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Image Analysis of Off-Eutectic Alloys with Lamellar Microstructures

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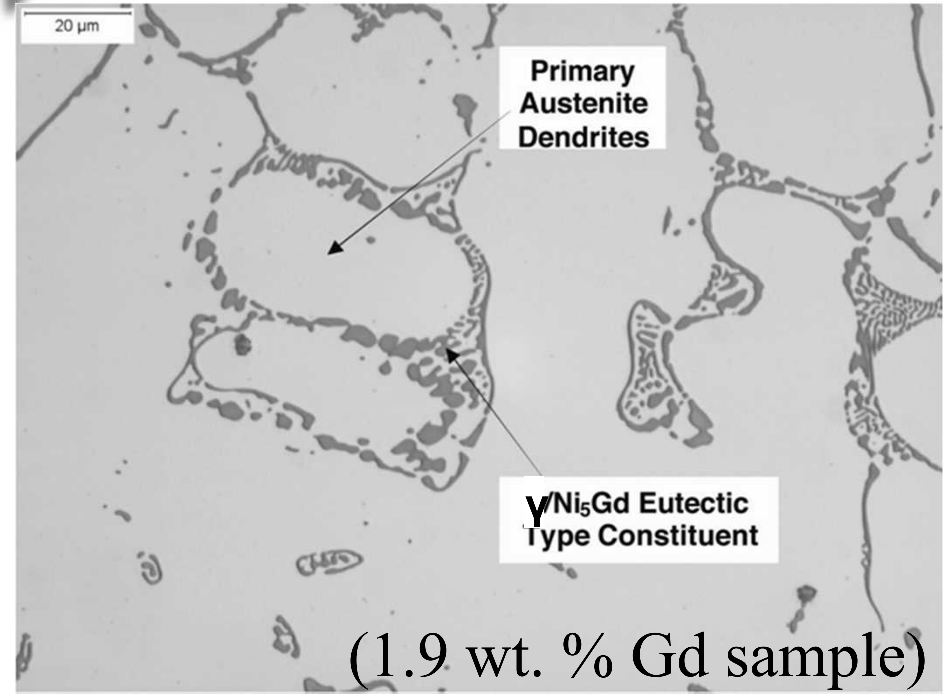
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Motivation

- Ni-Cr-Mo-Gd alloys are being developed for safe transportation and storage containers for spent nuclear fuel.

- An understanding of the solidification behavior of these alloys is needed to aid in processing (ingot metallurgy, welding, etc.) and microstructural control.

Ni-Cr-Mo-Gd As-Cast Microstructure



(1.9 wt. % Gd sample)

- Microstructure consists of primary austenite (γ_{pri}) dendrites and interdendritic "eutectic-like" mixture of austenite (γ_{eut}) and $GdNi_5$.
- Beraha's 7A tint-etch used to delineate the $GdNi_5$ constituent. Good contrast is achieved for thresholding and image analysis.

	Element (wt%)										
Alloy	Ni	Mo	Cr	Fe	Gd	P	S	Si	O	N	C
1B	67.7	15.67	16.00	0.016	0.46	<0.001	<0.001	0.070	0.0044	0.0045	0.0052
2B	67.2	15.53	16.08	0.017	1.01	<0.001	<0.001	0.081	<0.001	0.0044	<0.005
3B	66.9	15.56	15.91	0.015	1.49	<0.001	<0.001	0.070	<0.001	0.0039	0.0064
4B	66.8	15.16	15.95	0.016	1.9	<0.001	<0.001	0.070	0.0021	0.0041	0.0065
5B	66.7	15.12	15.56	0.016	2.45	<0.001	<0.001	0.071	0.0056	0.0043	0.0066

Five alloys studied with increasing Gd content

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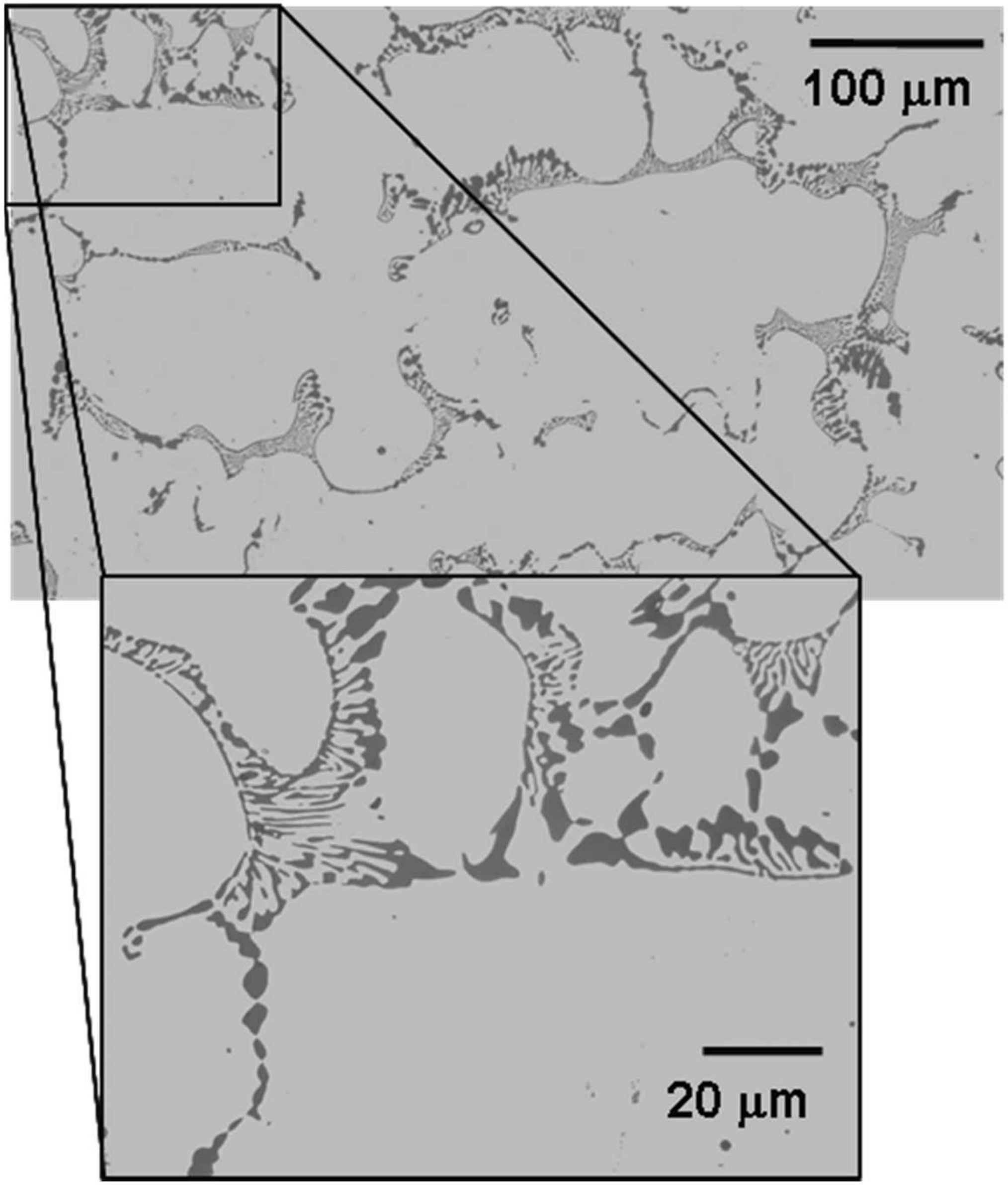
Mosaic (montage) Images used for Image Analysis



• Ability to cover a significant area of the sample and collect statistically significant IA data, while maintaining higher resolution of the individual images.

• Each mosaic typically has 12 images obtained at 500x magnification. Three or four separate mosaic images analyzed for each alloy sample (36-48 individual images).

