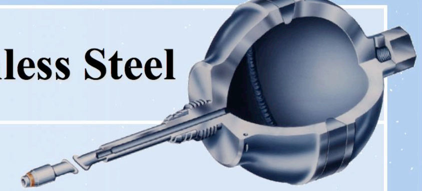
SAND2010-5660P

Evaluation of Microstructural Features in Forged Austenitic Stainless Steel



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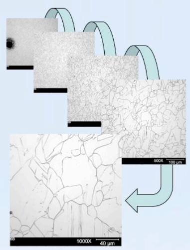
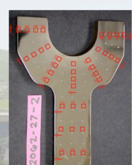
Abstract

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 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.

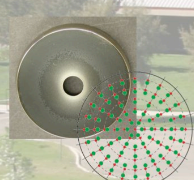
Procedure

All twelve samples (alloy 304L - .03% carbon, 18-20% chromium, 2% manganese, 8-10% nickel, 1% silicon, 67.97-70.97% iron) underwent conventional metallographic polishing and etching using 75% nitric acid at 1.15 volts. Hardness testing was then done under the specifications dictated by Rockwell Hardness Scale A. Using the Leitz Wetlzar optic microscope, we took digital images of the forgings at strategic points near the hardness indentations so that the ASTM three circle grain sizing method could be used to get an accurate representation of grain sizes within the different forgings. The data we collected with these techniques provide models that we used to judge the nuances in hardness and grain size of the forgings as a whole.

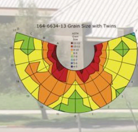




The image above shows the container before the forging and etching processes. The image to the right shows a forging before hardness testing; the red points are where the indentations are going to be and the green points are where digital images will be taken.



The image to the left is a typical location map showing where and the orientation digital images were taken on the actual forging. The graph below is a standard contour map of grain size. It maps out grain size relatively within a forging.



Flaws of Grain Sizing

$H_o : \mu_m - \mu_a = 0$

$H_\alpha : \mu_m - \mu_a \neq 0$

$\alpha = 0.05 \quad n = 87$

$\overline{x_m} = 8.596 \quad S_m = 1.150$

$\overline{x_a} = 8.873 \quad S_a = 1.025$

$$t^* = \frac{(\overline{x_m} - \overline{x_a}) - (\mu_m - \mu_a)}{\sqrt{\frac{S_m^{\,2} + S_a^{\,2}}{n}}}$$

$$t^* = \frac{(8.596 - 8.873) - (0)}{\sqrt{\frac{1.150^2 + 1.025^2}{87}}}$$

$t^* = -1.675 \quad df = 169.759$

$p(t > 1.675 \cap t < -1.675) = .09571$

Figure 1

$H_o : \mu_n - \mu_t = 0$

$H_\alpha : \mu_n - \mu_t \neq 0$

$\alpha = 0.05 \quad n = 334$

$\overline{x_n} = 8.146 \quad S_n = 1.552$

$\overline{x_t} = 8.641 \quad S_t = 1.356$

$$t^* = \frac{(\overline{x_n} - \overline{x_t}) - (\mu_n - \mu_t)}{\sqrt{\frac{S_n^{\,2} + S_t^{\,2}}{n}}}$$

$$t^* = \frac{(8.146 - 8.641) - (0)}{\sqrt{\frac{1.552^2 + 1.356^2}{334}}}$$

$t^* = -4.393 \quad df = 654.210$

$p(t > 4.393 \cap t < -4.393) = 1.302 \times 10^{-5}$

Figure 2

ASTM grain sizing is inherently inaccurate since the computer cannot distinguish the difference between imperfections within the forging and the actual grain boundaries. We were mistrusting of automation, thus we did all the grain size counting manually. Figure 1 is a significance test (with μ_m being the true mean of manual counts and μ_a being the true mean of automatic counts) showing that our worries were actually unsubstantiated, and that there is a good likelihood that the differences seen were due to chance. However, with manual counting, we were able to differentiate between twin lines and grain boundaries. Because twin lines act similar to grain boundaries, but are artificially induced, 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 impact of the size of the grains.

Summary of Results

These austenitic stainless steels are meant 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 automatic grain sizing and manual grain sizing, we have opted to take the more tedious approach for many reasons. First, automatic counting is dictated by the conditions. If there were imperfections within the forging or if our digital images were taken under unfavorable conditions, the automatic 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 program is unable to distinguish between twin lines, which result from the forging process, and actual grain boundaries brought out by etching. Even though twin boundaries act like grain boundaries within the realm of our experiment, twinning can be controlled and regulated in the steps of forging creation; this disparity could have a huge impact on the future of this research.

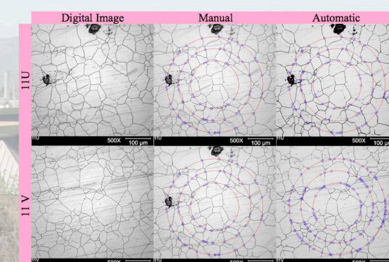


Figure 3

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