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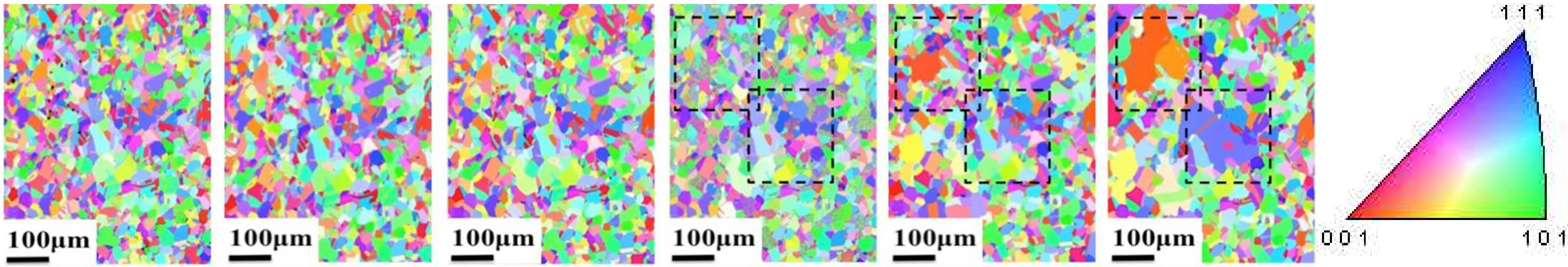


# Onset of Abnormal Grain Growth in Nickel-200 Within the Low-Strain Regime

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# Outline

- Introduction
  - Nickel
  - Abnormal Grain Growth (AGG)
  - Thermomechanical Processing
- Experimental
  - Preliminary study as a motivation for the current investigation
  - Rolling procedure
  - Metallography, cyclic annealing, & microscopy
- Results & Discussion
  - Provide implications for value/use of this study
- Conclusion
  - Summarize what we observed & State what we found

# Nickel

- Nickel is used in a variety of critical engineering applications. e.g. nickel based superalloys (Ni-Al), Monel based alloys (Ni-Cu), and Nichrome (Ni-Cr)

- **Applications:**
  - Aircraft engines
  - Power generation equipment
  - Nuclear and chemical processing plant


*Nickel is the main constituent in nickel based alloys.*

- **Favorable properties:**
  - high strength at high temperatures
  - high toughness
  - excellent service performance
  - strong creep rupture strength
  - significant fatigue resistance
  - high resistance to degradation in corrosive or oxidizing environments

**Grain structure can be critical in controlling properties**


1. T.M. Pollock, S. Tin, **Journal of Propulsion and Power** 22 (2006) 361.
2. B.M. Guyot, N.L. Richards, **Materials Science and Engineering: A** 395 (2005) 87.
3. L. Viskari, M. Hornqvist, et.al., **Acta Materialia**, 61(2013) 3630.
4. J. Tong, et al., **International Journal Fatigue**, 23(2001) 897.
5. R. H. Bricknell, D.A. Woodford, **Metallurgical Transactions A**, 12 (1981) 425.
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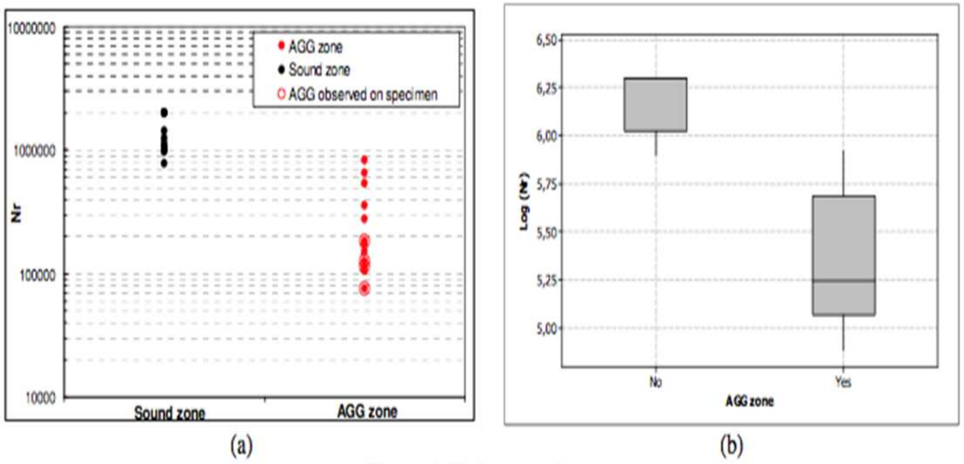
# Abnormal Grain Growth

- A phenomenon referred to as abnormal grain growth (AGG) occurs when grain growth is not self-similar, and a specific subset of grains grow at a rate faster than other grains
  - Detrimental impact on mechanical properties

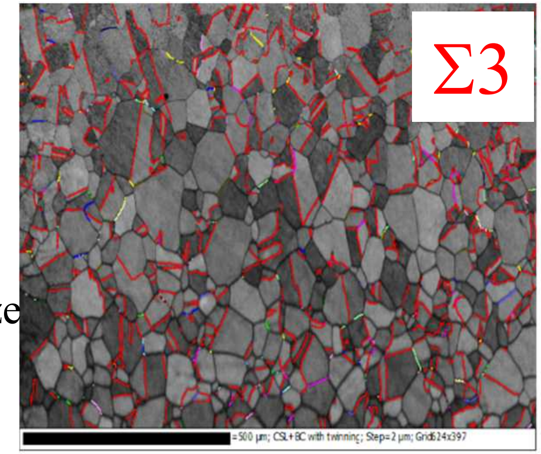



Reduction in fatigue life in nickel-based superalloy 718 in the presence of AGG from B. Flageolet, et al. "Characterization of Microstructures Containing Abnormal Grain Growth Zones in Alloy 718", in proceedings of 7th Int. Symp. On Superalloy 718 and Derivatives, (2010), TMS


