SAND2010-5664P

Material Characterization of High Rate Forged 304L Stainless Steel

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Outline

- Objective and Motivation
- Materials
- Procedure
- Experimental Data and Results
- Summary
- Sonny’s Presentation (title?)
- Albert’s Presentation (title?)

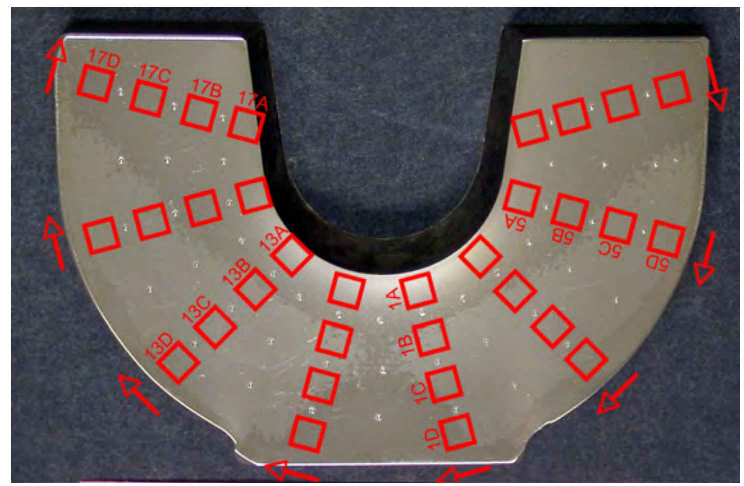
Motivation and Objective

Motivation

The forging process is intrinsically non-uniform, resulting in large variations of microstructural and mechanical properties in the forgings. Quantification of forging microstructure and hardness distribution provides a necessary scientific basis for understanding the performance of the materials

Objective

- To determine the distribution of the ASTM grain size through out the forgings using the three-circle intercept method from optical images of a polished forging cross section
- To determine the distribution of the hardness using the Rockwell Hardness Scale A indentation on the metallographic polished forging cross section



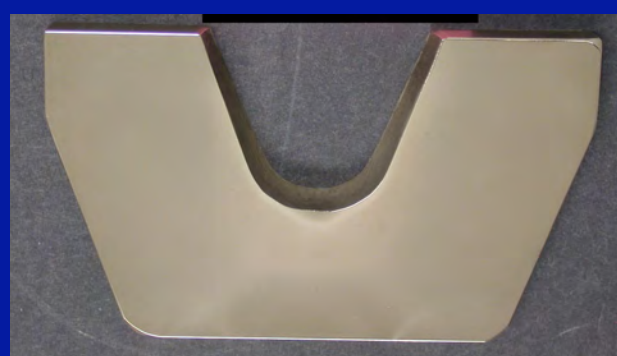
Materials

Three different types of forging designs made of the alloy 304L with typical composition of ≤.03% carbon, 18-20% chromium, ≤ 2% manganese, 8-10% nickel, and a balance of iron

1AO164

458591

7K0001



*photos courtesy of Chris San Marchi

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Experimental Procedure

<u>Sample preparation</u>

Forging cross section was prepared by a conventional metallographic polishing and etching using 75% nitric acid at 1.15 volts.

<u>Rockwell hardness measurement</u>

Rockwell hardness indentation was performed on the forging cross sections.

<u>Optical imaging</u>

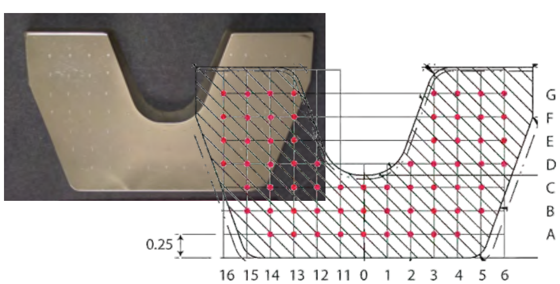
Optical images were obtained using Leitz Wetzlar optic microscope adjacent to each indent. (see the figure below)

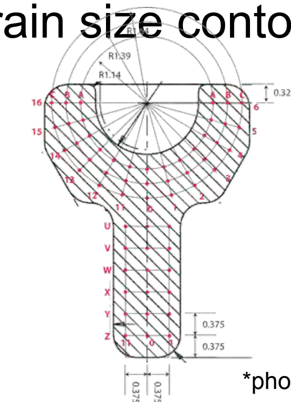
<u>Grain size measurement</u>

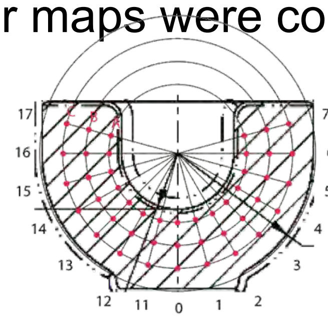
ASTM grain size was measured by the three circle intercept method from optical images