• Clemex Vision PE image analysis system

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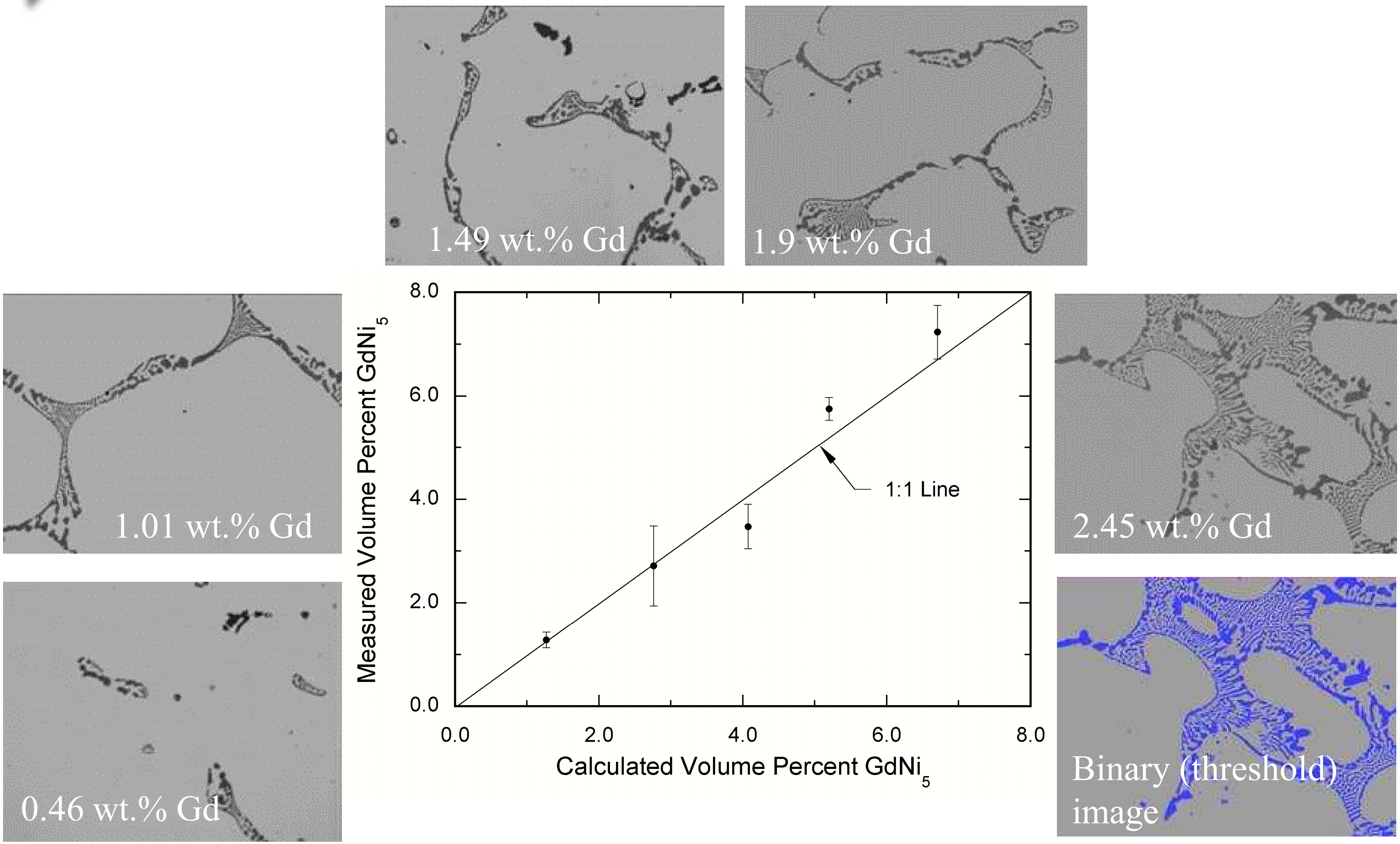
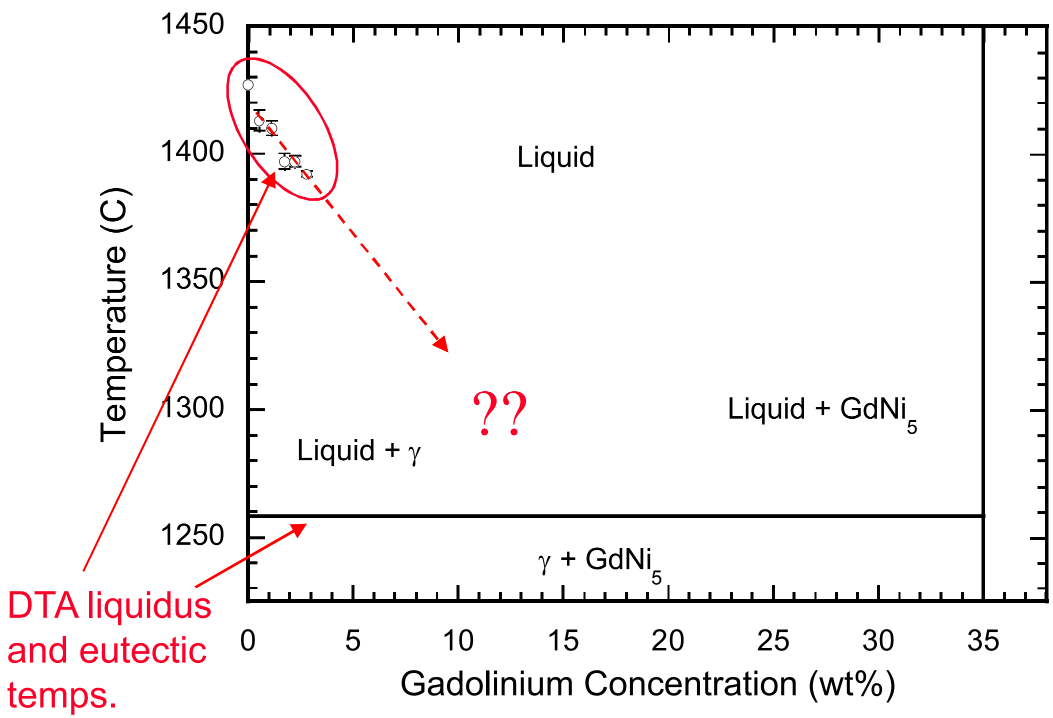
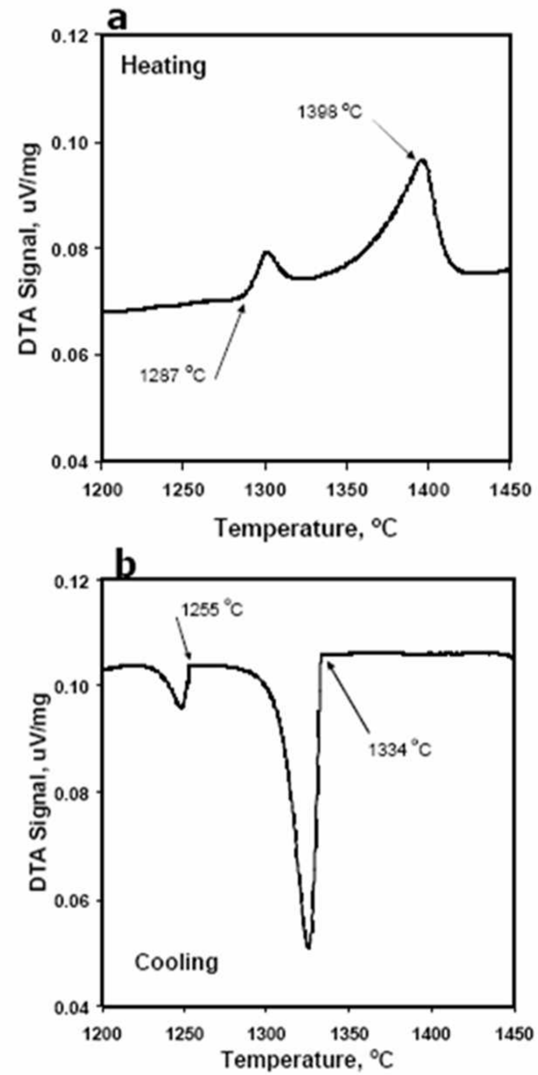
Amount of $GdNi_5$ increases with Gd content in alloy



Differential Thermal Analysis (DTA) used to determine liquidus temps. and approximate "eutectic" temp.