1. T. Gabb, T., et al., Fatigue Resistance of the Grain Size Transition Zone in a Dual Microstructure Superalloy Disk. **33**, 414-26
2. E. Holm, et al., On Abnormal Subgrain Growth and the Origin of Recrystallization Nuclei. **51**, 2701-16
3. S. Fang, et al., The Abnormal Grain Growth of P/M Nickel-base Superalloy: Strain Storage and CSL Boundaries. **1064**, 49-54
4. S. Lee, et al., Grain Boundary Faceting and Abnormal Grain Growth in Nickel. **13A**, 985-93
5. R. Watson, et al., Characterization of Abnormal Grain Coarsening in Alloy 718. **14**, 1-5
6. C. Park, et al., New Understanding of the Role of Coincidence Site Lattice Boundaries in Abnormal Grain Growth of Aluminum Alloy. **95**, 220-28

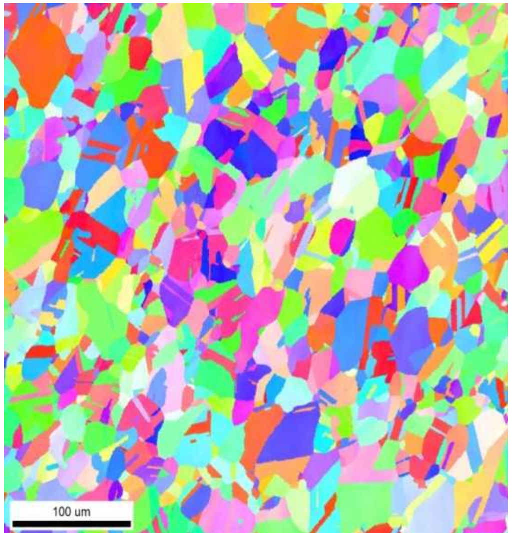
# Thermomechanical Processing

Grain boundary engineering (GBE) uses **thermomechanical processing** to improve the properties by controlling the **grain boundary character distribution** (GBCD) and by increasing the percentage of special grain boundaries ($3 \leq \Sigma \leq 29$).



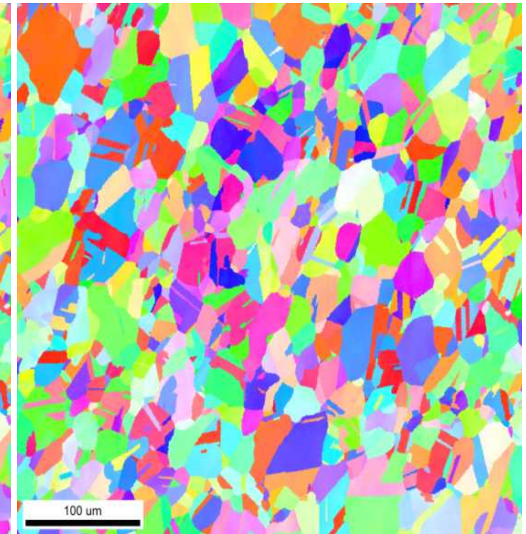

- TEM, Ni thin film (nanocrystalline), between 30° C and 600° C.
  - G.B. Thompson, J.G. Brons, ***Acta Materialia*** 61 (2013). 3936
- EBSD, 25% cold rolled pure Ni sample annealed at 490° C.
  - B. Lin, G.S. Rohrer, A.D. Rollett, Y. Jin, N. Bozzolo, M. Bernacki, ***Materials Science Forum*** 753 (2013) 97.
- Some experiments have shown that twinning does increase with grain size but only at particular strain-temperature combinations.
  - B.M. Guyot and N.L. Richards, ***Materials Science and Engineering A*** 395 (2005) 87.
  - V. Randle, ***Acta Materialia***, 52 (2004) 4067.
  - S. Lee and N.L. Richards, ***Materials Science and Engineering A*** 390 (2005) 81.
  - B.M. Guyot, S. Lee, and N.L. Richards, ***Journal of Materials Engineering and Performance*** 14 (2005) 85.
  - S. Lee and N.L. Richards, ***Materials Science and Engineering A*** 405 (2005) 74.
- High strain studies on polycrystalline alloys have also shown slight decreases in the fraction of special grain boundaries while in the presence of AGG.
  - A.J. Schwartz, W.E. King, M. Kumar, ***Scripta Materialia*** 54, 963-68 (2006)
- Strain increases precipitates a decrease in the onset temperature for AGG.
  - O.D. Underwood: An Investigation of Grain Boundary Character Evolution in Nickel 200. Ph.D. Thesis, (2015)
  - S.B. Lee, N.M. Hwang, D.Y. Yoon, M.F. Henry, ***Metallurgical and Materials A*** **31A**, 985-93 (2000)
  - J. Dennis, P.S. Bate, F.J. Humphreys, ***Materials Science Forum*** **558-889**, 717-22 (2007)