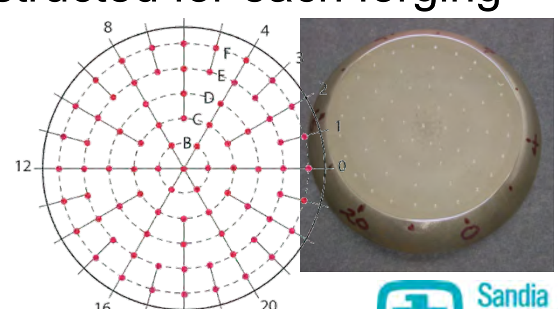
<u>Data analysis</u>

ASTM grain size and Rockwell hardness results were tabulated, plotted and the hardness and grain size contour maps were constructed for each forging









*photos and grids courtesy of Chris San Marchi

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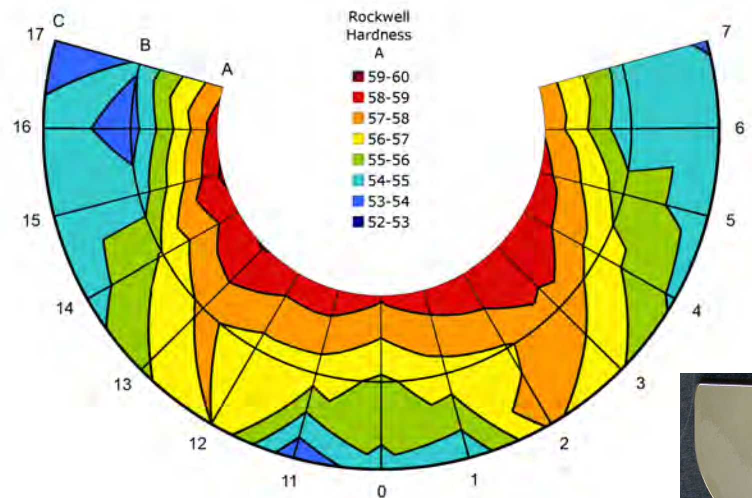
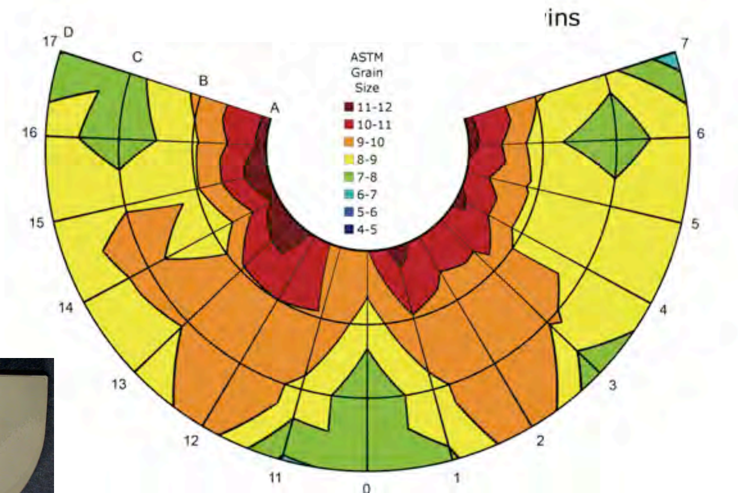
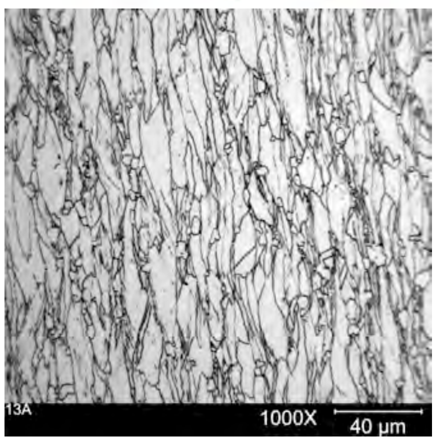
Typical Experimental Output

