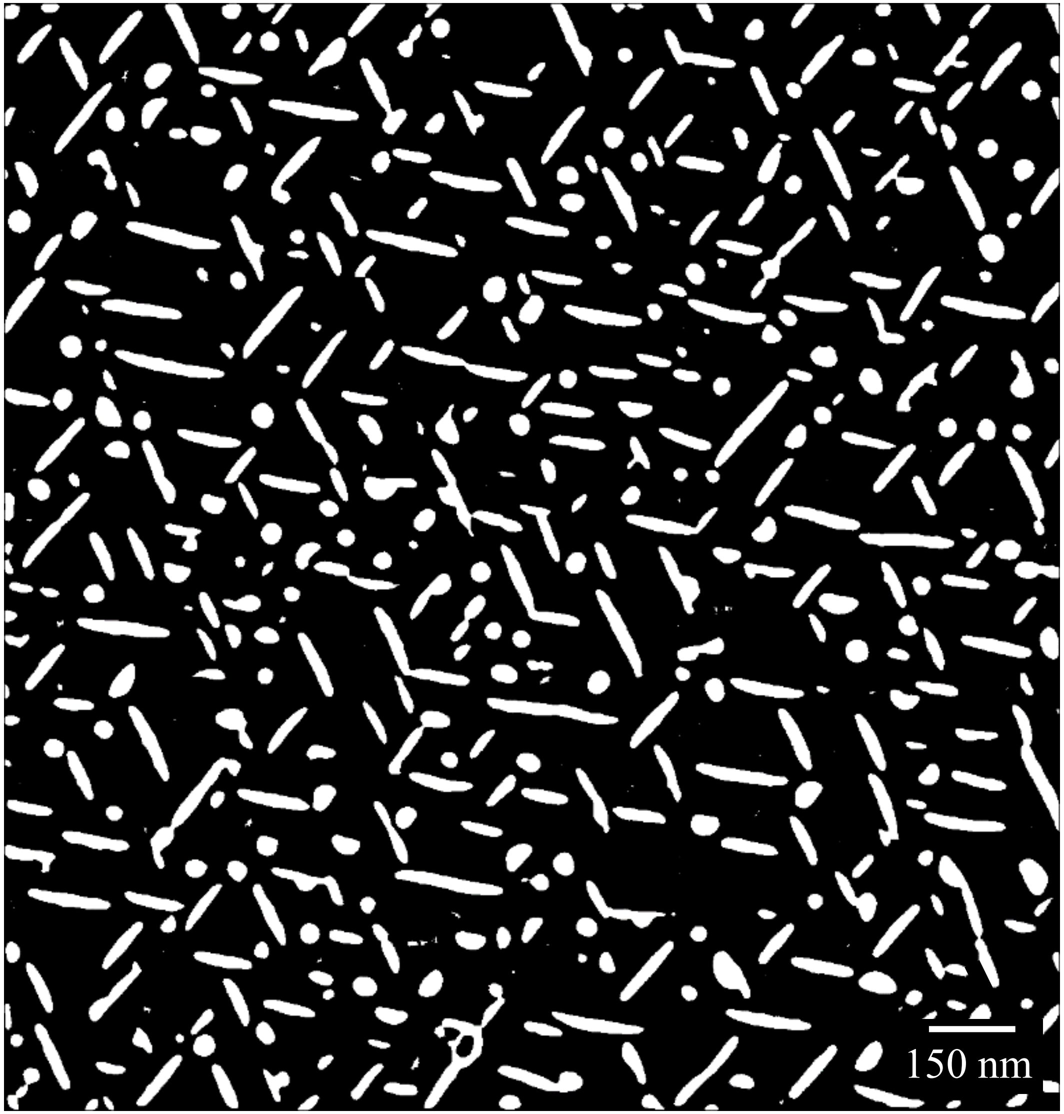
Microstructural Characterization – Precipitates Procedure

Z1 – Age 2



Scan-corrected

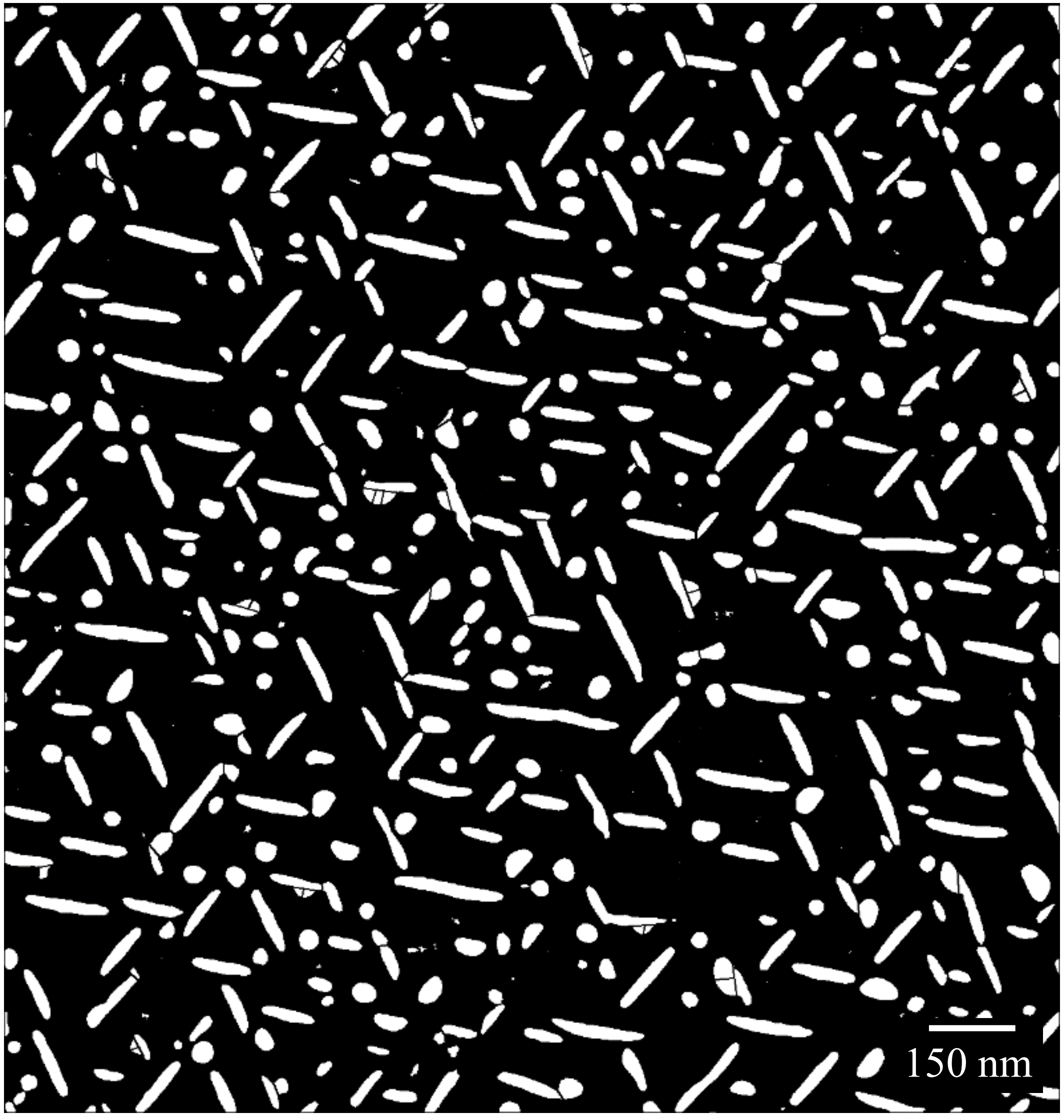
Microstructural Characterization – Precipitates Procedure