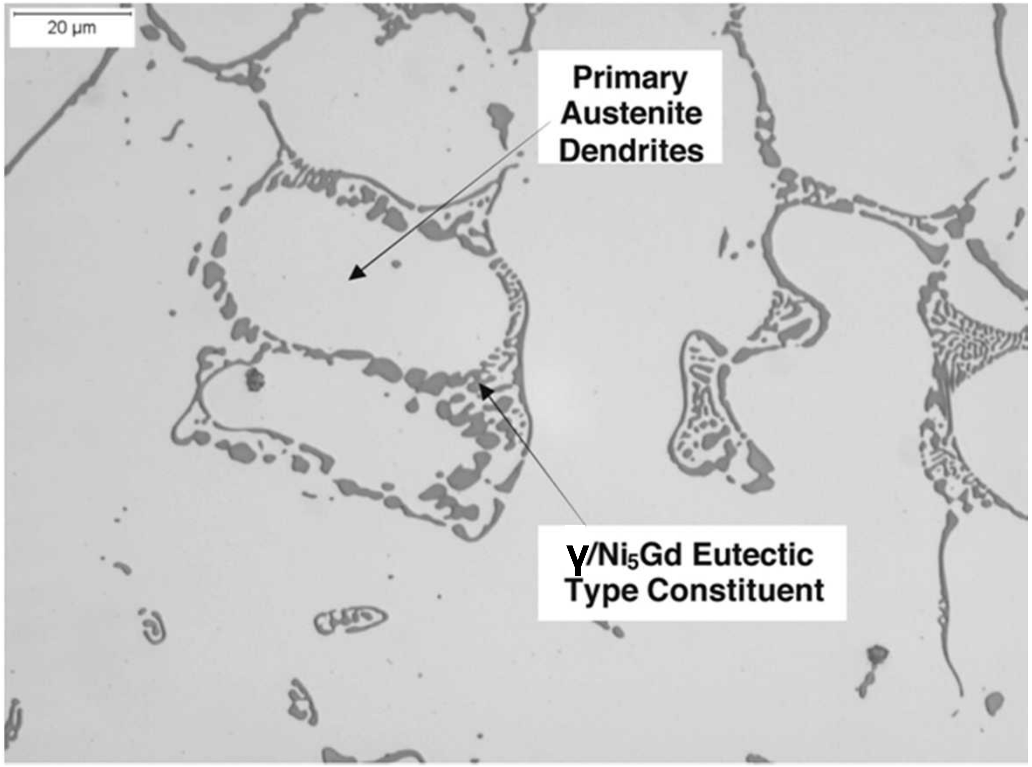


Partial γ-Gd "solidification diagram"



On-heating and on-cooling DTA scans

Challenge: Determine the eutectic-like point by image analysis of a few, relatively dilute alloys. Specifically, determine the ratio of γ and $GdNi_5$ *within* the eutectic-like regions (Lever Rule).



Attempt to threshold and "fill-in" eutectic regions ("closed" $GdNi_5$ image) is incorrect--this method does not account for γ_{eut} that forms on the primary γ_{pri} dendrites.

In this alloy system, the fill-in technique results in 58-65 vol.% $GdNi_5$ within the eutectic – this value is inaccurate, too high.

Problem: γ_{eut} cannot be distinguished from γ_{pri} in the microstructure.

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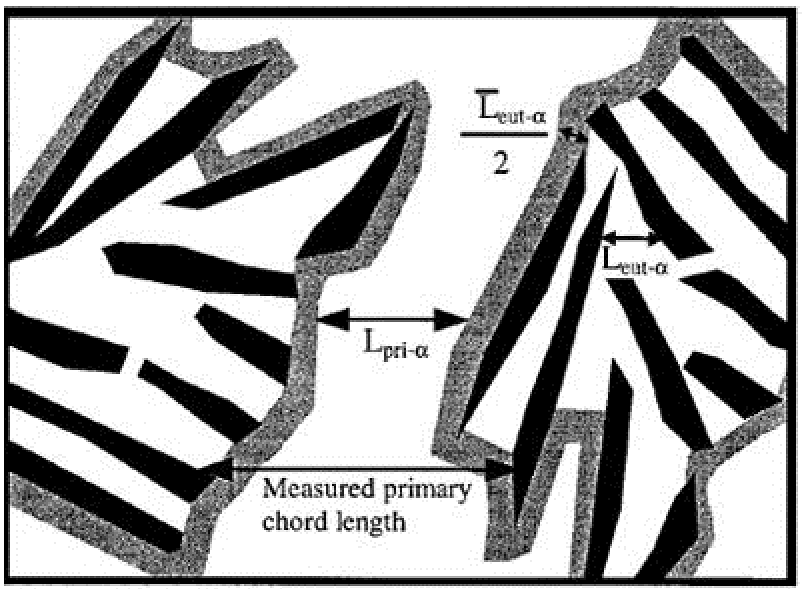
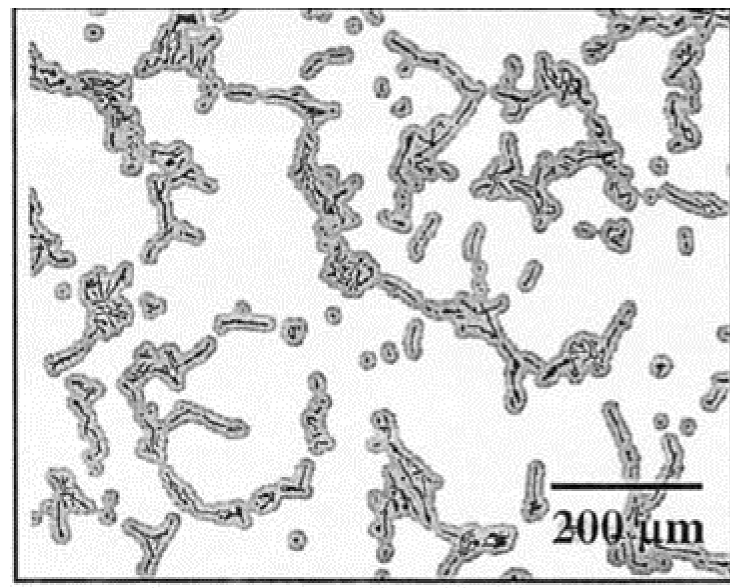
Method of Dutta and coworkers

- Uses a dilation and erosion technique (also called the "chord size technique") to determine the eutectic fraction, while taking into account the portion of γ_{eut} that solidifies on the primary γ_{pri} dendrites
- Dutta et al. studied several image analysis methods for analyzing Al-Si, Al-Fe-Si, and Al-Cu alloys



Schematic diagram of eutectic structure with layer of α_{eut} which solidifies on the primary α_{pri} dendrites (ref 1)



"Reconstructed" eutectic microstructure in Al-0.8Fe-0.8Si alloy (ref 1)

1. B. Dutta, O. Pompe, and M. Rettenmayr: *Materials Science and Technology*, 2004, vol. 20, pp. 1011-18.
2. B. Dutta and M. Rettenmayr: *Materials Science and Technology*, 2002, vol. 18, pp. 1428-34.
3. B. Dutta and M. Rettenmayr: *Materials Science and Engineering A*, 2000, vol. A283, pp. 218-24.
4. O. Pompe: *Prakt. Metallogr.*, 1994, vol. 31, (6), pp. 274-83.