1AO164 Rockwell Hardness Contour Map



1AO164 ASTM Grain Size Contour Map





Optical Image

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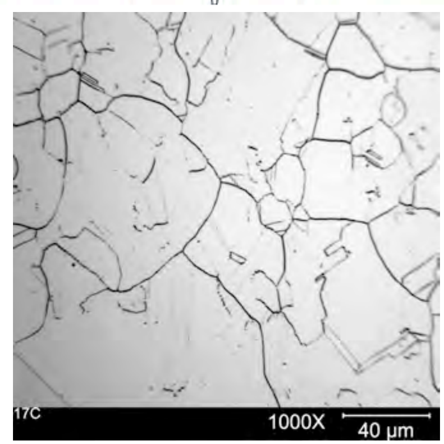
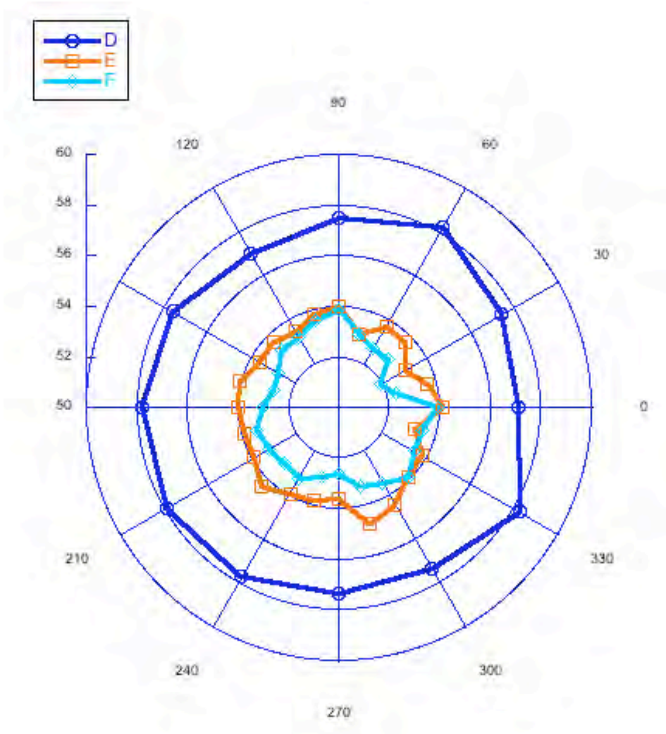


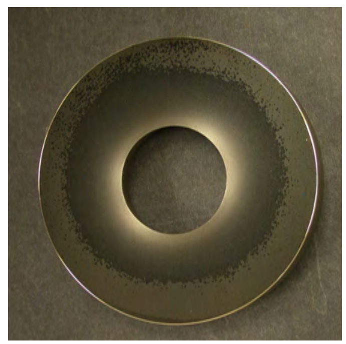
Point 13A is the harder spot, 59.1, with higher ASTM grain size, or small grain.

Point 17C is the soft spot, 53.0, with lower ASTM grain size, or large grain.

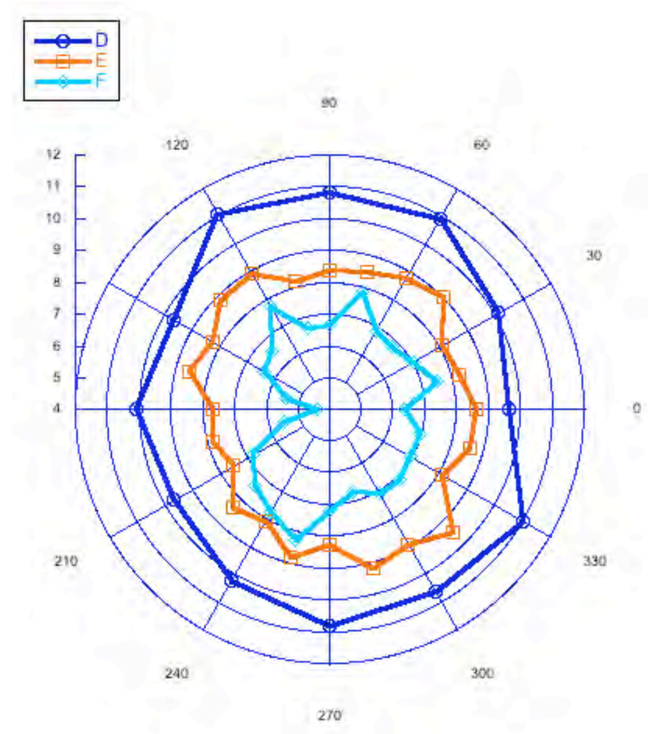
Typical Experimental Output (cont.)

<u>1AO164 Rockwell Hardness Polar Coordinate</u>





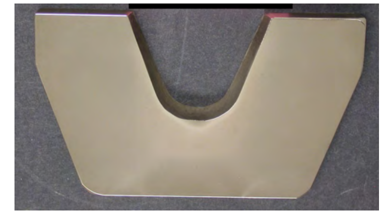
<u>1AO164 Grain Size Polar Coordinate</u>