Z1 – Age 2

Normalize contrast and brightness: adaptive threshold: make binary (ImageJ)

Microstructural Characterization – Precipitates Procedure

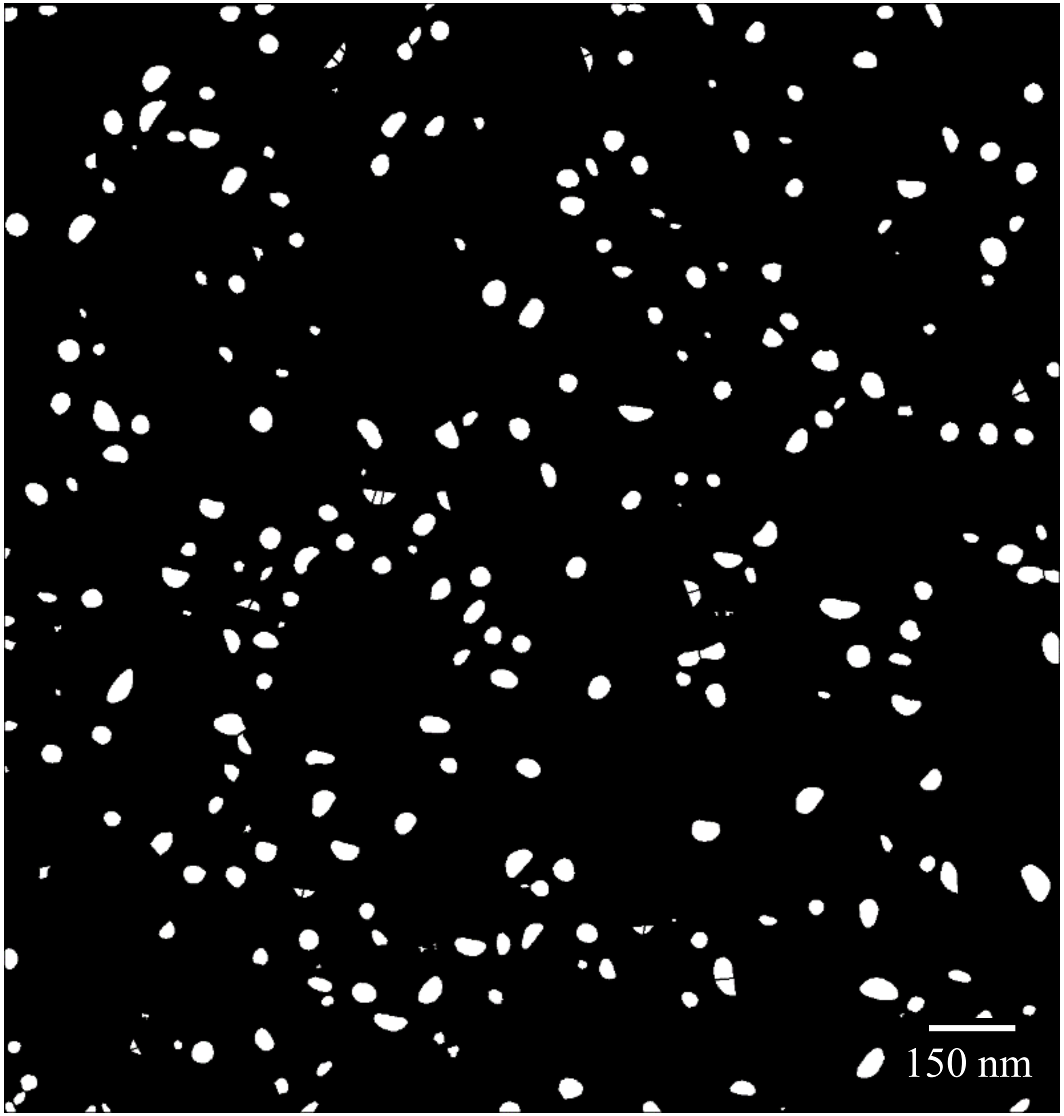


Z1 – Age 2

Watershed by hand (ImageJ)

Currently working on automating this process

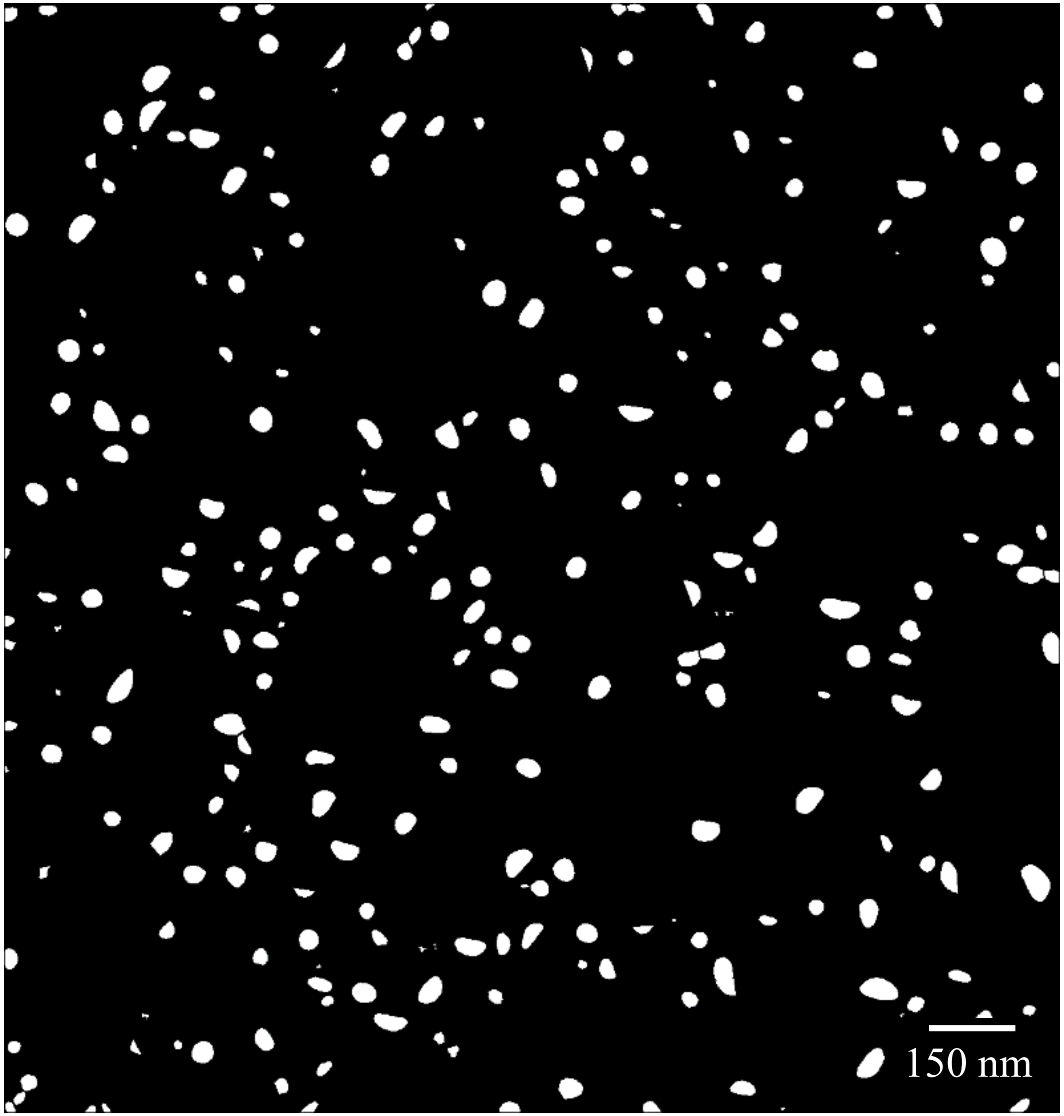
Microstructural Characterization – Precipitates Procedure