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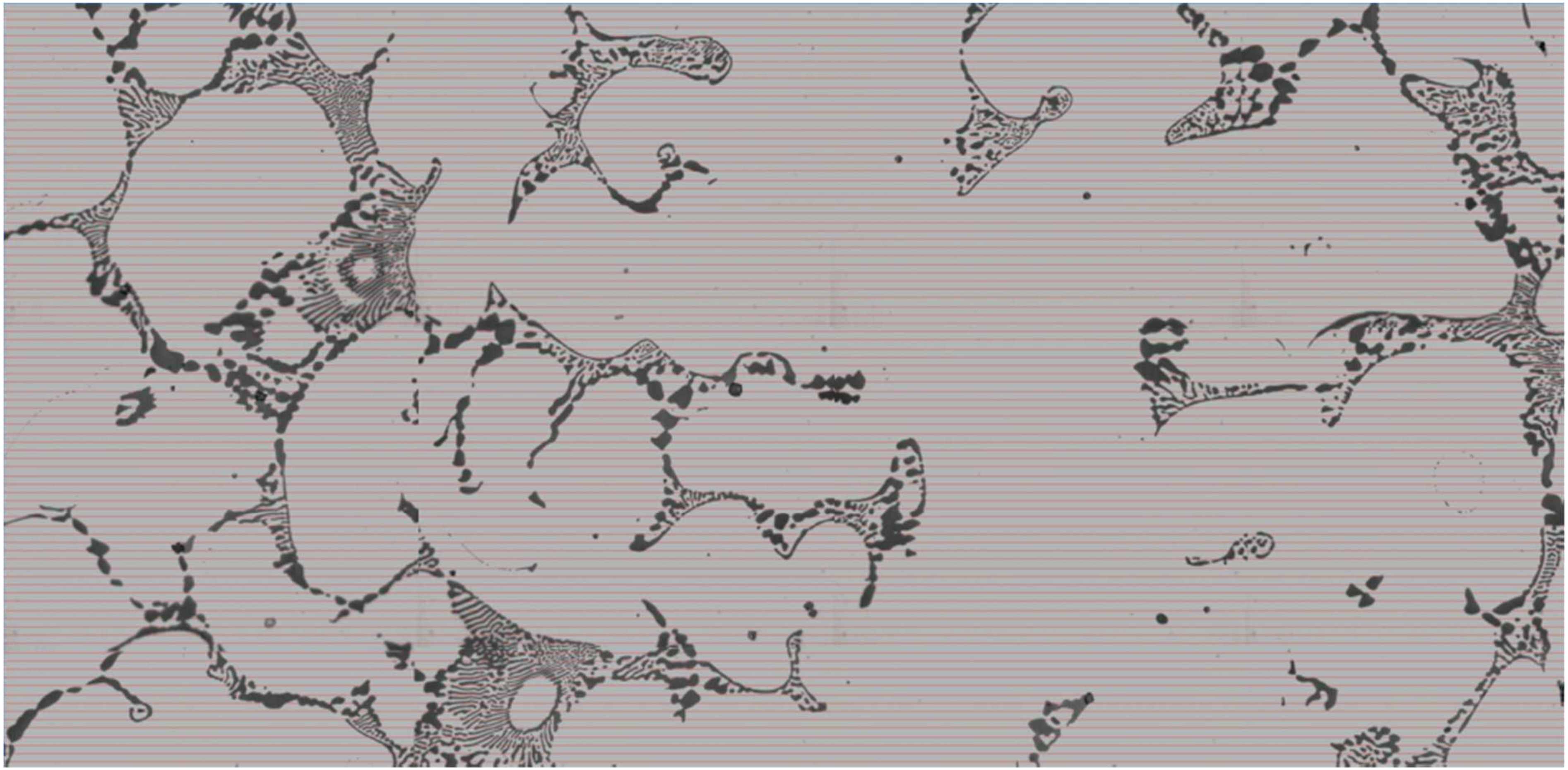
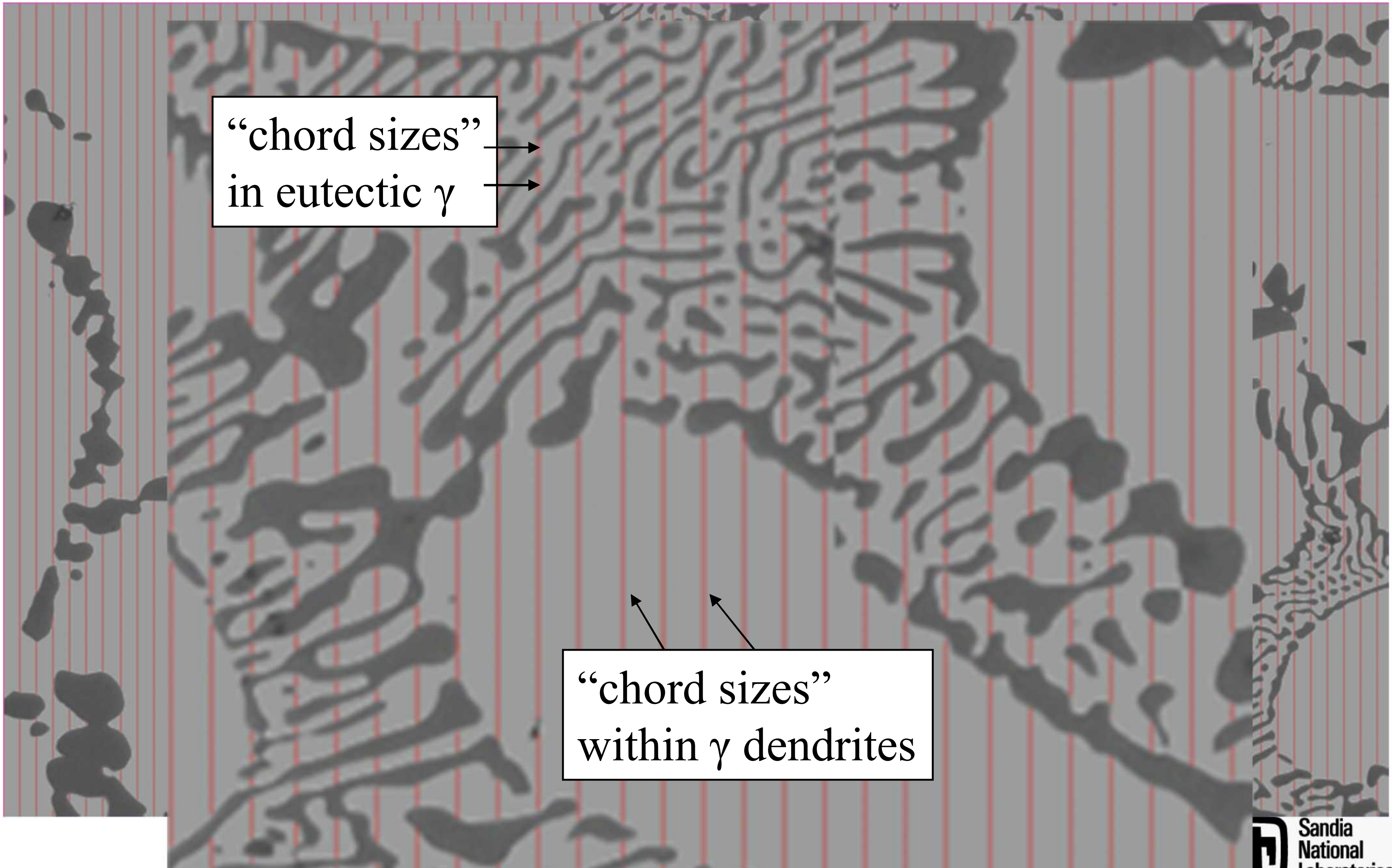
1. Estimate the γ_{eut} Spacing with Chord-Size Technique

