Measurement of Grain Size in Twinned FCC Metals

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Grain Size Measurement

Types of Grain Sizes

- Non-twinned (ferrite, BCC metals, Al)
- Twinned FCC Metals (austenite)
- Prior-Austenite
  (Parent Phase in Q&T Steels)
Intercept Grain Size Example: Single Phase Twinned Grain Structure

The 100X micrograph is that of a twinned FCC Ni-base superalloy, X-750, in the solution annealed and aged condition after etching with Beraha’s reagent which colored the grains. This is a much more difficult microstructure for intercept counting. The three circles measure 500 mm and P is 63 (intersections with twin boundaries are ignored).
This is a 100X micrograph of 304 stainless steel etched electrolytically with 60% HNO$_3$ (0.6 V dc, 120 s, Pt cathode) to suppress etching of the twin boundaries. The three circles have a total circumference of 500 mm. A count of the grain boundary intersections yielded 75 (P=75).
Intercept Grain Size Example – Single Phase

\[ P_L = \frac{75}{500/100} = 15 \text{ mm}^{-1} \]

\[ l = \frac{1}{15} = 0.067 \text{ mm} \]

\[ G = [-6.644 \log_{10}(0.067)] - 3.288 = 4.5 \]
Grain Size Comparison Chart Ratings

Highly subjective

Precision ?

Biased?

Is the chart properly graded?

How well do the images reveal the grain and twin boundaries?
Twinning Frequency

How much does it vary with:

Grain boundary energy

Stacking fault energy

Grain size

Prior deformation
Twin frequency is proportional to grain boundary energy and inversely proportional to the stacking fault energy.

Twin frequency has been claimed to increase with increasing grain size, or is independent of grain size – or, it increases with grain size and then decreases with further increases in grain size!
Studies on the influence of deformation have claimed that prior strains of 5-20% produced a greater twin frequency than greater levels of prior strain.

Temperature of straining seems to have little influence.

Compositional differences appear to influence twin frequency.

“Study of the influence of grain and twin boundaries on the hardness of recrystallized cartridge brass, Cu – 30% Zn”
Babyak & Rhines – Cartridge Brass

Babyak and Rhines, GBs Only

![Graph showing the relationship between ASTM Grain Size Number, G, and N_L, No. per mm. The equation is G = 0.214N_L + 0.748.]

Babyak + Rhines, GBs Only

![Graph showing the relationship between ASTM Grain Size, G, and MLI, Ī, mm. The equation is G = (-6.644Log Ī) - 3.288.]

R^2 = 0.9275 (N_L) and 1.0 (Log of L – per E 112)
Babyak & Rhines – Cartridge Brass

\[ G = 0.161N_L' + 0.10 \]

\[ G = (\frac{-7.717\log \bar{I}}{\log}) - 6.24 \]

\[ R^2 = 0.9529 \text{ (for } N_L' \text{)} \text{ and } 0.9835 \text{ (for } \log L \text{)} \]
Babyak & Rhines – Cartridge Brass

Babyak and Rhines

\[ G = 0.161N_L' + 0.10 \]
\[ G = 0.214N_L + 0.748 \]

\[ R^2 = 0.9529 \text{ (GBs + TBs)} \] and \[ 0.9275 \text{ (GBs Only)} \]

Strictly speaking, a linear fit should not be done.
Babyak & Rhines – Cartridge Brass

G = (6.644 \log N_L) - 3.288

G = (7.717 \log N_L') - 6.24

R^2 = 0.9835 (GBs + TBs) and 1.0 (GBs Only – per E 112)
Babyak & Rhines – Cartridge Brass

$G = (-6.644 \log L) - 3.288$

$G = (-7.717 \log L') - 6.24$

$R^2 = 1.0$ (GBs Only – per E 112) and 0.9835 (GBs + TBs)
Babyak & Rhines - Cartridge Brass

Babyak & Rhines, G vs G'