Z1 – Age 2

Separate precipitates using aspect ratio cutoffs determined using EDS (ImageJ)

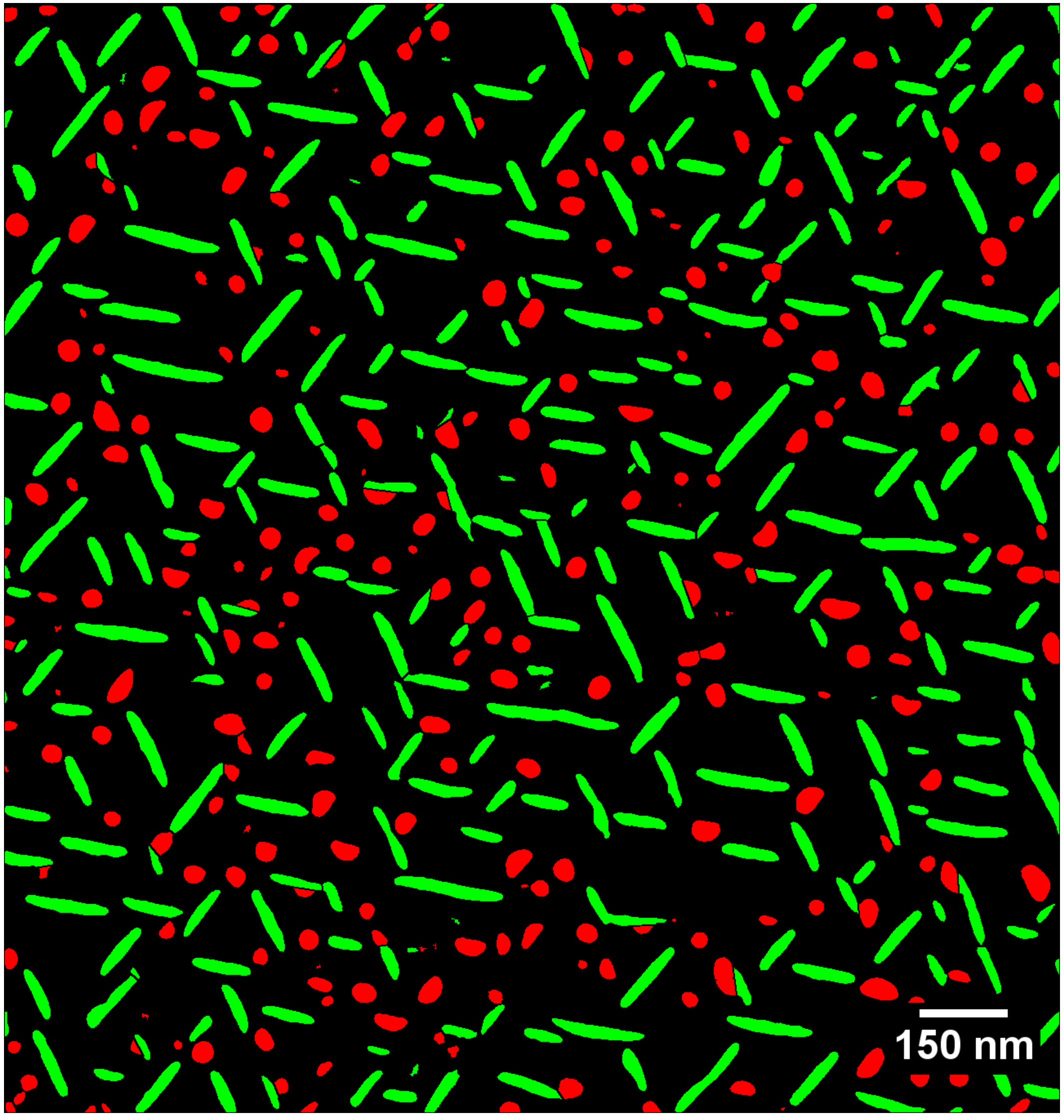
Microstructural Characterization – Precipitates Procedure



Z1 – Age 2

Repair composite γ' precipitates (ImageJ)