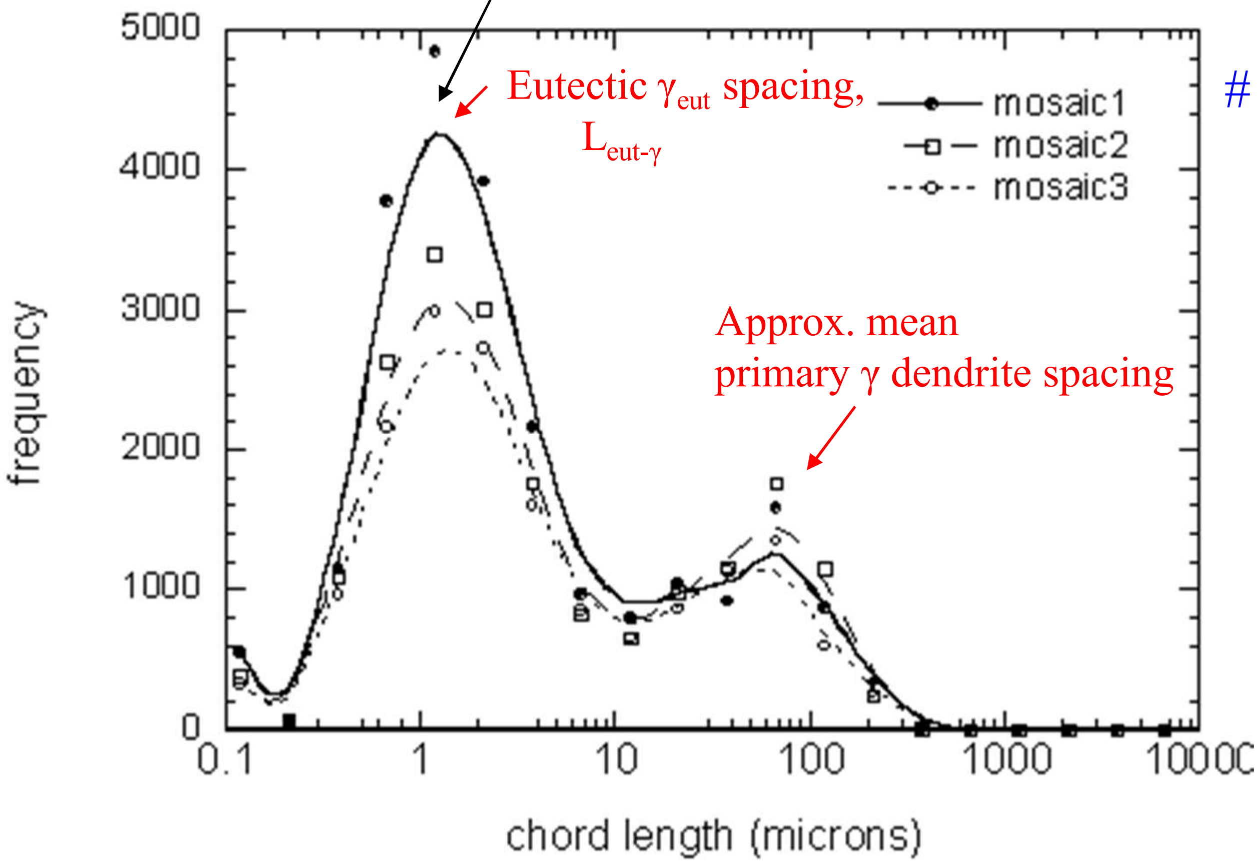


1. Estimate the γ_{eut} Spacing with Chord-Size Technique



Histograms of γ (Austenite) Chord Lengths used to determine γ eutectic spacing

This value is used in dilation-erosion technique to add a layer of $(1/2)L_{eut-\gamma}$ on the γ primary dendrites



$$D - E = \frac{\overline{L}_{eut-\gamma}}{2L_{pix}}$$

\# dilations

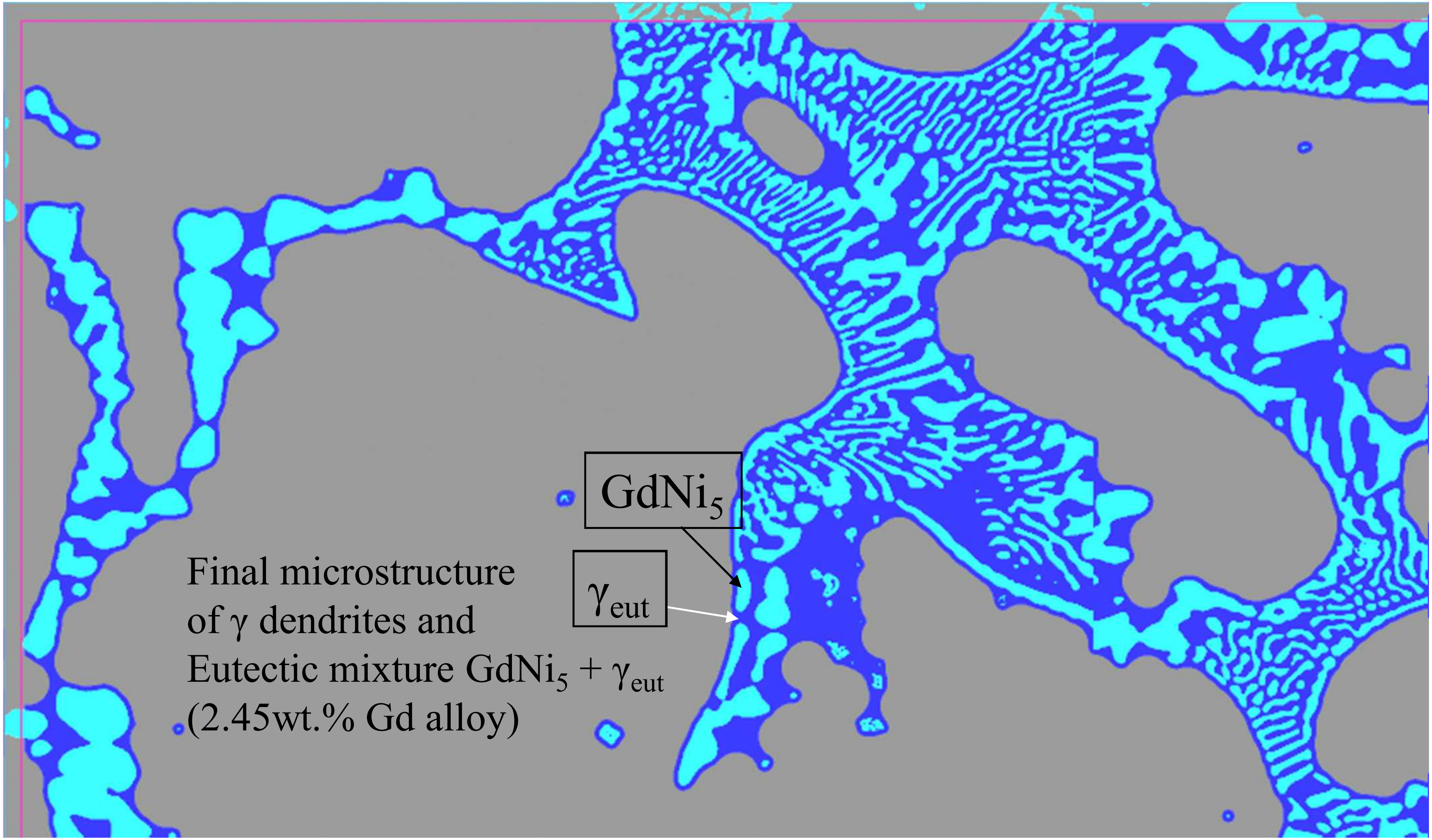
γ eutectic spacing

\# erosions

Pixel size in image

In our alloys, D-E ≈ 6

2. Dilation-Erosion technique adds a layer of γ_{eut} onto the γ_{pri} dendrites, while simultaneously filling in the eutectic regions.