G = 1.161 G' - 2.42

R² = 0.9835 for G' vs. G

“The Formation of Low-Energy Interfaces During Grain Growth in Alpha and Alpha-Beta Brasses”
Hu and Smith, GBs Only

\[ G = (6.644 \log N_L) - 3.288 \]

Hu and Smith, GBs Only

\[ G = (-6.644 \log \bar{I}) - 3.288 \]
Hu & Smith – Cartridge Brass

Hu & Smith, Gbs + TBs

$G = (7.186 \log N_L') - 6.635$

Hu and Smith, GBs + TBs

$G = (7.186 \log \bar{l}') - 6.635$

$R^2 = 0.9988$ for both
Hu & Smith, GBs vs. GBs + TBs

G = 0.1376N_L - 0.00036 N_L^2 + 0.8655
G = 0.0745 N_L' - 0.000105N_L'^2 + 0.444

R^2 = 0.8643 (for N_L) and 0.8971 (for N_L')
Hu & Smith – Cartridge Brass

Hu and Smith, GBs vs. GBs+TBs

\[ G = 6.644 \log(N_L) - 3.288 \]

\[ G = 7.186 \log(N_L') - 6.635 \]

\[ R^2 = 1.0 \text{ (for } N_L \text{ per E 112) and 0.9988 (for } N_L' \text{)} \]
Hu & Smith – Cartridge Brass

Hu and Smith, GBs + TBs

\[ G = (-6.644 \log \bar{I}) - 3.288 \]

\[ G = (7.186 \log \bar{I}') - 6.635 \]

\[ R^2 = 1.0 \text{ (for MLI – per E 112)} \text{ and 0.9988 (for MLI')} \]
Hu & Smith – Cartridge Brass

Hu & Smith, G vs G'

\[ G = 1.082 \ G' - 3.08 \]

\[ R^2 = 0.9988 \text{ (slight better than 0.9835 for Babyak & Rhines)} \]
Comparison of $\alpha$-Brass Data

There is a consistent difference between these data sets.

“Control of Annealing Twins in Type 316 Austenitic Stainless Steel”
Varin & Kruszynska – 316 γSS

Varin & Kruszynska, GBs Only

\[ G = (6.644 \log N_L) - 3.288 \]

Varin & Kruszynska, GBs Only

\[ G = (6.644 \log \bar{I}) - 3.288 \]
Varin & Kruszynska – 316 γSS

Varin & Kruszynska, GBs+TBs

$G = (7.344 \log N_L') - 5.589$

Varin & Kruszynska, GBs + TBs

$G = (-7.344 \log \bar{I}') - 5.589$

$R^2 = 0.9733$ for both
Varin & Kruszynska – 316 γSS

Varin & Kruszynska, GBs+TBs

\[ G = (7.344 \log N_L') - 5.589 \]

\[ G = (6.644 \log N_L) - 3.288 \]
Varin & Kruszynska, Gbs+TBs

\[ G = (-6.644 \log \bar{I}) - 3.288 \]

\[ G = (-7.344 \log \bar{I}') - 5.589 \]

MLI, \( \bar{I} \) and \( \bar{I}' \), mm

ASTM Grain Size, G

GBs Only

GBs + TBs
Varin & Kruszynska – 316 γSS

ASTM Grain Size, G vs G'