# Preliminary Survey- 3% Strain

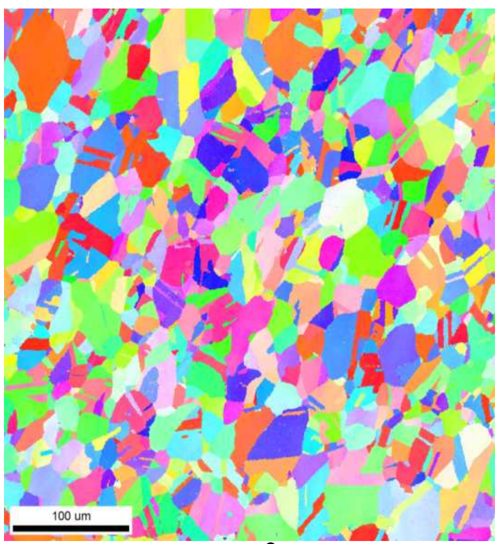



a. 25° C

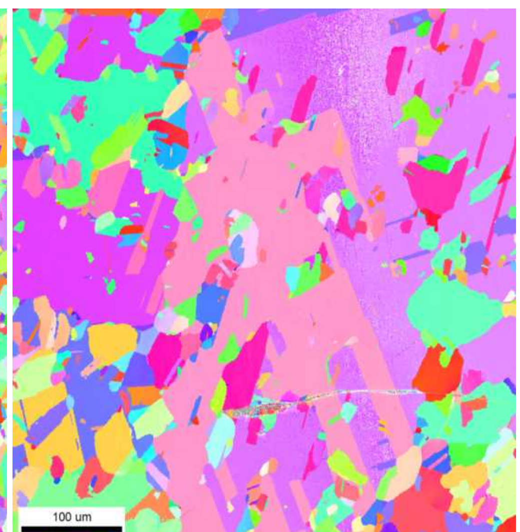

b. 200° C



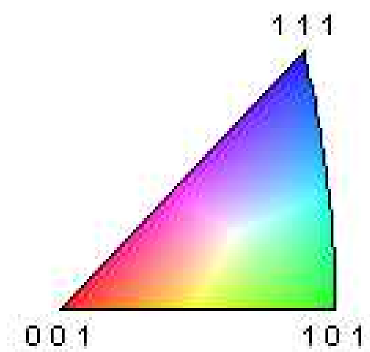

c. 400° C




d. 600° C




e. 800° C



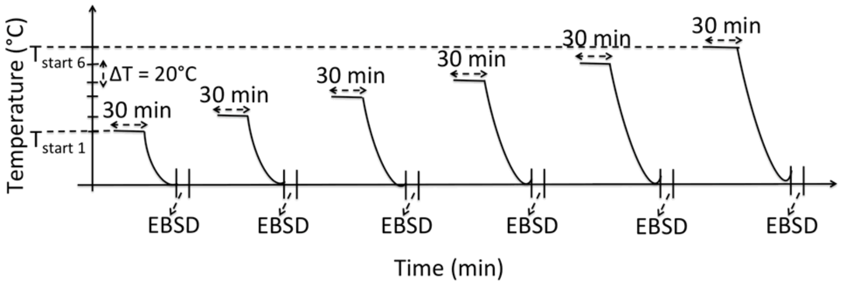

Onset of grain growth occurred between 600-800° C.

# Experimental Approach

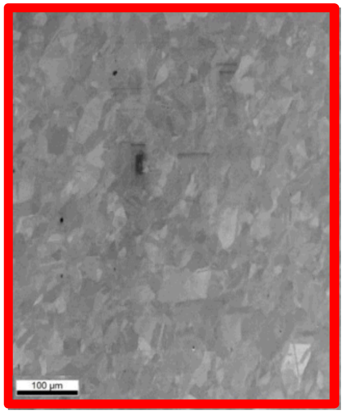
- Ni 200 (commercial alloy)

| C | S | Mn | Si | Ti | Cu | Fe | Mg | Co | Ni |
|---|---|---|---|---|---|---|---|---|---|
| 0.01 | <0.002 | 0.09 | 0.05 | 0.01 | 0.01 | 0.03 | 0.02 | 0.01 | R99.7 |

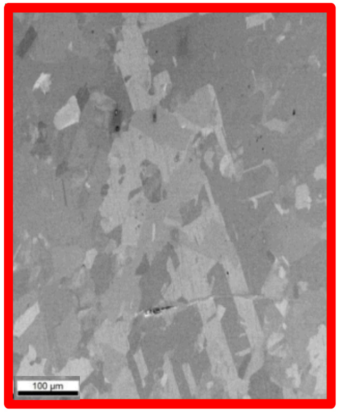
- Initial 6.35mm thick plate
- Rolled to 3%, 6%, 9% compressive strain
- Annealed at 25ºC (room temperature) and 700ºC through 800ºC for 30 minutes




- Focused on same region of interest (ROI) between annealing cycles.

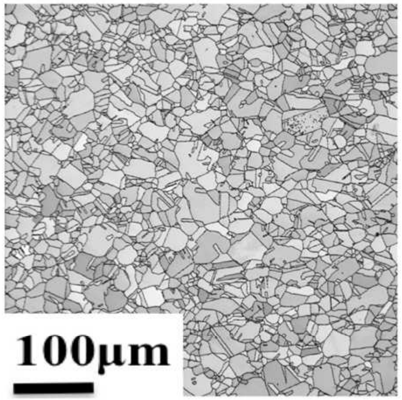


SEM image of the region of interest for 3% strain at 25° C

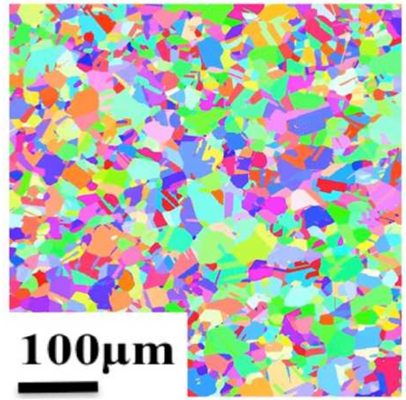


SEM image of the region of interest for 3% strain at 800° C

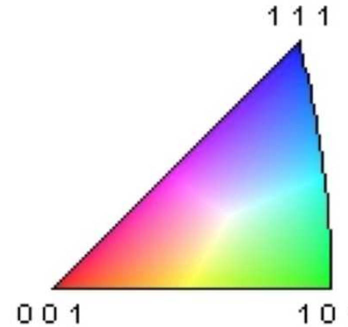
# Baseline Grain Boundary Metrics



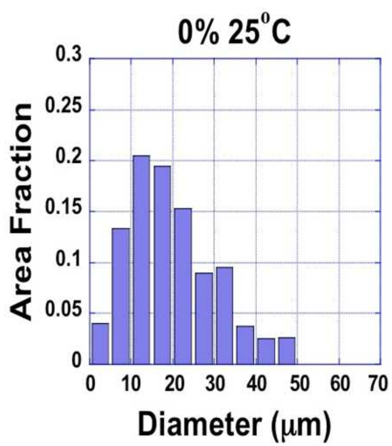

(a)



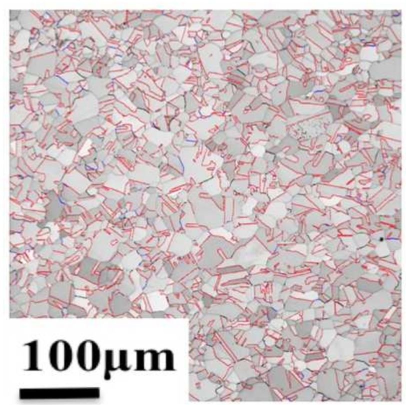

(b)