Microstructural Characterization – Precipitates Procedure

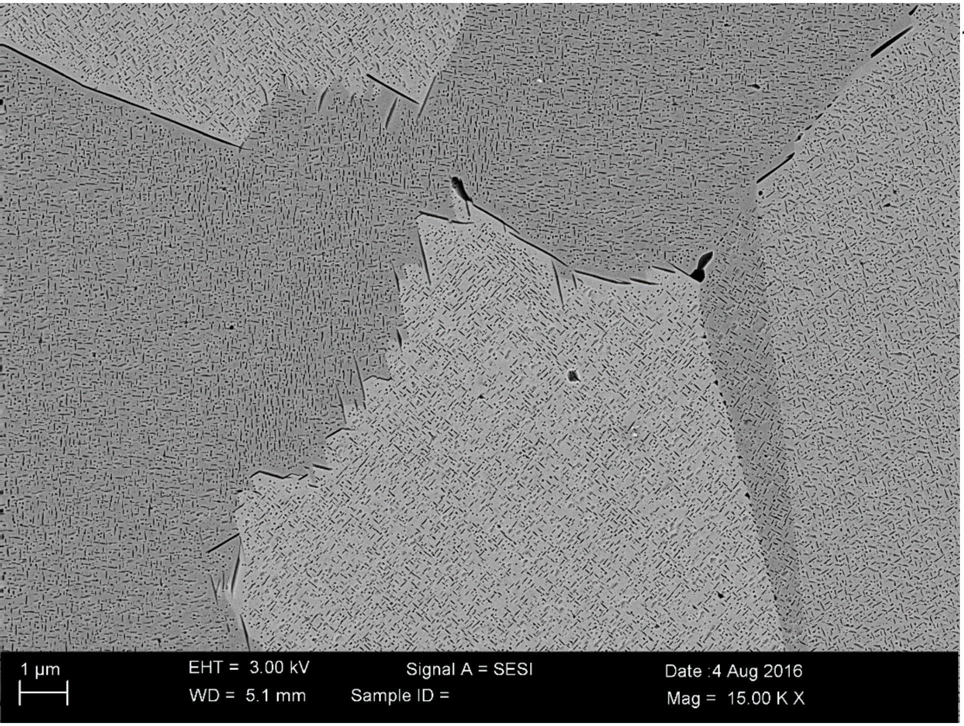


Z1 – Age 2

Same steps for γ'' precipitates. Merge Images. Extract statistics (Size and area fractions for both γ' and γ'') (ImageJ). Repeat until at least >500 particles from each phase is analyzed.

Microstructural Characterization – δ Precipitates

Etched Surface



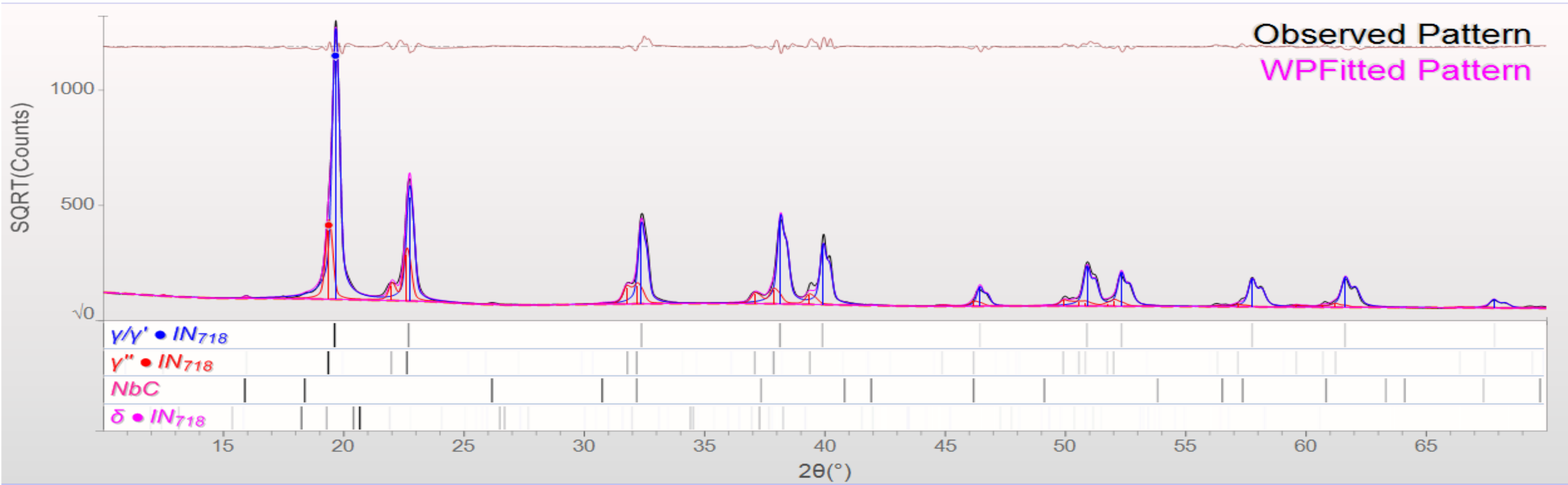
Thresholded Image

Precipitate Parameter	Experimental	Model
δ area percent	.369 ± .24 %	2.0 %
δ average size	.03 ± .01 um^2	
δ feret dia.	.69 ± .15 um	

XRD – Volume Fraction Validation

Phase ID (4)	Chemical Formula	PDF-#	Space Group	Vol% (esd)	Wt% (esd)	a (esd)	c (esd)
γ/γ'	IN_{718}	98-000-1033	cFm-3m (225)	89.34 (3.11)	88.30 (2.26)	3.59588 (17)	3.59588 (17)
γ"	IN_{718}	98-000-1036	tI4/mmm (139)	10.57 (0.63)	11.62 (0.63)	3.61425 (63)	7.42937 (132)
NbC	NbC	04-001-1554	cFm-3m (225)	0.09 (0.02)	0.09 (0.02)	4.43000 (0)	4.43000 (0)
δ	IN_{718}	98-000-1035	oPmmn² (59)	0.00 (0.00)	0.00 (0.00)	5.14100 (0)	4.53400 (0)

Refinement Halted (R/E=13.92), ♣ Round=4, Iter=6, P=31, R=9.0% (E=0.65%, EPS=0.5)



Crystal structure of γ and γ′ phases are to similar to separate in XRD

Precipitate Parameter	SEM	XRD
γ' volume fraction	5.1 ± 0.8 %	N/A
γ" volume fraction	11.1 ± 0.9 %	10.6 ± 0.6
δ volume fraction	.37 ± .24 %	≈ 0 %

Phase Extraction

Precipitate Parameter	Experimental	Phase Extraction
($\gamma'/\gamma''/\delta$) volume fraction	16.6 ± 1.2 %	15.7 %