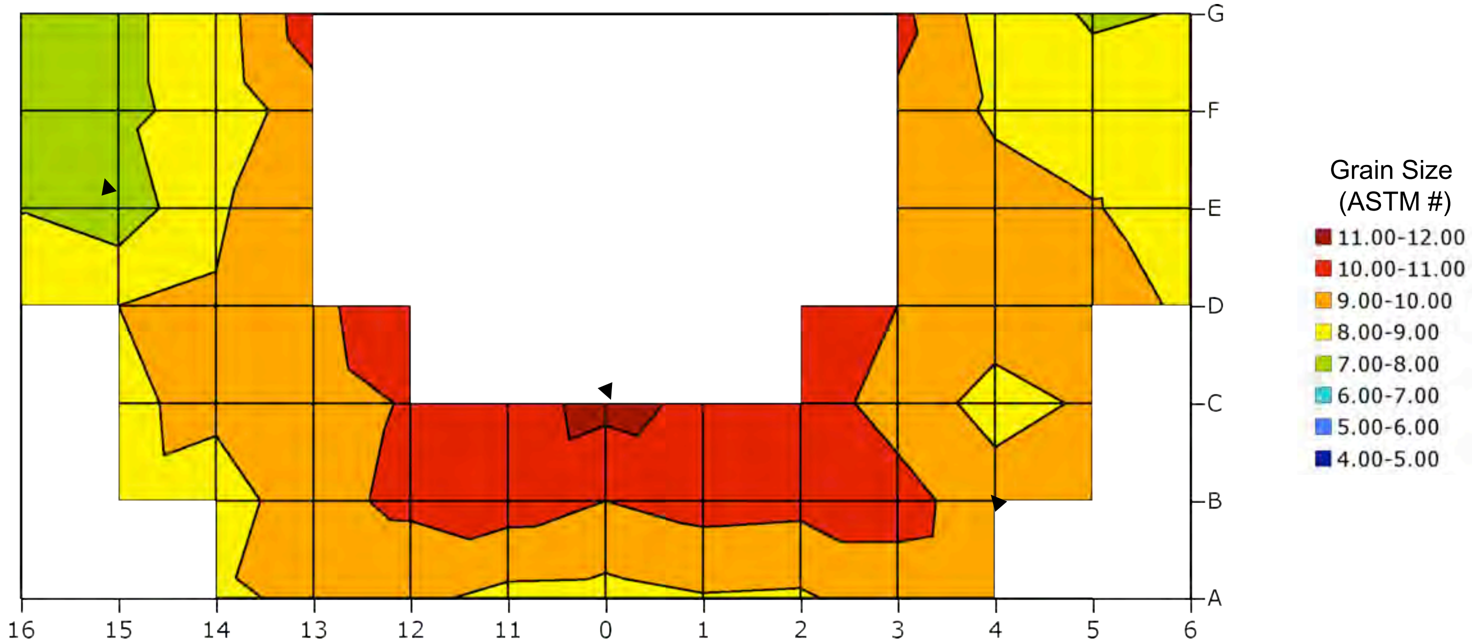
Each color represents a row in the forging. As the hardness increases, the ASTM grain size number also increases.

Typical Experimental Output (cont.)

7K0001 ASTM Grain Size Contour Map



There are smaller grains where the forging was strained due to the punching process



Where very little compression occurred, the grain size is large

There are small grains where the forging was compressed in a dye

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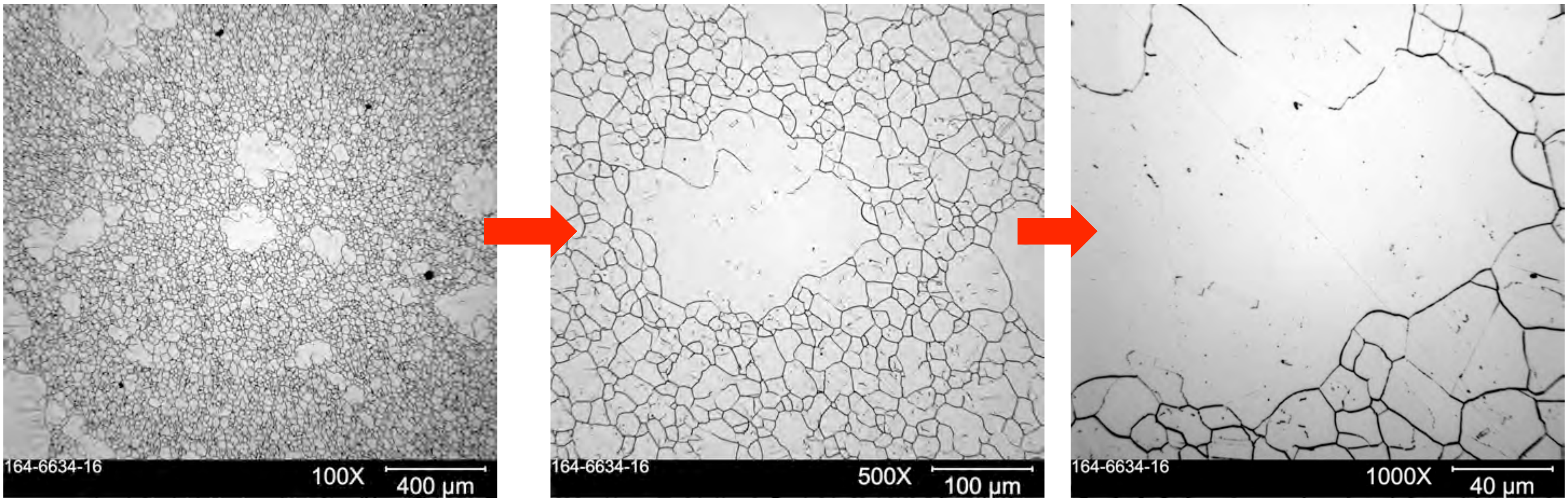
Bimodal Distributions of Grain Size