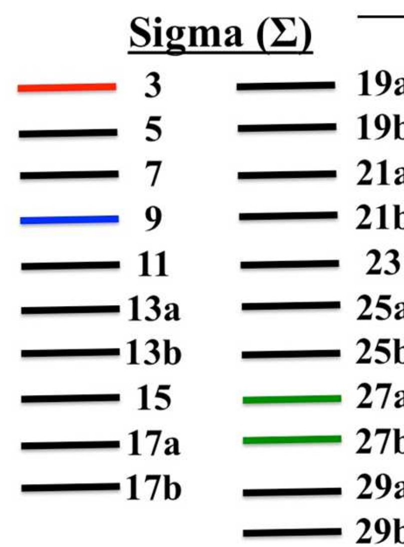

| Induced Compressive Strain (%) | Initial Avg. Grain Size (μm) |
|---|---|
| 0% | 7.1 |
| 3% | 7.4 |
| 6% | 7.6 |
| 9% | 13.8 |




(c)

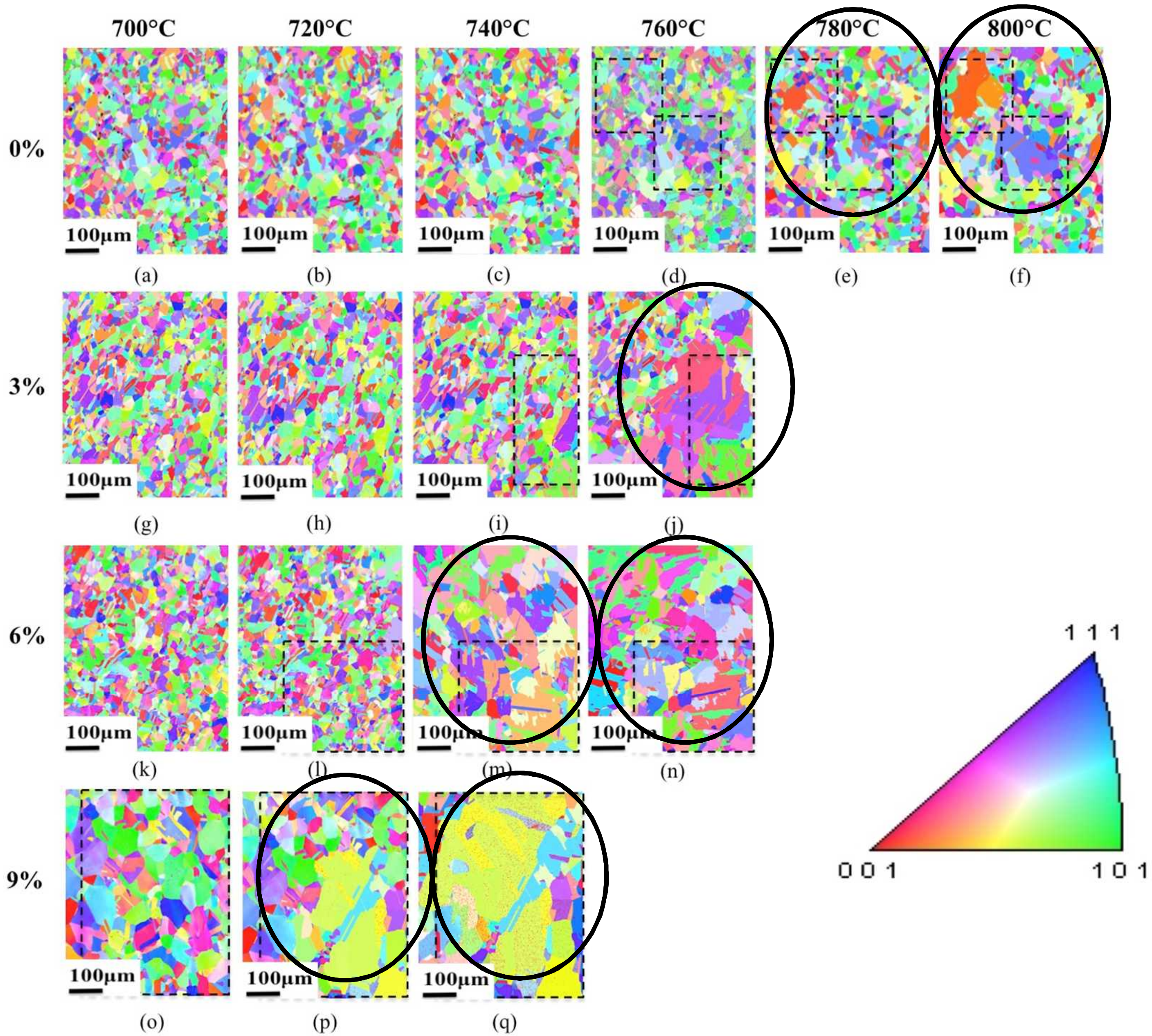



(d)