G = 1.105 G' - 1.955
Vander Voort Data - γSS

<table>
<thead>
<tr>
<th>Element</th>
<th>316L</th>
<th>316</th>
<th>904L</th>
<th>Gall Tough</th>
<th>SCF-19</th>
<th>18-18+</th>
<th>20Mo6</th>
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<tbody>
<tr>
<td>C</td>
<td>0.015</td>
<td>0.05</td>
<td>0.018</td>
<td>0.103</td>
<td>0.034</td>
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<tr>
<td>Mn</td>
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<td>1.63</td>
<td>1.25</td>
<td>5.55</td>
<td>4.88</td>
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<tr>
<td>Si</td>
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<td>3.40</td>
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<tr>
<td>P</td>
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<td>0.025</td>
<td>0.025</td>
<td>0.020</td>
<td>0.028</td>
<td>0.020</td>
<td>0.021</td>
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<tr>
<td>S</td>
<td>0.025</td>
<td>0.020</td>
<td>0.001</td>
<td>0.023</td>
<td>0.006</td>
<td>0.004</td>
<td>0.004</td>
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<tr>
<td>Cr</td>
<td>16.30</td>
<td>17.29</td>
<td>19.92</td>
<td>16.21</td>
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<td>17.78</td>
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<tr>
<td>Ni</td>
<td>12.60</td>
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<td>5.04</td>
<td>17.76</td>
<td>0.46</td>
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<tr>
<td>Mo</td>
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<td>2.14</td>
<td>4.09</td>
<td>0.26</td>
<td>5.14</td>
<td>1.09</td>
<td>5.55</td>
</tr>
<tr>
<td>Cu</td>
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<td>1.15</td>
<td>0.27</td>
<td>0.30</td>
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<tr>
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<td>0.30</td>
<td>-</td>
<td>0.12</td>
<td>0.01</td>
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<td>-</td>
<td>0.03</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>N</td>
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<td>0.042</td>
<td>0.038</td>
<td>0.11</td>
<td>0.36</td>
<td>0.45</td>
<td>-</td>
</tr>
</tbody>
</table>

3 specimens of Alloy 625, a Ni-based superalloy were also tested
This Work, GBs Only

Vander Voort Data - γSS
Vander Voort Data - γSS

This Work, GBs + TBs

This Work, GBs + TBs

\[ P'_L, \text{No. per mm} \]

\[ \text{ASTM Grain Size, G'} \]

\[ \text{MLI}, Ī', \text{mm} \]

\[ \text{ASTM Grain Size, G'} \]

- 316L
- 316
- 904L
- Gall Tough
- SCF-19
- 18-18+
- 625
- 20 Mo 6
For 316, 316L, 904L, Gall Tough, SCF-19 and 18-18+
\[ G = (-7.369 \log L') - 6.127 \quad (R^2 = 0.9005) \]

For the above plus 20Mo6
\[ G = (-7.414 \log L') - 6.2 \quad (R^2 = 0.8942) \]

Adding in the 3 data points for Alloy 625:
\[ G = (-6.9338 \log L') - 5.604 \quad (R^2 = 0.8791) \]
This Work, G vs G'

G = 1.044 G' – 2.172

R² = 0.8791
Comparison of Published Data

All Published Data, GBs Only

$G = (6.644 \log N_L) - 3.288$

MLI, Ī, mm

$G = (-6.644 \log \bar{L}) - 3.288$

Excellent fit when only grain boundaries are counted
Correlation is not as good between G and GBs and TBs. The austenitic stainless steels agree better with Babyak & Rhines’ brass data than with Hu & Smith’s brass data.
Comparison of Published Data

All Published Grain Size Data
GBs vs GBs + TBs

- Babyak&Rhines
- Hu&Smith
- Varin&Kruszynska
Comparison of $\gamma$SS Data

Comparison of $\gamma$SS Data

This Study

Varin & Kruszynska

Alloy 625 Data

GB+TB Grain Size, $G'$

ASTM Grain Size, $G$

This Study

Varin & Kruszynska
Comparison of All Data

This Work vs Published Data

ASTM Grain Size, G'

This Work

Babyak&Rhines

Hu&Smith

Varin&Kruszynska

Alloy 625 Data
Conclusions

- Overall, the calculation of ASTM G can be done with a count of grain boundaries and twin boundaries with acceptable precision for most work.

- Although there were only 3 data points for the Ni-based alloy 625, it appears that its twin frequency is somewhat different from the other FCC alloys.

- For highest precision, it is best to develop correlation equations for each major type of twinned FCC alloy.

- The two sets of $\gamma$ SS data were in better agreement than the two sets of cartridge brass data.