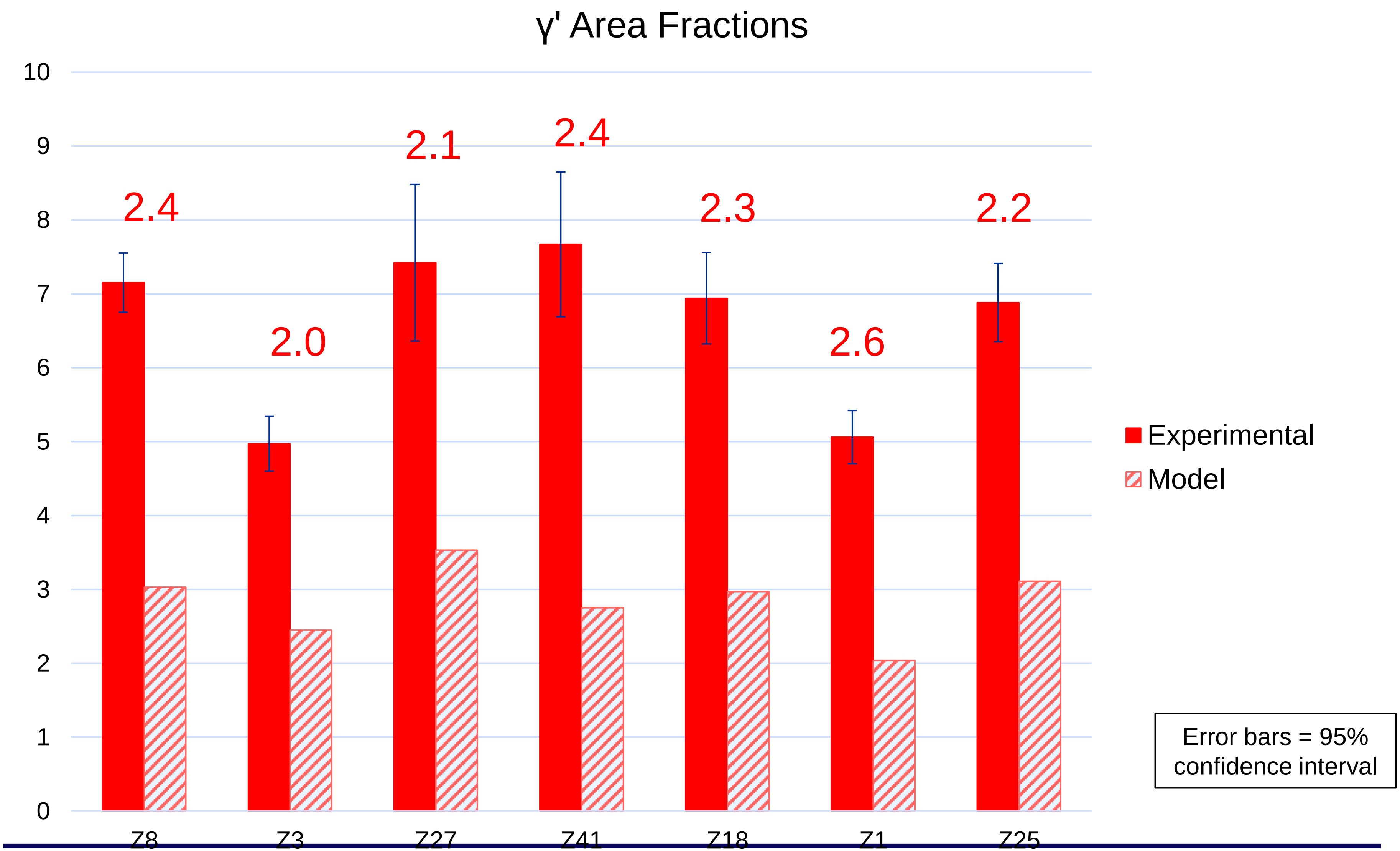
Can not separate $\gamma'/\gamma''/\delta$ phase due to similar chemistries

XRD and Phase Extraction Combined

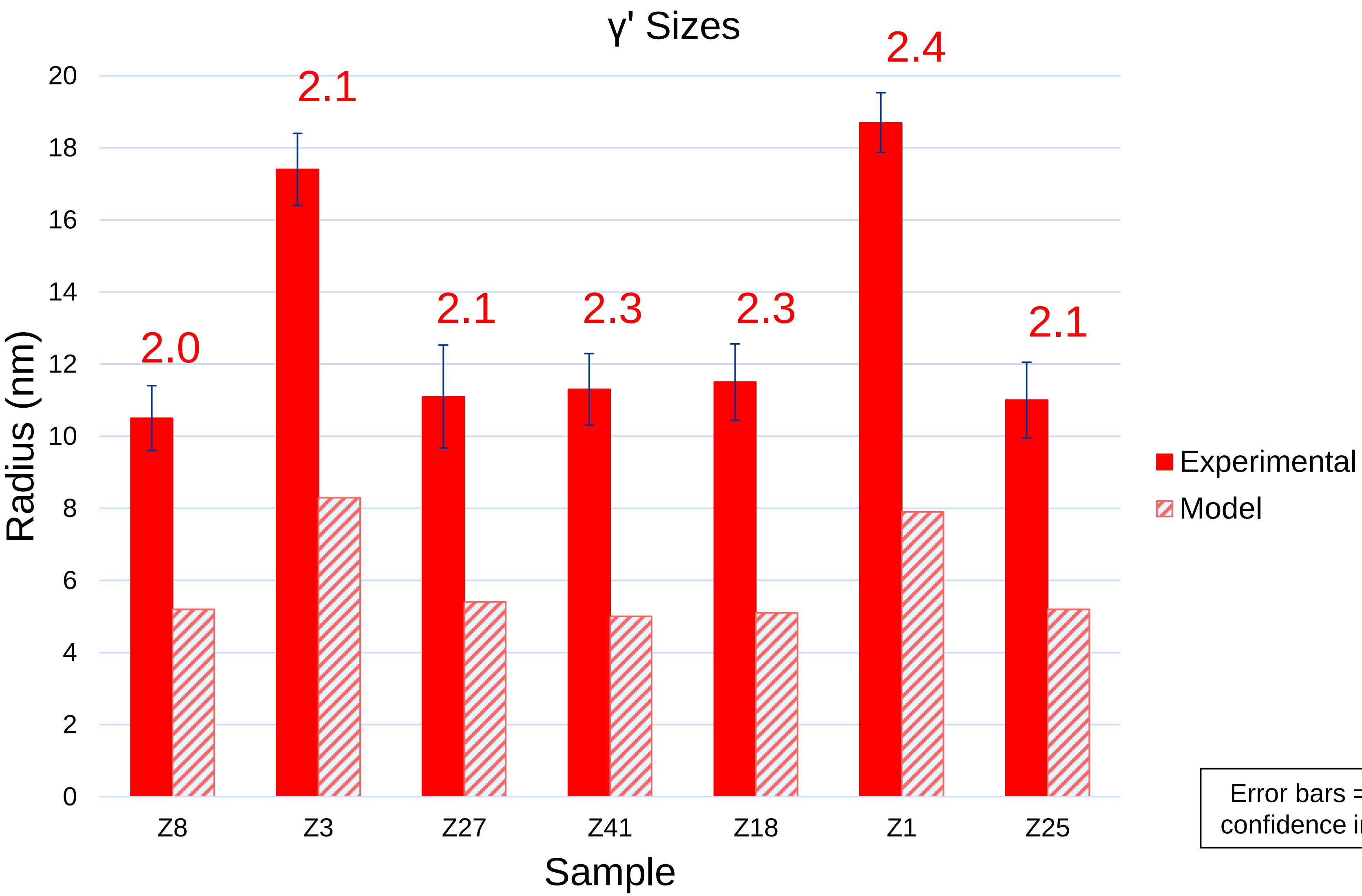
Precipitate Parameter	SEM	XRD + PE	Model
γ' volume fraction	5.1 ± 0.8 %	5.1 ± 0.6	2 %
γ'' volume fraction	11.1 ± 0.9 %	10.6 ± 0.6	14 %
δ volume fraction	.37 ± .24 %	0 %	2 %

The XRD + PE analysis validates the new SEM characterization technique!