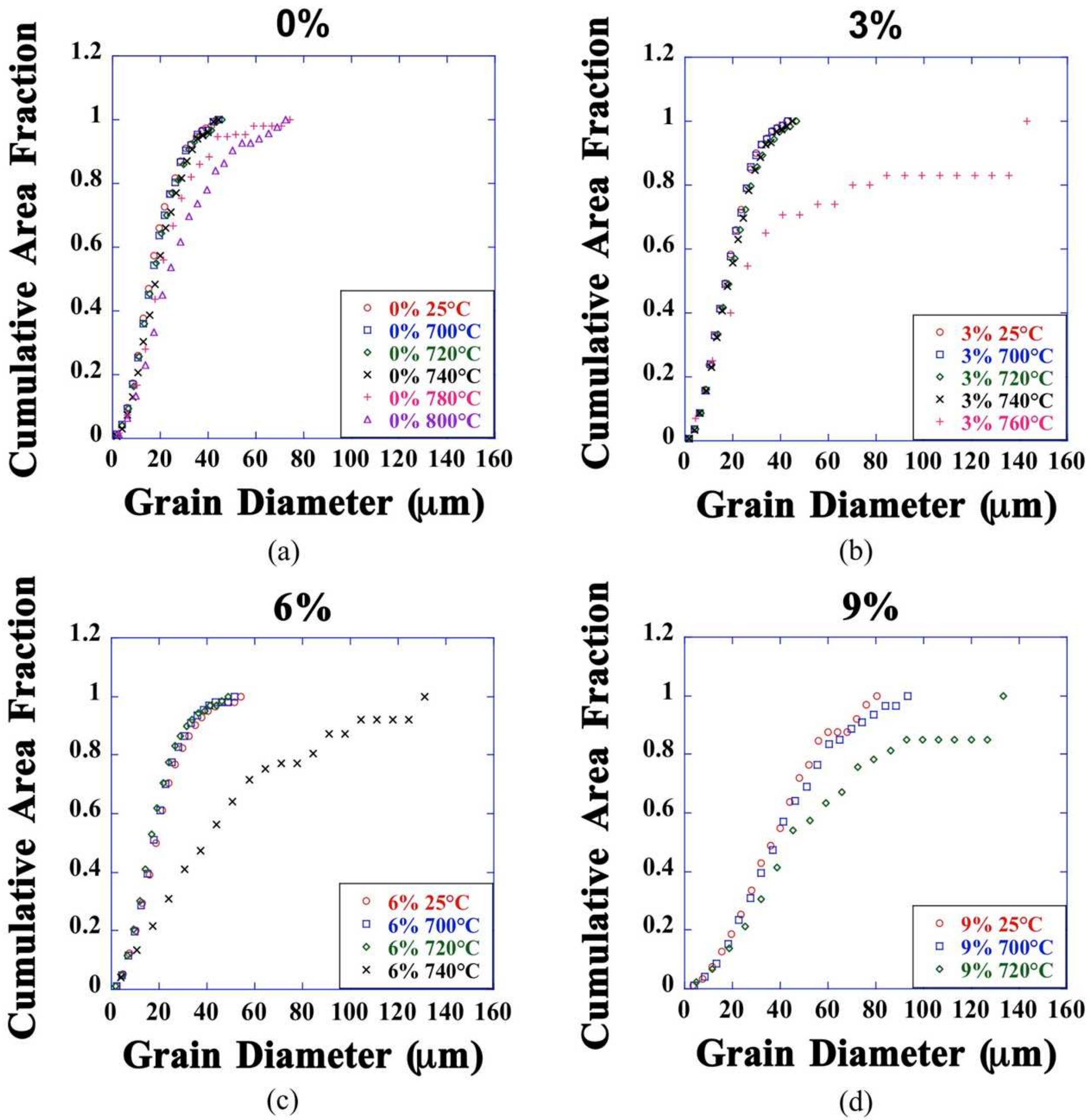



Average grain size = 7μm

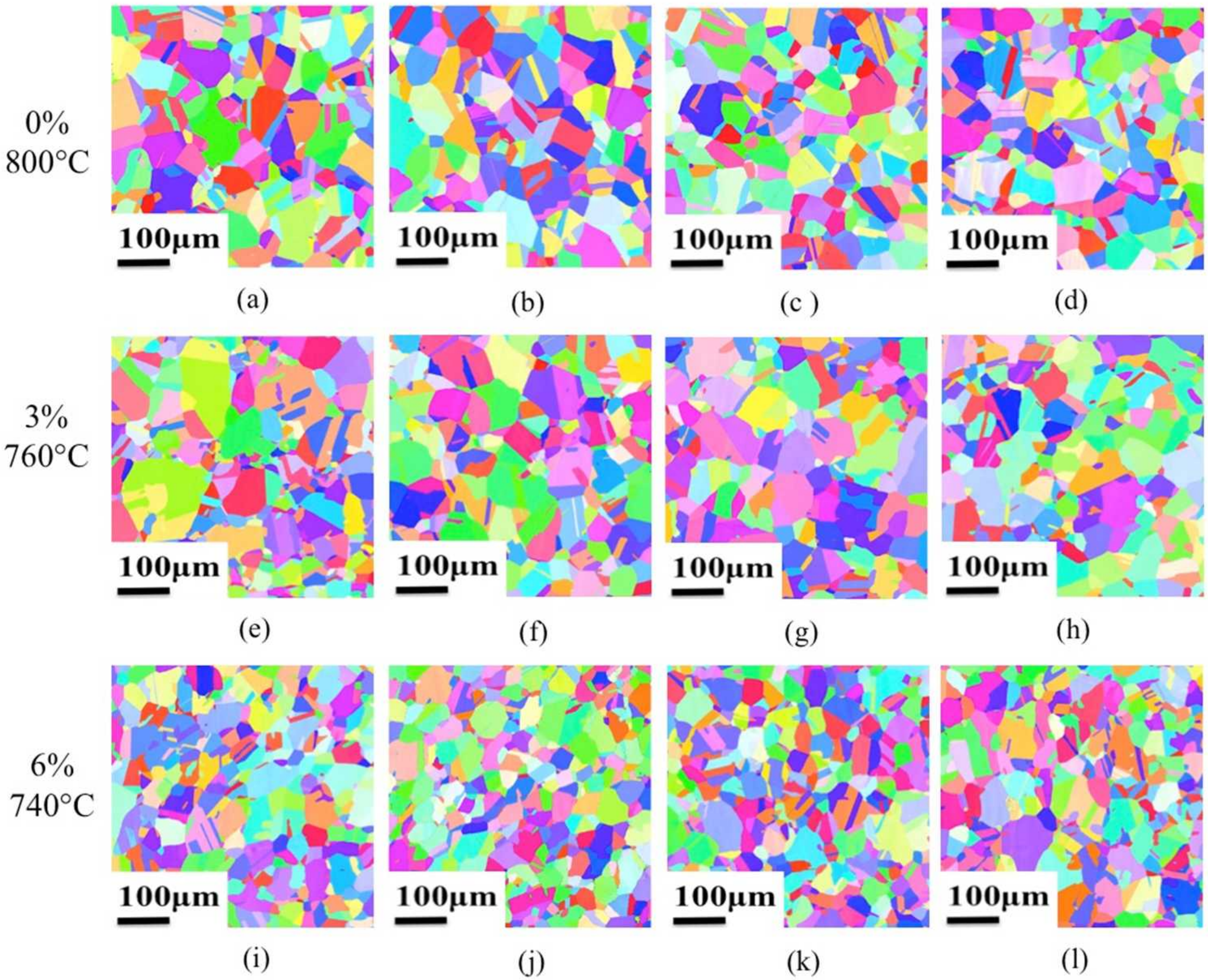
# EBSD Progression Maps



# Cumulative Area Fractions



# Isothermal Anneals




Four different samples annealed at the temperature which AGG was observed for 0%, 3%, and 6% strain cases.