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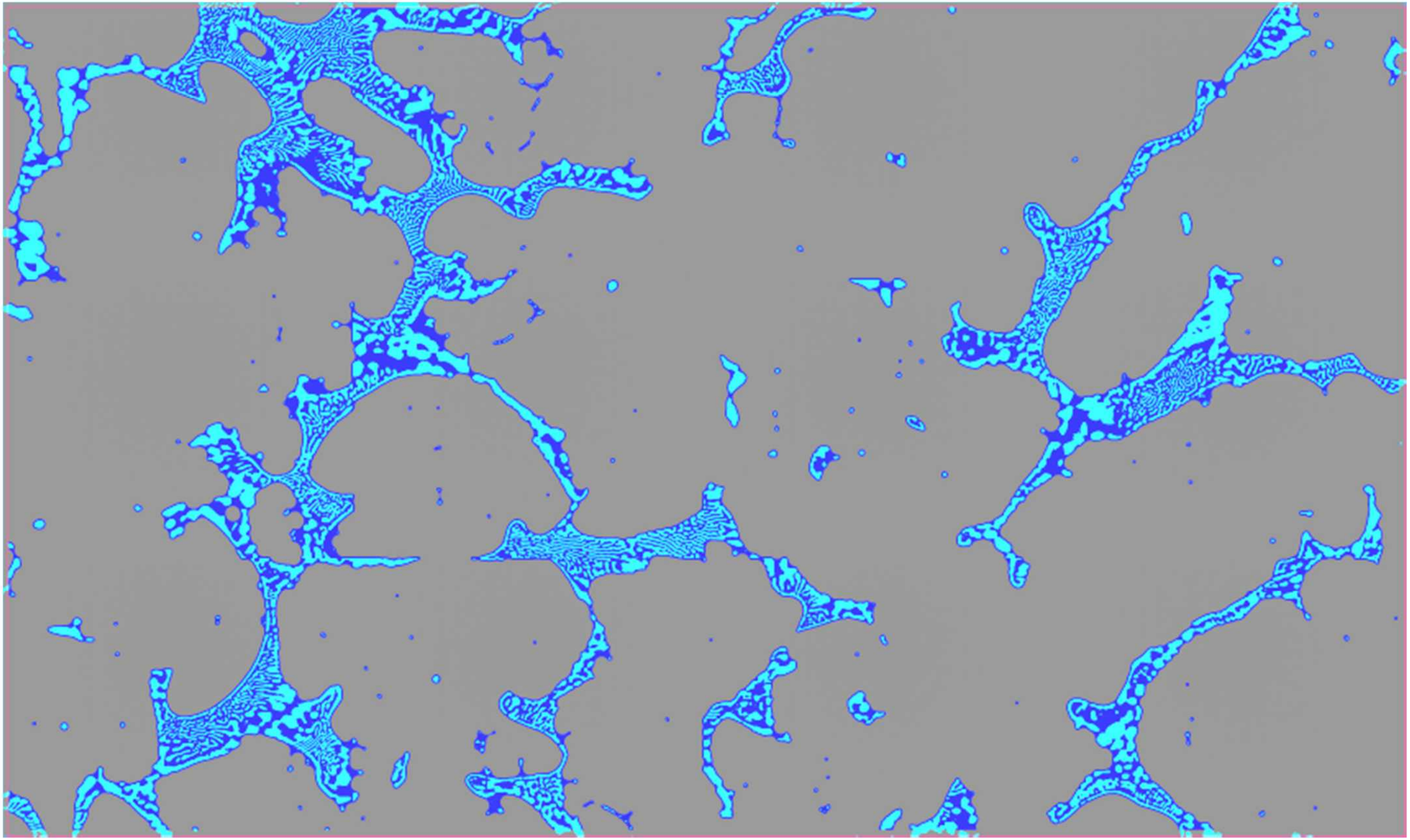
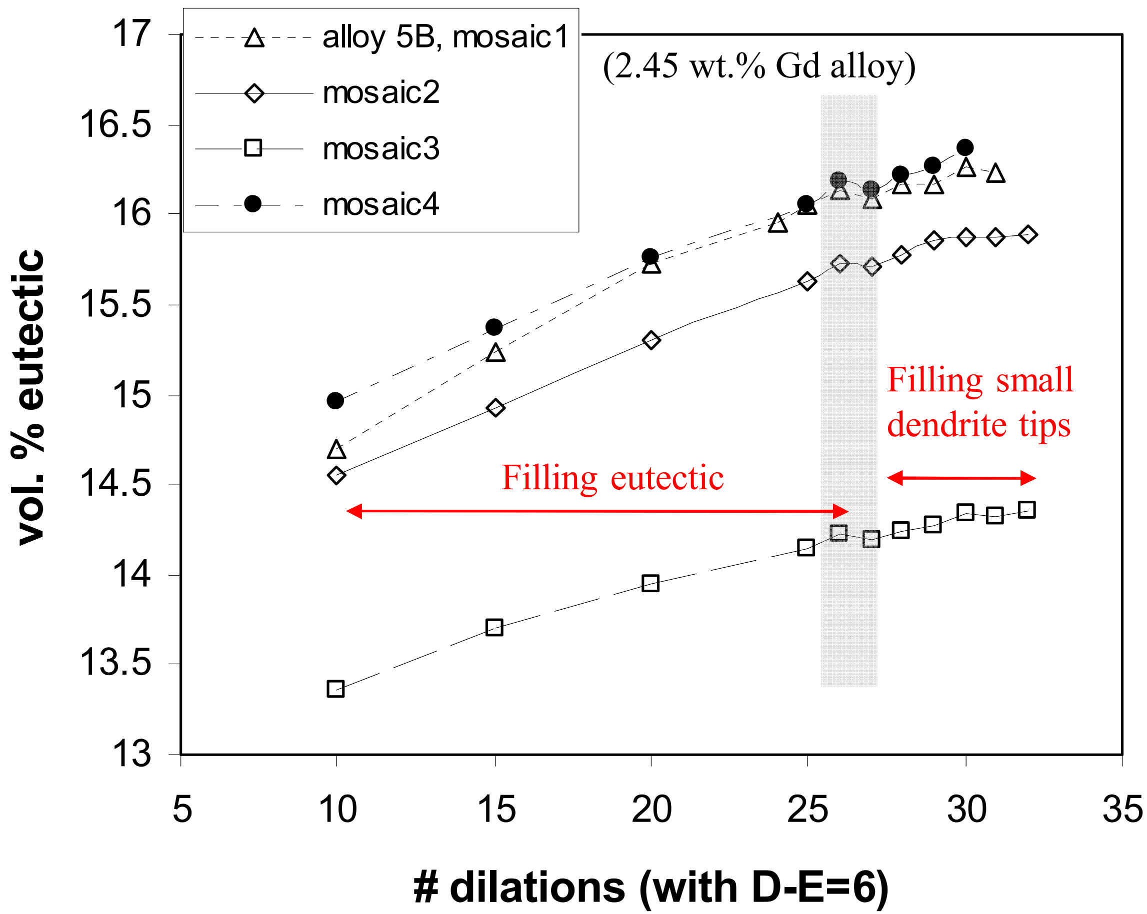
Reconstructed interdendritic eutectic microstructure of $GdNi_5 + \gamma_{eut}$.