Microstructural Analysis – Results
Gamma Prime Phase

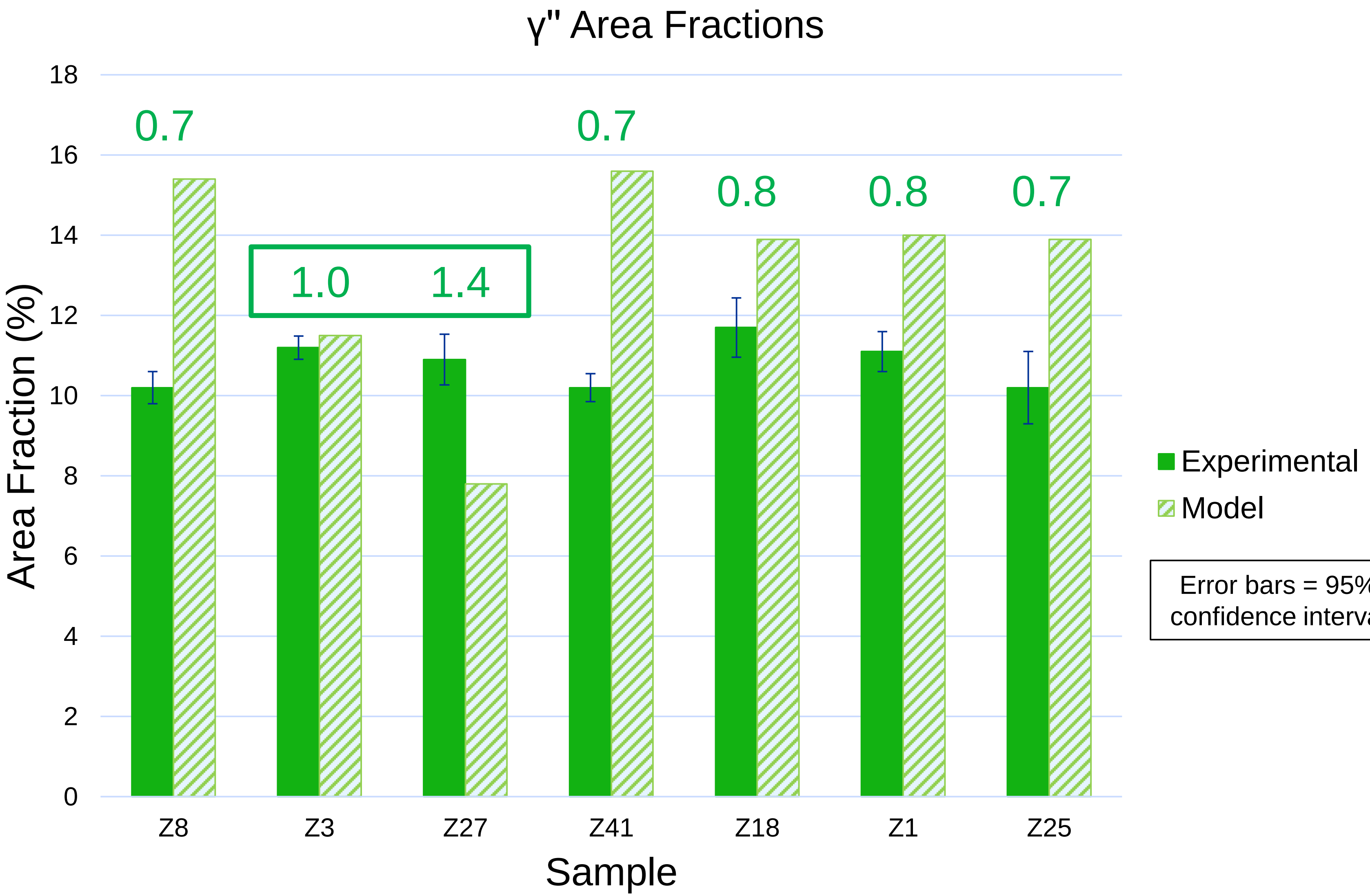


Microstructural Analysis – Results
Gamma Prime Phase



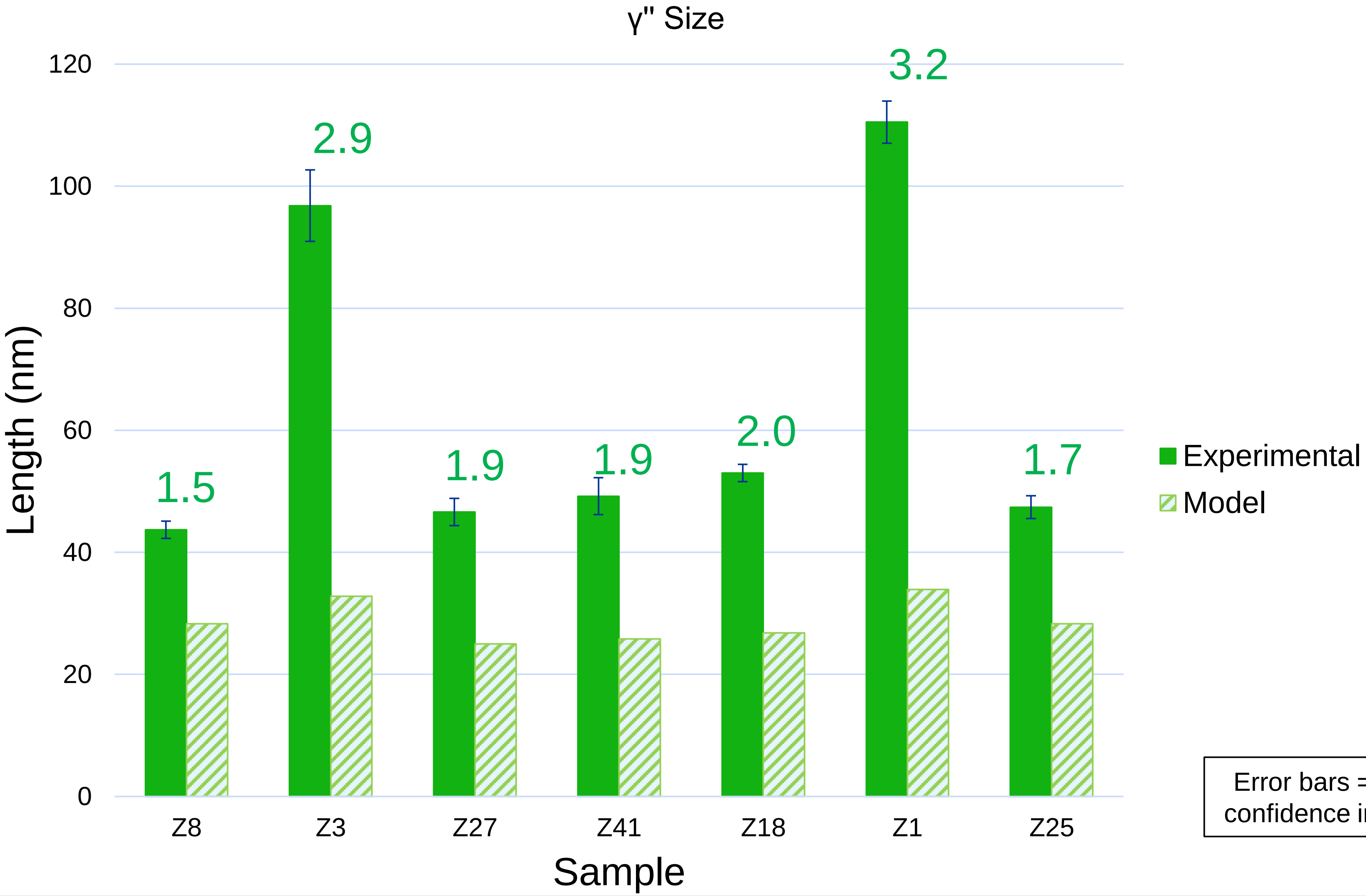
Error bars = 95% confidence interval

Microstructural Analysis – Results
Gamma Double Prime Phase



Error bars = 95% confidence interval

Microstructural Analysis – Results
Gamma Double Prime Phase



Error bars = 95% confidence interval

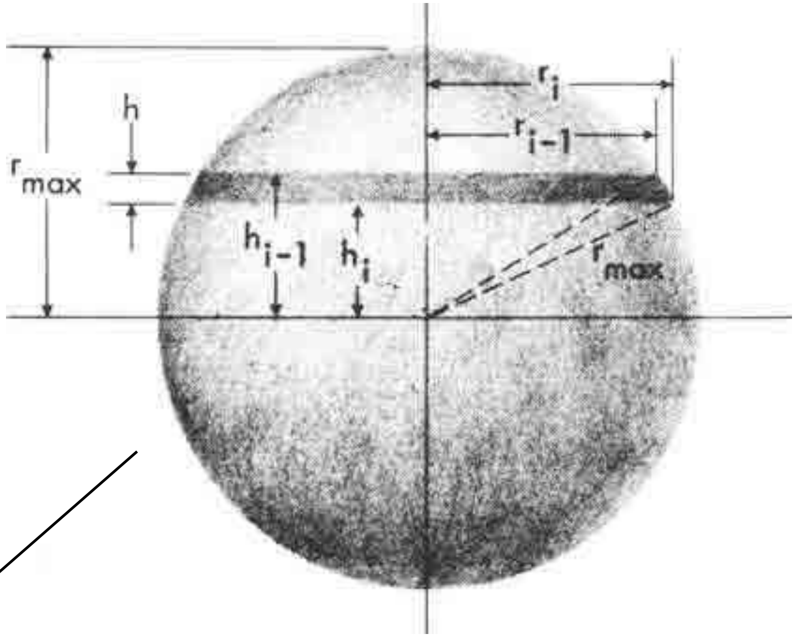
Methodology – 3D Size distributions

γ'' Size Analysis: [001] oriented grains
γ' Size Analysis: Any orientation