1. Sahay, S.S., Malhotra, C.P., Kolkhede, A.M.: Accelerated Grain Growth Behavior During Cyclic Annealing. 51, 339-46 (2003)

# Comparison With Isothermal Anneal

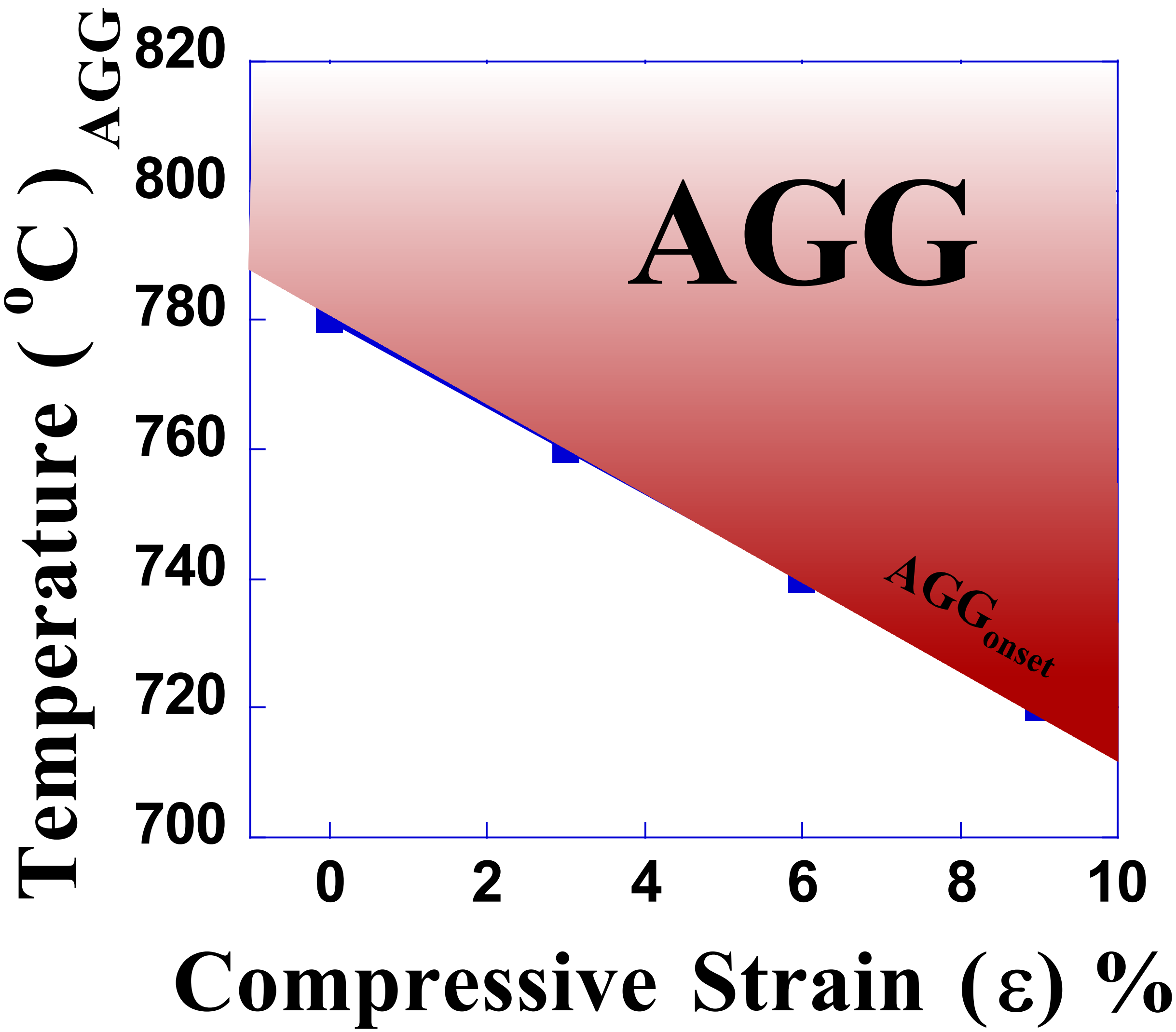
| Induced Compressive Strain (%) | Initial Avg. Grain Size (µm) | Thermal Anneal (°C) | Isothermal Anneal Avg. Grain Size (µm) [Measured] | Cyclic Anneal Avg. Grain Size (µm) [Measured] |
|---|---|---|---|---|
| 0% | 7.1 | 800 | 20.4 **~3X** | 8.6 **(-)** |
| 3% | 7.4 | 760 | 16.3 **~2.5X** | 7.6 **(-)** |
| 6% | 7.6 | 740 | 14.9 **~2X** | 7.5 **(-)** |
| 9% | 13.8 | 720 | -- | 12.3 **(-)** |

**(-) No significant change**

**-Average grain size increased 2X – 3X for isothermal anneal.**

**-Average grain size did not increase significantly for cyclic annealing.**

# Empirical Relation



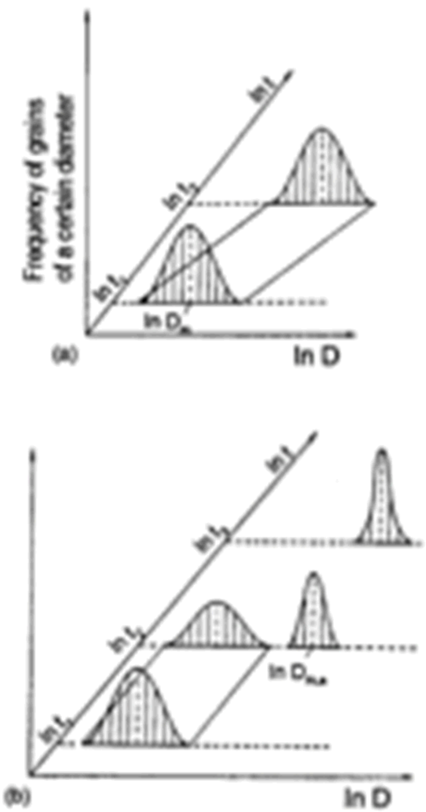

- $T_{AGG}$ = AGG Observation Temperature
- n = number of thermal cycles
- ($\varepsilon_c$= initial induced compressive strain)