Optical Images of AO1164 14D



Bimodal grain structures were seen in the forging design AO1164. A statistical representative ASTM grain size and hardness numbers is hard to reach due to their inconsistency with the surrounding area. The scientist would need to determine whether to use an average ASTM grain size number or treat the points individually.

Summary

•The forgings' microstructure, with respect to grain size, and its hardness are found to be non-uniform.

•There is a common trend found among the forgings: the hardness is directly related to the ASTM grain size number. The larger the ASTM grain size number (i.e smaller grains) the harder the area.

•The inner surfaces of the forgings or the areas which correspond to high strain and compression locations during the forging process, have a large ASTM grain size number and tend to be harder than the surrounding areas.

Material Characterization of High Rate Forged 304L Stainless Steel

Lauren Hughes Mills College
Mentors: Nancy Yang 08651 Chris San Marchi 08222
Sandia National Laboratories, CA

Introduction

Austenitic stainless steels are commonly used in high-pressure gaseous hydrogen environments due to their resistance to hydrogen embrittlement. As hydrogen fuel cell technologies advance, the use of austenitic stainless steels is expected to grow. While designs of hydrogen systems can be constrained by the low strength of austenitic stainless steels, high-energy rate forging can be used to increase the strength of austenitic stainless steels. The forging process, however, is intrinsically non-uniform and results in large variations of microstructural and mechanical properties in the forgings. The microstructure and hardness were characterized as a function of location in several forgings to determine the distribution of properties in the forgings. The hardness was measured using standard Rockwell Hardness Scale A with spacing measurements approximately 6mm apart. The ASTM grain size was measured with similar spacing using the three-circle method. Grain size was measured with and without consideration of the annealing twins as grain boundaries. Three forging designs were examined over an axial cross section. One forging design was examined over several radial cross sections. Replicate forgings were analyzed for each condition. Mapping of the hardness and grain size show the microstructure and hardness to be non-uniform. The analysis also appears to show a correlation between grain size and hardness: the harder the areas have smaller grains.

Objective

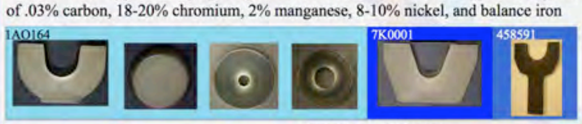
The objective is to measure the ASTM grain size through out the forgings using the three-circle intercept method from the optical images of a polished forging cross section and to measure the hardness using the Rockwell Hardness Scale A indentation on the metallographic polished forging cross section.

Procedure

The forging cross section was prepared by a conventional metallographic polishing and etching using 75% nitric acid at 1.15 volts. Rockwell hardness indentation was performed on the forging cross sections. Optical images were obtained using Leitz Wetzlar optic microscope adjacent to each indent. (see the figure below). ASTM grain size was measured by the three circle intercept method from optical images. ASTM grain size and Rockwell hardness results were tabulated, plotted and the hardness and grain size contour maps were constructed for each forging.

Materials

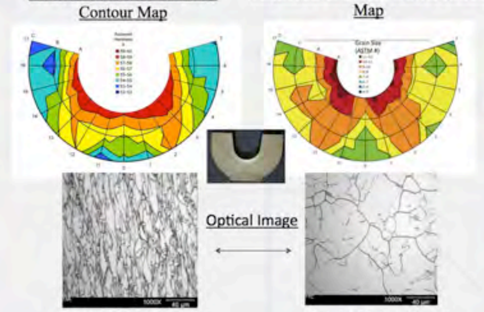
Three different types of forging designs made of the alloy 304L with typical composition of .03% carbon, 18-20% chromium, 2% manganese, 8-10% nickel, and balance iron



Photos and grids courtesy of Chris San Marchi

Experimental Results

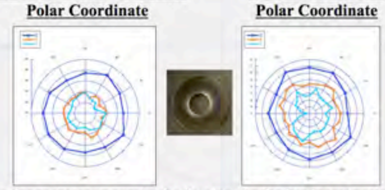
1A0164 Rockwell Hardness Contour Map | 1A0164 ASTM Grain Size Contour Map



Point 13A is the harder spot with higher ASTM grain size, or small grain.

Point 17C is soft spot with lower ASTM grain size, or large grain.