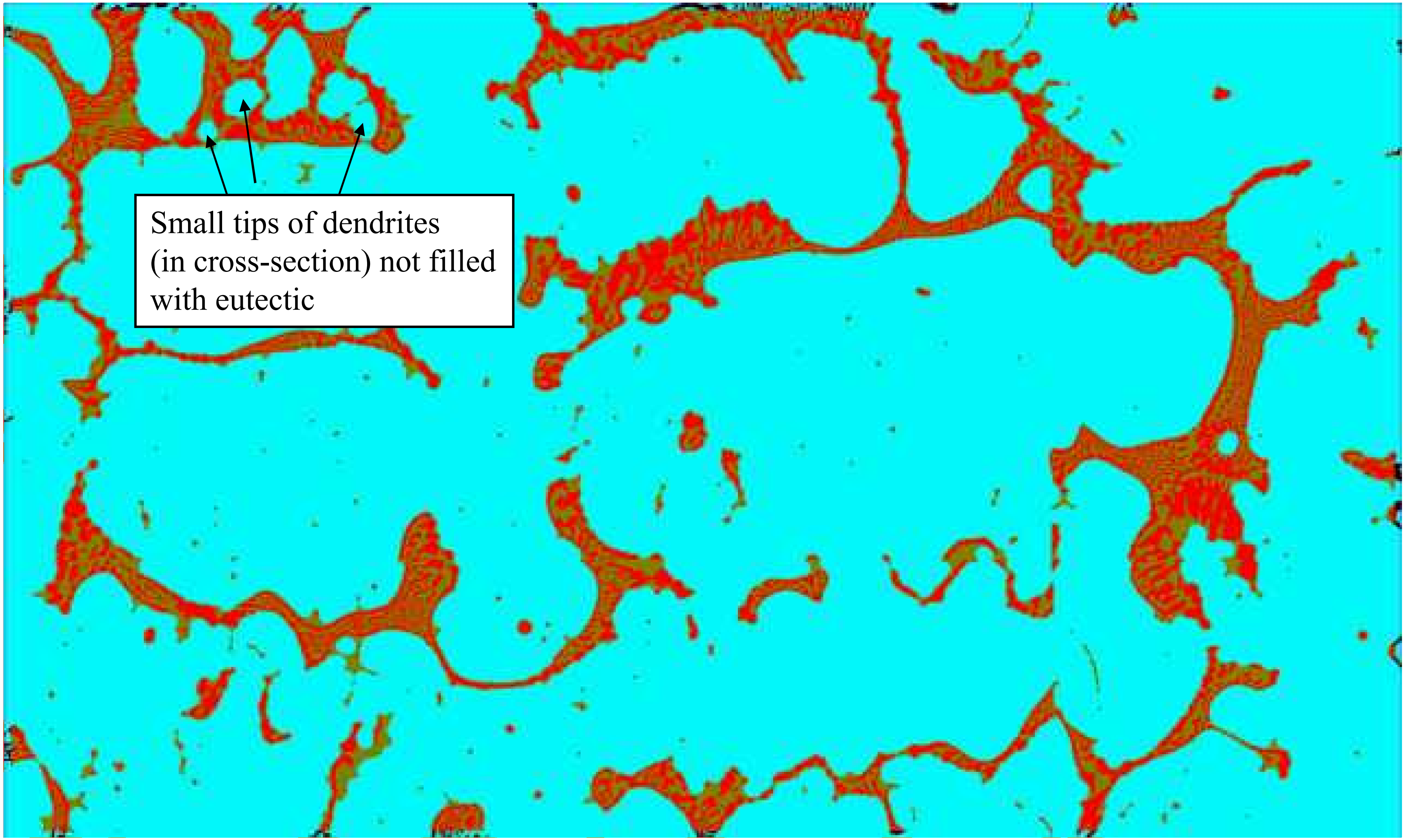
2.45 wt. % Gd alloy

How is the correct number of dilations determined?



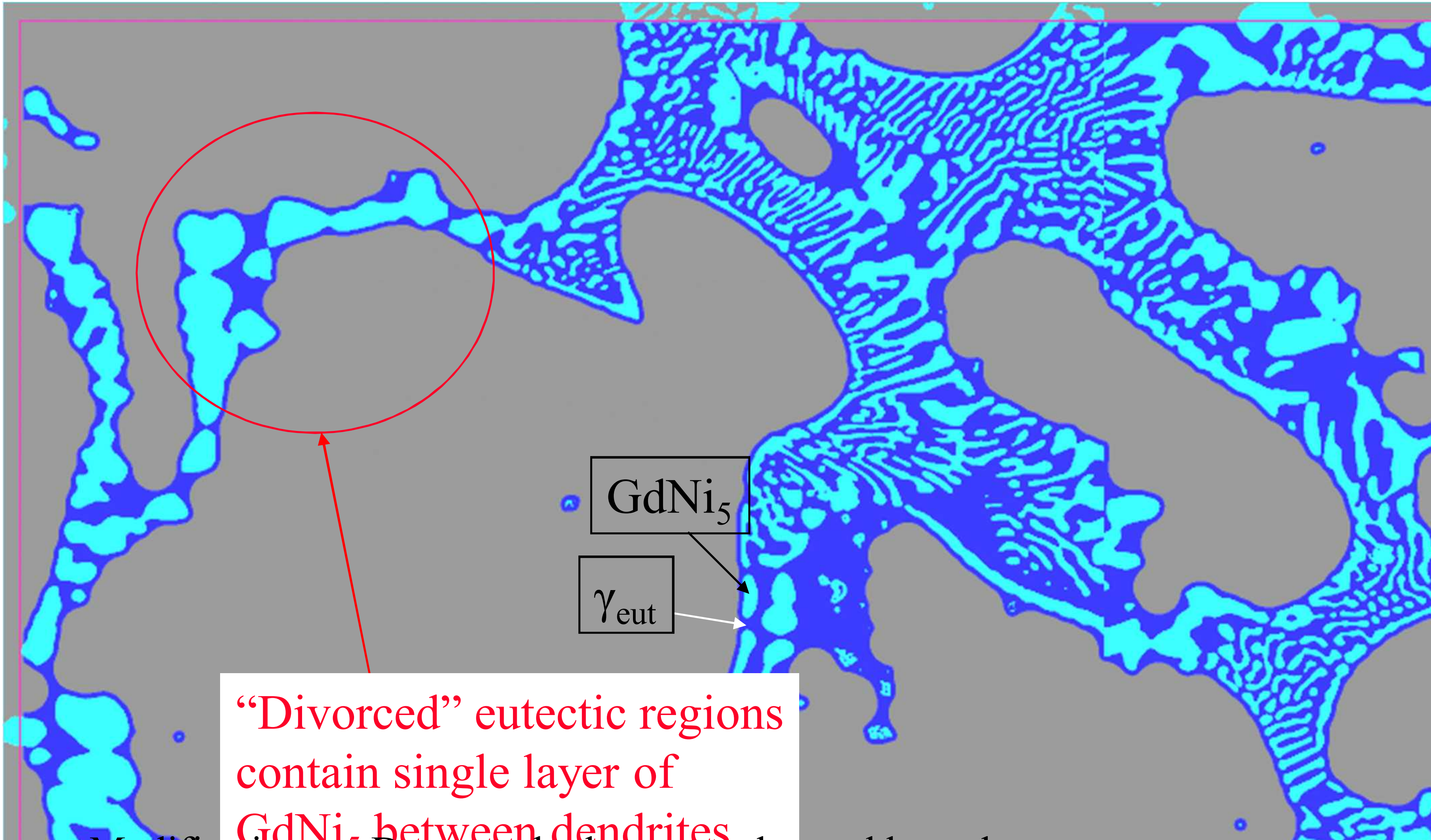
- Increase # dilations while keeping (D-E) constant

- Vol. % eutectic levels off when eutectic regions are filled and *before small, primary dendrite tips begin to be filled…*