Using the measured area size distributions of each precipitate, the numerical volumetric size distributions were calculated using the equation below assuming a spherical particle*. This works for γ' for all orientations. For γ'' precipitates, it must be performed only on the two edge-on variants of γ'' in [001] oriented grains.

$$(N_v)_j = \frac{1}{\Delta}\sum_{i=j}^{k} \alpha_i \, (N_A)_i$$

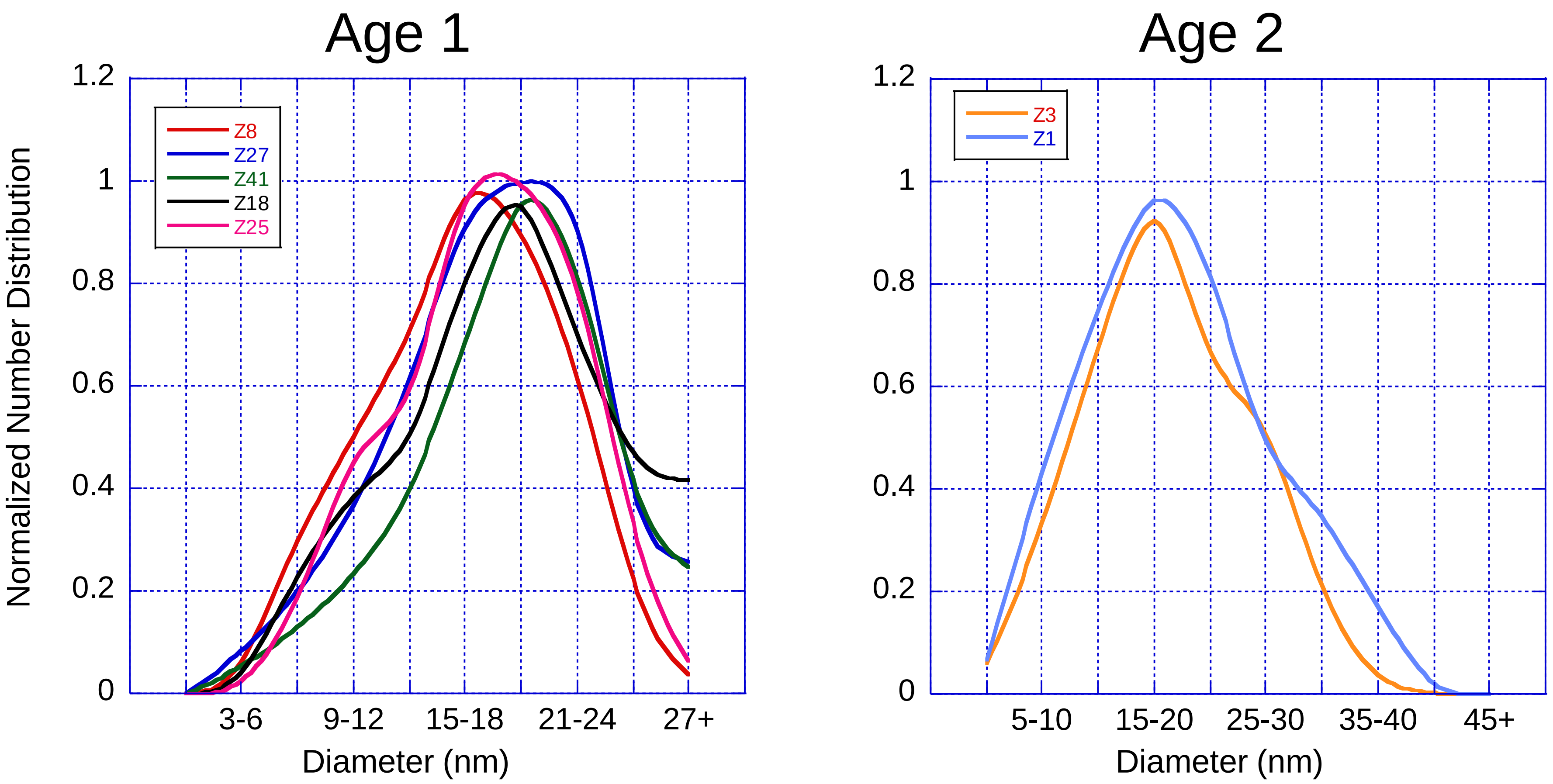
Where N_A is the experimentally obtained area number densities, D_{max}=kΔ, and k equals the total number of size groups. α is a pre-determined coefficients associated with the probability of the polish surface plane cutting a sphere as revealed below.