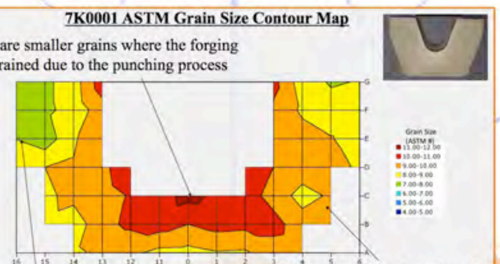
1A0164 Rockwell Hardness Polar Coordinate | **1A0164 Grain Size Polar Coordinate**



Each color represent a row in the forging. As the hardness increases, the ASTM grain size number also increases.

7K0001 ASTM Grain Size Contour Map

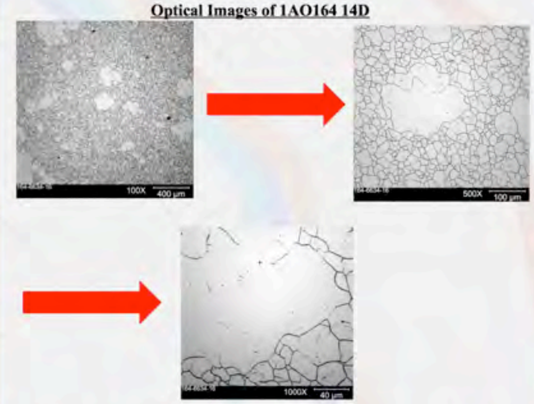
There are smaller grains where the forging was strained due to the punching process



Where very little compression occurred, the grain size is large

There are small grains where the forging was compressed in a dye.

Optical Images of 1A0164 14D



Bimodal grain structures were seen in in the forging 1A0164. A statistical representative ASTM grain size and hardness number is hard to reach because these large grains tend to be softer and have inconsistent ASTM grain size numbers in comparison to surrounding area.

Conclusion

The forgings' microstructure, with respect to grain size, and its hardness are found to be non-uniform.

There is a common trend found among the forgings: the hardness is directly related to the ASTM grain size number. The larger the ASTM grain size number (i.e smaller grains) the harder the area.

The inner surfaces of the forgings or the areas, which correspond to high strain and compression locations during the forging process, have a large ASTM grain size number and tend to be harder than the surrounding areas.

There are bimodal distributions of grain size within multiple forgings, which brought a challenge to the ASTM grain size and hardness measurements because these bimodal distributions do not represent the forging as a whole.

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Material Development and Characterization of Stainless Steel used for Hydrogen Gas Storage

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Mentors: Nancy Yang Org. 8651
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Sandia National Laboratories, CA
U.S. Department of Energy

National Nuclear Security Administration

Austenitic stainless steels are commonly used in high-pressure gaseous hydrogen environments due to their resistance to hydrogen embrittlement. As hydrogen fuel cell technologies advance, the use of austenitic stainless steels is expected to grow. While designs of hydrogen systems can be constrained by the low strength of austenitic stainless steels, high-energy rate forging can be used to increase the strength of austenitic stainless steels. The forging process, however, is intrinsically non-uniform and results in large variations of microstructural and mechanical properties in the forgings. The microstructure and hardness were characterized as a function of location in several forgings to determine the distribution of properties in the forgings. The hardness was measured using standard Rockwell Hardness Scale A with spacing measurements approximately 6mm apart. The ASTM grain size was measured with similar spacing using the three-circle method. Grain size was measured with and without consideration of the annealing twins as grain boundaries. Three forging designs were examined over an axial cross section. One forging design was examined over several radial cross sections. Replicate forgings were analyzed for each condition. Mapping of the hardness and grain size show the microstructure and hardness to be non-uniform. The analysis also appears to show a correlation between grain size and hardness: the harder the areas have smaller grains.

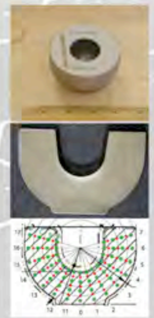
Procedure

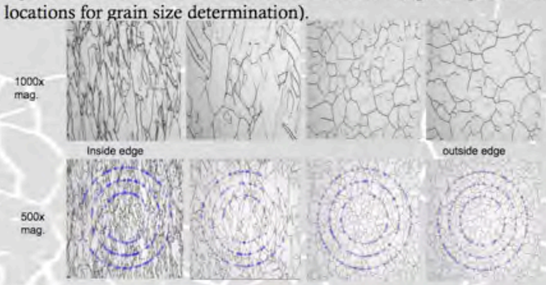
<u>Sample preparation</u>

The steel forging was made with alloy 304L with typical composition of .03% carbon, 18-20% chromium, 2% manganese, 8-10% nickel, 1% silicon, and 67.97-70.97% iron The cross section was prepared by a conventional metallographic polishing and etching using 75% nitric acid at 1.15 volts.