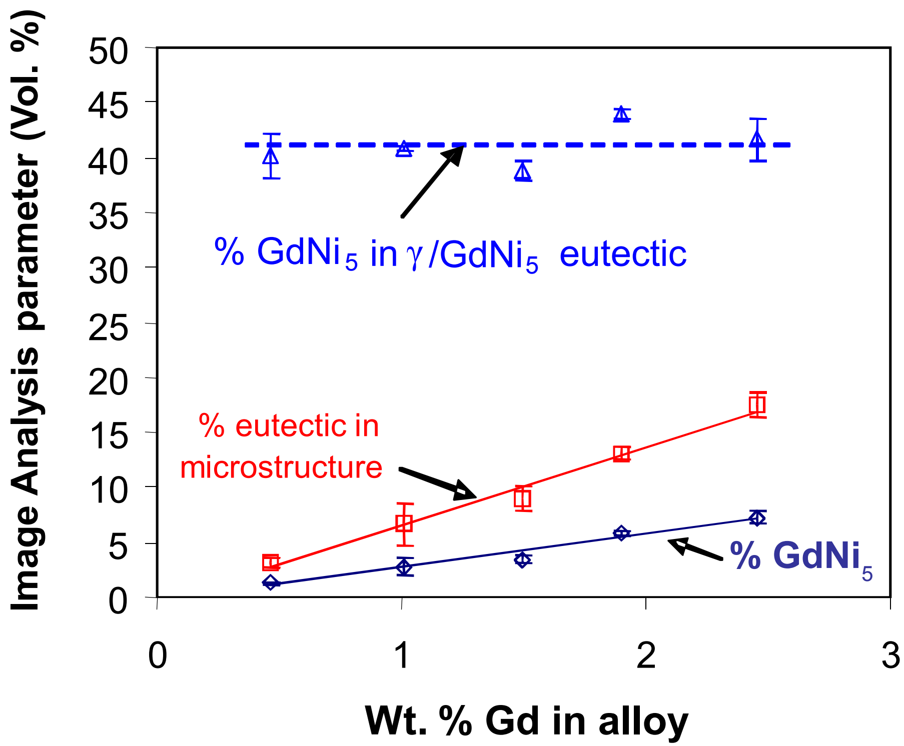
Mosaic image, 2.45wt.% Gd alloy

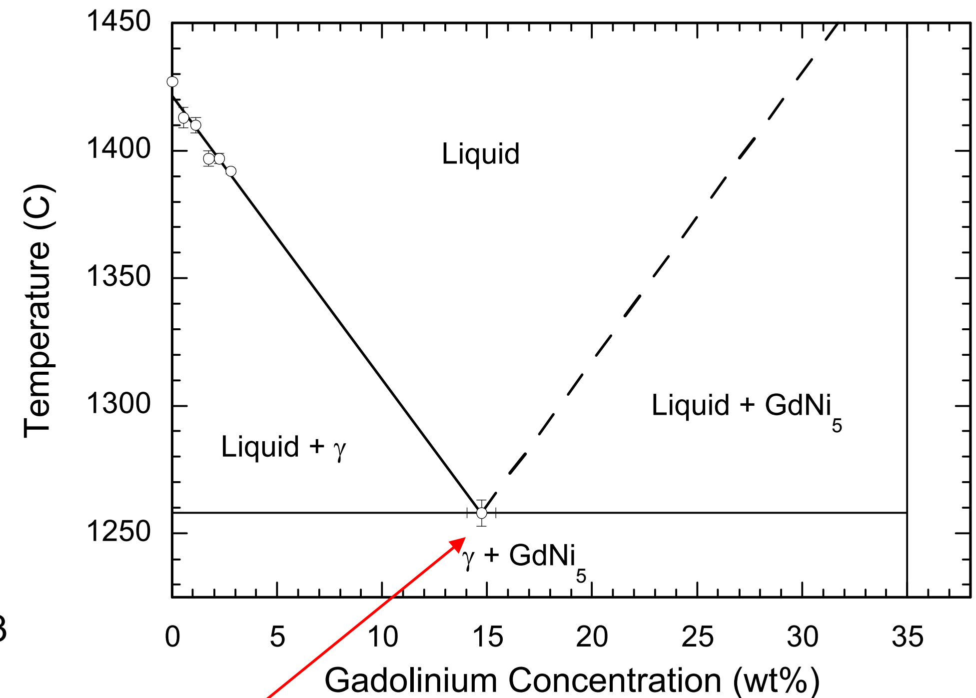
Regions with “Divorced” Eutectic Morphology



• Modifications to Dutta method were made to address the partly divorced eutectic microstructure (a semi-automated procedure)

Final Image Analysis Data and Solidification Diagram

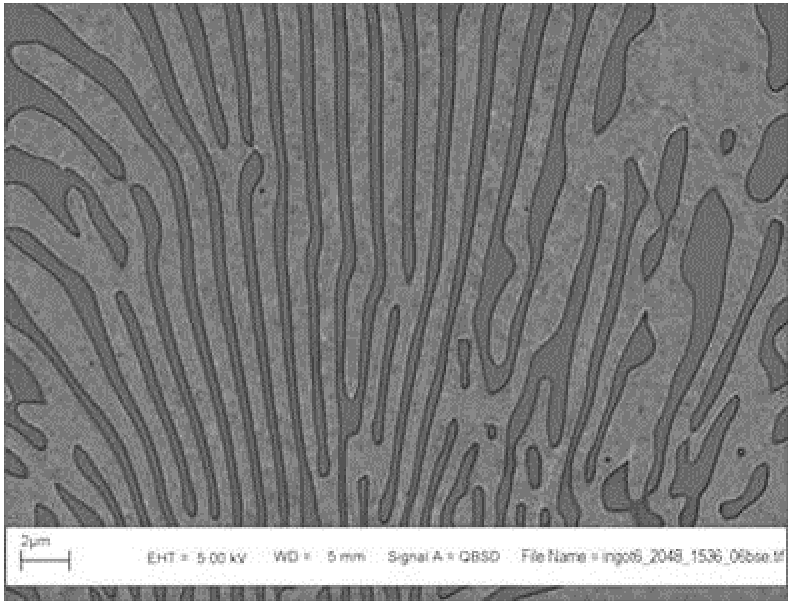




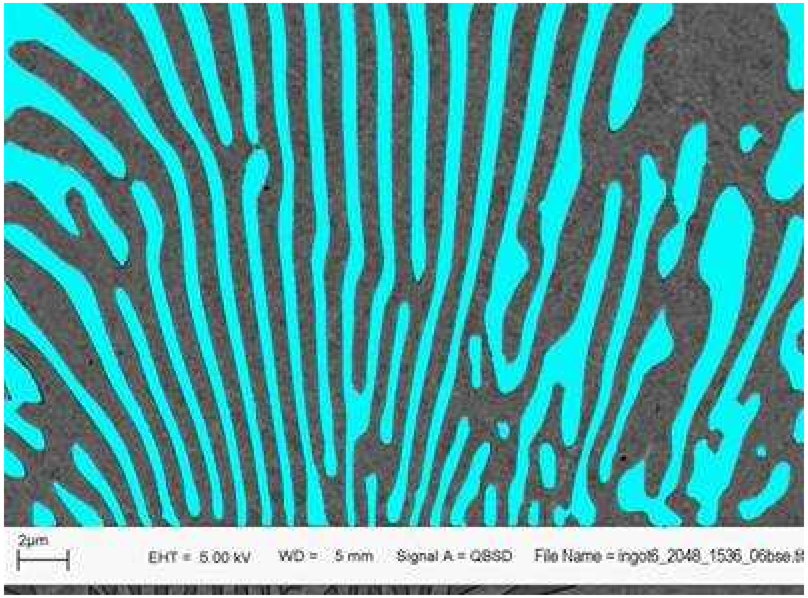
The 5 alloys show increasing $GdNi_5$, increasing eutectic content, but constant eutectic phase ratio, i.e. eutectic-like behavior

Eutectic composition is 14.7 wt.% Gd (41 vol. % $GdNi_5$ in eutectic mixture)

High Magnification SEM



BSE/SEM image of eutectic only



Binary threshold image of eutectic only

- Eutectic composition determined to be 40 ± 0.8 vol. % $GdNi_5$ (60 % γ)
- This is very close to 41 ± 1.9 % $GdNi_5$ obtained with optical microscopy/image analysis technique

(recall 58-65 vol. % $GdNi_5$ was found with simple OM "fill-in" of eutectic regions)

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Summary

- A dilation-erosion technique, first developed by Dutta et al., was employed to determine the eutectic point in a model solidification diagram for γ-Gd alloys. The method accounts for the portion of γ_{eut} that grows on the primary γ dendrites and, in principle, can be applied to any (off-eutectic) alloy system.

- A modified, semi-automated technique was employed to characterize the complex, partially divorced morphology in these alloys.

- The "eutectic point" was determined to be 14.7 ± 0.68 wt. % Gd. This value corresponds to a eutectic mixture of 41.0 ± 1.93 vol. % $GdNi_5$ (and ~59 vol. % γ_{eut}).

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Acknowledgements

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