$$P_{i,j} = \frac{1}{r_{max}}\left[\sqrt{(r_{max}{}^2) - (r_{i-1})^2} - \sqrt{(r_{max}{}^2) - (r_i)^2}\right]$$

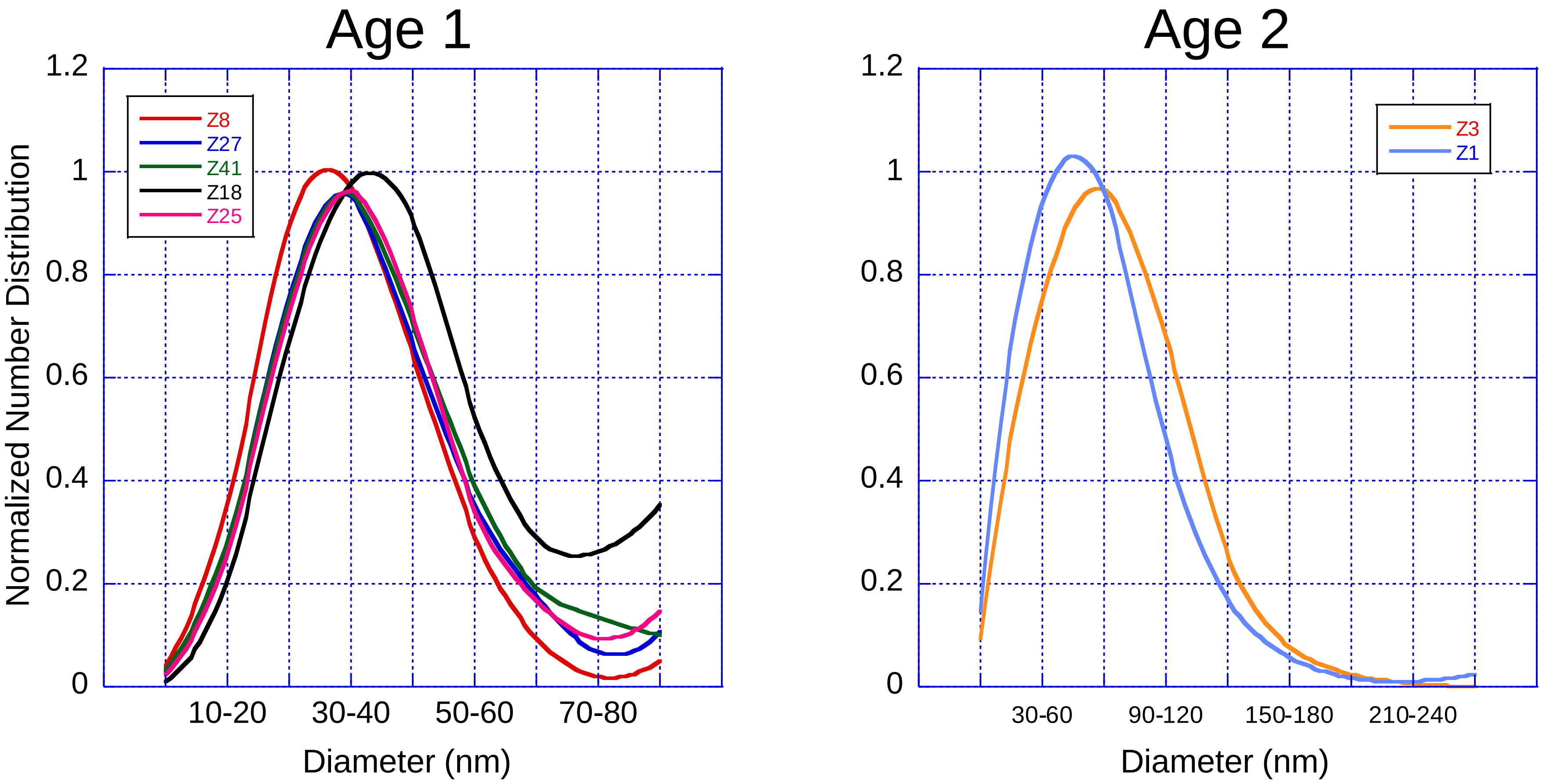
*Stereology and Quantitative Metallography, ASTM, STP 504

γ' Size Distributions



γ' precipitates possess a mostly normal size distribution.

γ'' Size Distributions



γ'' precipitates do not possess a normal size distribution.

Discussion

Experimental

- Composite particles are not completely separated (esp. Age 1 samples).

Assumptions:

- Perfectly etched samples
- γ' are spherical, γ'' are circular plates.
- No subsurface features are imaged.

Future work: further automate post-processing procedure and find more accurate ways to separate γ'/γ'' composite particles.

Model

- Carbides/Oxides were suspended to simplify calculations
- Inter-particle interactions not well established.

Tuning Parameters:

- Compatible thermodynamic database
- Compatible mobility database
- ΔE – phase energy shift for equilibrium phase fractions
- **D_{scale} – Diffusivity correction factor**
- Molar volume for each phase
- **Coherent surface energy (mJ/m^2)**
- Lattice misfit energy (mJ/m^2)
- Incoherent surface energy (mJ/m^2)

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Conclusions

- A new method using high resolution scanning electron microscopy combined with advanced processing techniques allows for unprecedented microstructural characterization of additively manufactured superalloy 718.
- XRD and Phase extraction support the findings from the SEM analysis.
- Differences in γ'' and γ' size distributions are currently unexplained.
- Currently, the precipitation models predict the microstructural trends resulting from different post-processing heat treatment steps.
- Calibrating future precipitation models using results from this new technique will further improve their accuracy.

Acknowledgments: Microstructural Characterization and Modeling of SLM Superalloy 718

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- Connor Slone - OSU

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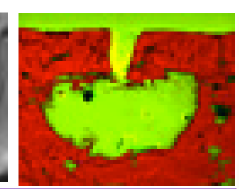


Questions?