<u>Rockwell hardness measurement</u>

Rockwell scale A - diamond cone indenter, 10kgf minor load, 60kgf major load. Rockwell hardness indentation was performed on the forging cross sections according to the grid to the right (red points represent the locations for hardness indentation, green points are locations for grain size determination).





<u>Optical imaging</u>

Optical metallographic images were obtained using Leitz Wetzlar optic microscope adjacent to each indent. (see the figure above) Grain size imaging along the inside rows reveals non-equiaxed grains

<u>Grain size measurement</u>

ASTM grain size was measured by the Abrams three circle intercept method from optical images per ASTM E112. Larger ASTM values translate to smaller grains.

$$G = (-6.6457\log_{10} L_m) - 3.298$$

$$NL = \frac{[\#\ \text{intercepts}]}{[length]/[mag]} \qquad L_m = \frac{1}{NL}$$

NL = average number of intercepts per unit length
L_m = average grain intercept length
G = ASTM grain size number

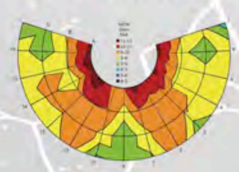
<u>Data analysis</u>

ASTM grain size and Rockwell hardness results were tabulated, plotted and the hardness and grain size contour maps were constructed for the forging.

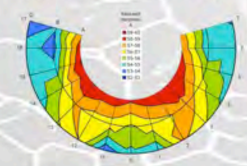
Results

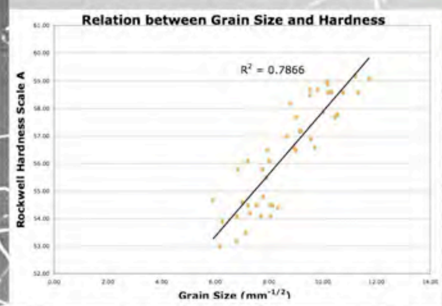
Visual examination of a contour plot of ASTM grain size with that of Rockwell Hardness Scale A suggests a correlation among the two values

ASTM Grain Size Contour Plot



Rockwell Hardness A Contour Plot





Both hardness and grain size correlate to the strength of a material. As a rule of thumb for most steels, TS (MPa) is 3.5x the Brinell Hardness. Also, Hall-Petch relationship predicts increase strength of a material with decreasing grain size. However, at a certain limit grain boundaries slide instead of impeding dislocation propagation.

$$\sigma_y = \sigma_o + \frac{k_y}{\sqrt{d}}$$

d = average grain diameter
σ = yield stress
k_y = strengthening coefficient, specific to the material

Discussion and Summary

- The shortest grain lengths measured were on the order of 10nm. It is believed that grain sizes smaller than 10nm would surpass the limit of strengthening by grain size reduction
- In this particular forging cross-section, moderately strong correlation exists between Hardness and grain length. Correlation between Rockwell Hardness A and average grain length is not significant enough to interpolate one value from the other with in a particular sample.
- Hardness and Grain Size data cannot be compared over a range of forging processes (different degree of work-hardening, recrystallization of grain size, bimodal grain size distribution, etc) Hardness and Grain Size data from other samples of different forging processes was collected, resulting in a weak correlation across the 12 forgings.

Ultimately, the material properties are characterized and tailored to optimize performance and reliability of the steel forging for use in hydrogen gas storage.

UCLA Engineering
HENRY SAMUELI SCHOOL OF ENGINEERING AND APPLIED SCIENCE

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8-12-10-AW-DP-LH-SZ

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Evaluation of Microstructural Features in Forged Austenitic Stainless Steel

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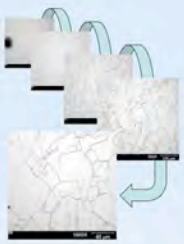
Chris San Marchi, Org. 08222
Sandia National Laboratories, CA

Abstract

Austenitic stainless steels are commonly used in high-pressure gaseous hydrogen environments due to their resistance to hydrogen embrittlement. As hydrogen fuel cell technologies advance, the use of austenitic stainless steels is expected to grow. While designs of hydrogen systems can be constrained by the low strength of austenitic stainless steels, high-energy rate forging can be used to increase the strength of austenitic stainless steels. The forging process, however, is intrinsically non-uniform and results in large variations of microstructural and mechanical properties in the forgings. The microstructure and hardness were characterized as a function of location in several forgings to determine the distribution of properties in the forgings. The hardness was measured using standard Rockwell Hardness Scale A with spacing measurements approximately 6mm apart. The ASTM grain size was measured with similar spacing using the three-circle method. Grain size was measured with and without consideration of the annealing twins as grain boundaries. Three forging designs were examined over an axial cross section. One forging design was examined over several radial cross sections. Replicate forgings were analyzed for each condition. Mapping of the hardness and grain size confirm that the microstructure and hardness are non-uniform. The analysis also appears to show a correlation between grain size and hardness: harder areas have smaller grains.