$$n = 5 - (\varepsilon_c/3)$$

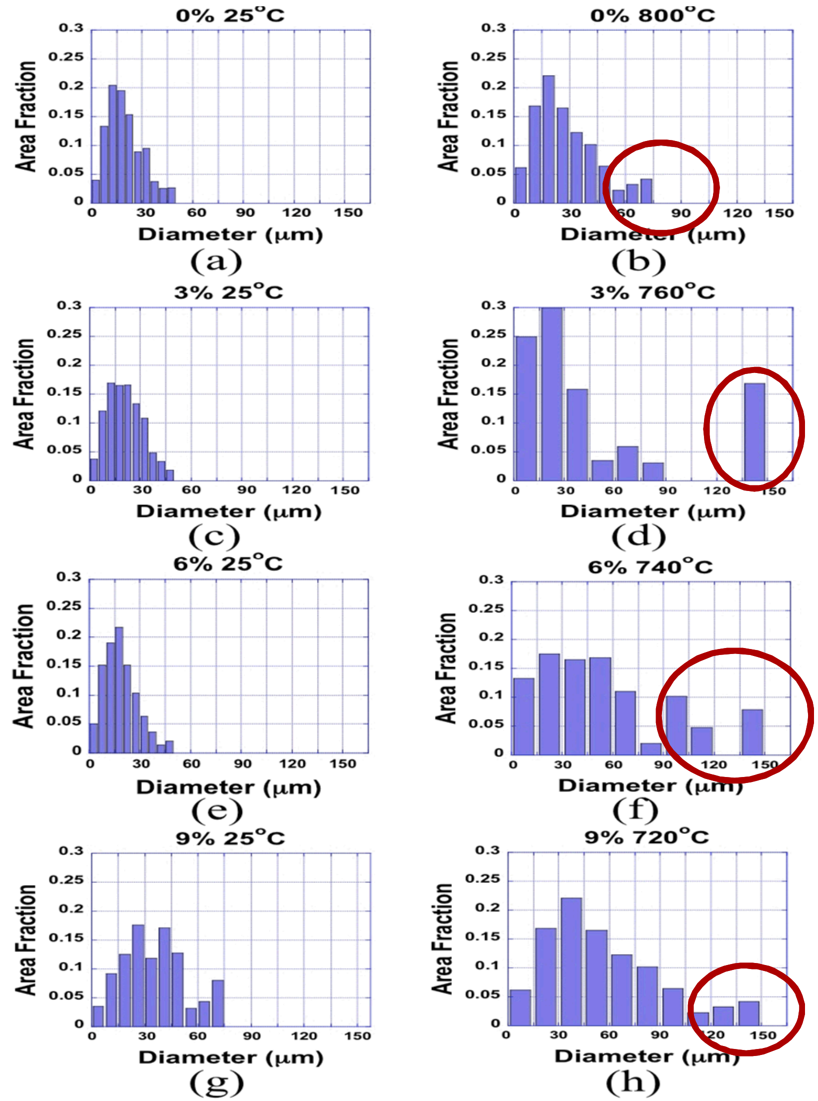
$$T_{AGG} \geq 20(5 - \varepsilon_c/3) + 680$$