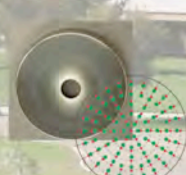
Procedure

All twelve samples (alloy 304L - ≤0.03% carbon, 18-20% chromium, ≤2% manganese, 8-10% nickel, balance iron) underwent conventional metallographic polishing and etching using 75% nitric acid at 1.15 volts. Hardness testing was then performed under the specifications dictated by Rockwell Hardness Scale A. Using the Leitz Wetlzar optic microscope, digital micrographs were taken at strategic points near the hardness indentations to evaluate the grain size using the ASTM three circle grain sizing method. The data we collected with these techniques provide maps of the microstructure and strength distributions in the forgings.



The image to the left is a typical location map showing location and orientation of the digital micrographs on the forging. Below is a standard contour map of grain size showing distribution of grain size in the forging.

The image above shows the container before the forging and etching processes. The image to the right shows a forging before hardness testing; the red points represent the locations for hardness indentations and the green points are the locations for grain size determination.



Flaws in Grain Size Determination

$$H_o: \mu_m - \mu_a = 0$$
$$H_a: \mu_m - \mu_a \neq 0$$

$$\alpha = 0.05 \quad n = 87$$
$$\overline{x_m} = 8.596 \quad S_m = 1.150$$
$$\overline{x_a} = 8.873 \quad S_a = 1.025$$

$$t^* = \frac{(\overline{x_m} - \overline{x_a}) - (\mu_m - \mu_a)}{\sqrt{\frac{S_m^2 + S_a^2}{n}}}$$
$$t^* = \frac{(8.596 - 8.873) - (0)}{\sqrt{\frac{1.150^2 + 1.025^2}{87}}}$$
$$t^* = -1.675 \quad df = 169.759$$
$$p(t > 1.675 \cap t < -1.675) = .09571$$

Figure 1

$$H_o: \mu_n - \mu_t = 0$$
$$H_a: \mu_n - \mu_t \neq 0$$

$$\alpha = 0.05 \quad n = 334$$
$$\overline{x_n} = 8.146 \quad S_n = 1.552$$
$$\overline{x_t} = 8.641 \quad S_t = 1.356$$

$$t^* = \frac{(\overline{x_n} - \overline{x_t}) - (\mu_n - \mu_t)}{\sqrt{\frac{S_n^2 + S_t^2}{n}}}$$
$$t^* = \frac{(8.146 - 8.641) - (0)}{\sqrt{\frac{1.552^2 + 1.356^2}{334}}}$$
$$t^* = -4.393 \quad df = 654.210$$
$$p(t > 4.393 \cap t < -4.393) = 1.302 \times 10^{-5}$$

Figure 2

Automated ASTM grain size determinations are influenced by many factor including the quality of the digital images, the metallographic preparation, and the presence of microstructural features other than grain boundaries. To confirm the autmated software routines, manual grain size determinations were compared to the software results. Figure 1 is a significance test (with μ_m being the true mean of manual counts and μ_a being the true mean of automated counts) showing that differences in manual and automated measurements were due primarily to statistical variation. However, with manual counting, we were able to differentiate between twin boundaries and grain boundaries. Because twin boundaries behave differently than grain boundaries under the influence of stress, they could play a key role in the effectiveness of forged stainless steel containers. Figure 2 is a similar test (with μ_n being the true mean of grain size with no twin counts and μ_t being the true mean of grain size with twin counts) showing that twins indeed have a noticeable effect on the grain size determination.

Summary of Results

Austenitic stainless steels can be used to store hydrogen under extremely high pressures, reaching up to 10,000 psi for commercial usage. Therefore, our measurements and calculations need to be as accurate as possible. Although figure 1 proves, at least from a statistical standpoint, that there is no difference between automated grain sizing and manual grain sizing, we have opted to take the more tedious approach for many reasons. First, automated counting is dictated by the conditions. If there were imperfections or if our digital images were taken under unfavorable conditions, the automated count would have a large margin of error; this includes counting dust particles, dents, scratches, or flow lines as grain boundaries as seen in figure 3. Secondly, the software is unable to distinguish between twin boundaries, which result from the forging process, and actual grain boundaries brought out by etching. Even though twin boundaries behave similar to grain boundaries within the realm of our experiment, twinning can be controlled and regulated in the forging steps; this disparity could have a huge impact on the future of this research.

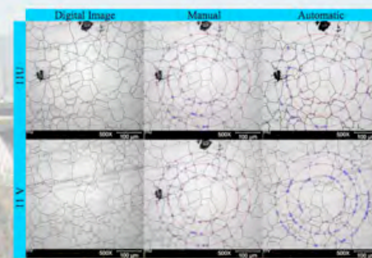


Figure 3

8-12-10-AW-DP-LH-SZ