Onset of AGG as a function of strain and annealing temperature

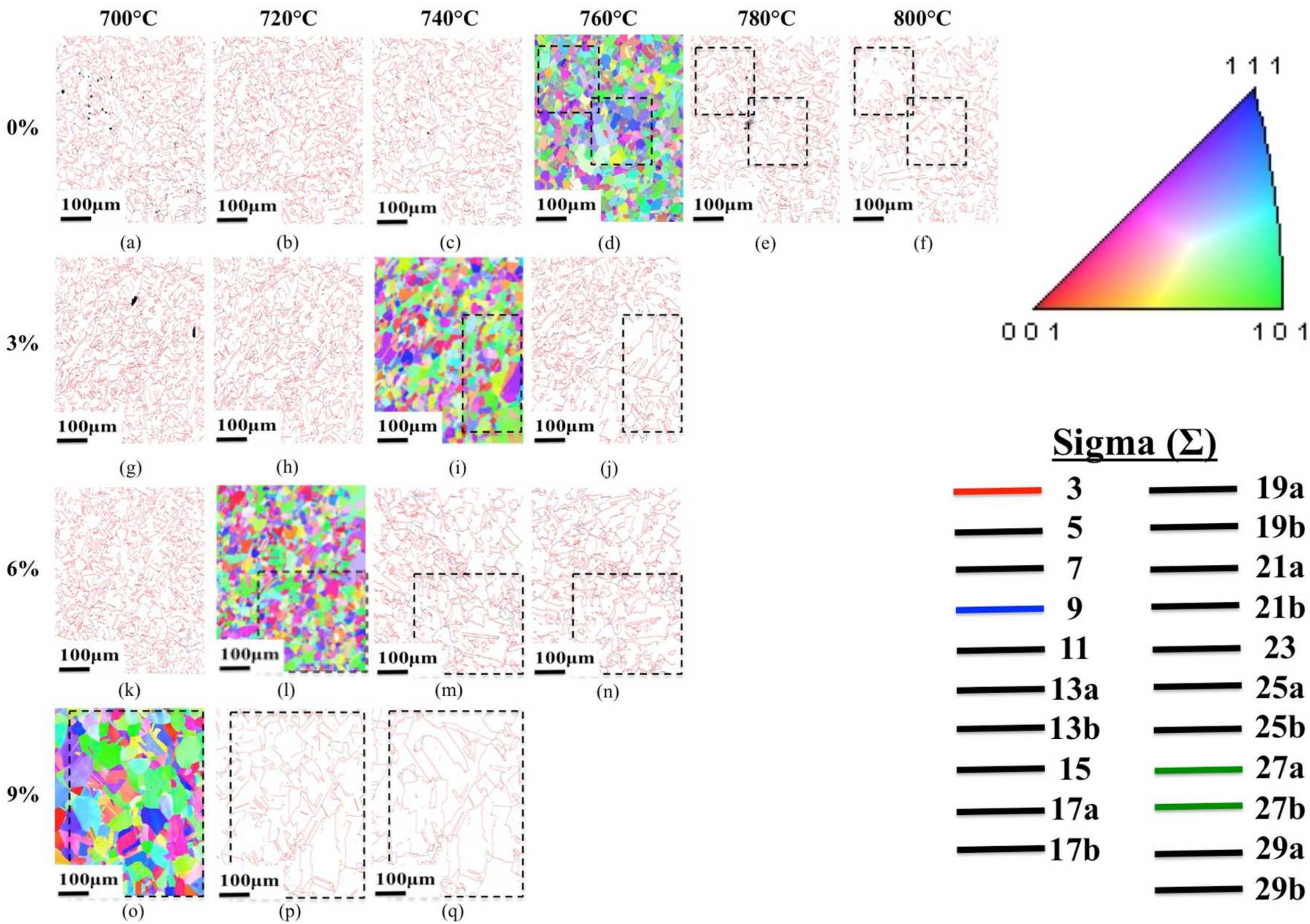
# Comparison of Grain Size



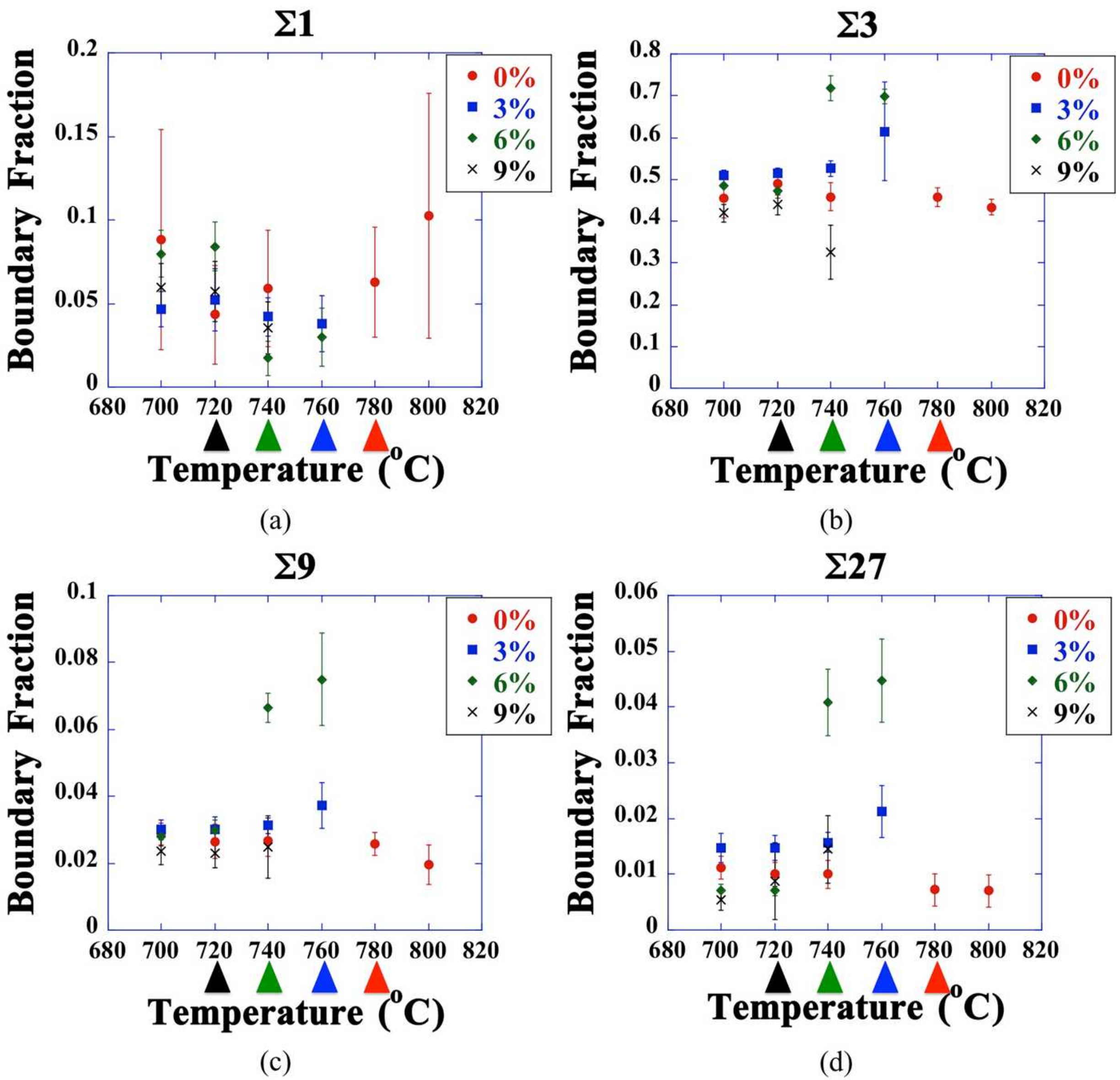

G. Gottstein, Physical Foundations of Materials Science (2004)



# CSL Boundary Maps



# CSL Boundary Fractions



# Conclusions

- As imposed strain was increased, temperature for the onset of AGG decreased to successively lower temperatures following cyclic heat treatment.

- Cumulative area fractions and grain size distributions confirmed a clear correlation between $AGG_{onset}$, annealing temperature, and compressive strain.

- An empirical predictor for the onset of AGG was derived as a function of annealing temperature, quantity of thermal cycles imposed, and the induced compressive strain. This empirical model takes the following form: $T_{AGG} \geq 20(5-\varepsilon_c/3)+680$ where $T_{AGG}$ is the AGG observation temperature, $e_c$ is the initial induced compressive strain and $e_c$ is related to the number of thermal cycles ($n$) by the following relation: $n=5-(\varepsilon_C/3)$.

- While AGG was observed to occur throughout the low strain regime, ($0 < \varepsilon_C < 10\%$), anticipated increases of Σ3, Σ9, and Σ27 were only observed as local maxima and occurred at or immediately after the AGG-onset temperature.

- The onset of AGG in the current study is complimentary to other high strain thermo-mechanical treatments in terms of the qualitative correspondence of AGG with temperature, strain, and thermal cycling. However, low strained Nickel-200 appears to have a higher suceptibility to AGG at shorter thermal dwell times when compared to scenarios of high strain.