



LATROBE SPECIALTY STEEL COMPANY

Challenges of Prior Austenite Grain Size Determination in Vacuum-melted Steels

Grain Size still a hot topic after all these years:

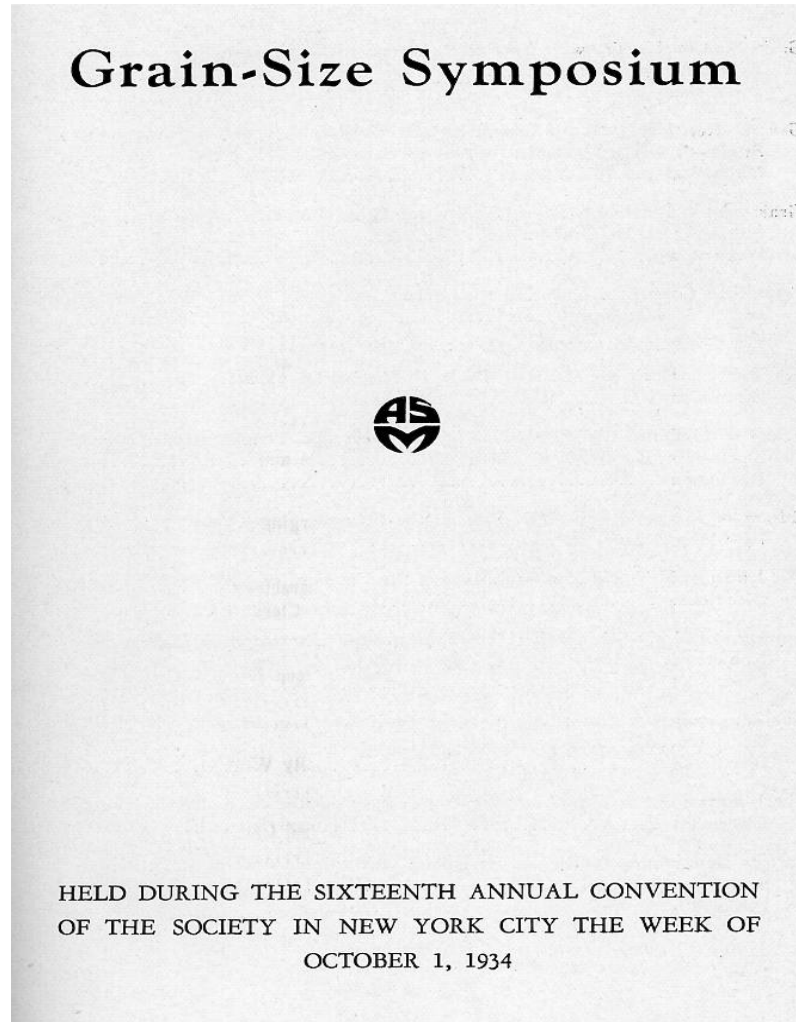


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Marcus Grossman:

- “...grain size relationships...refer to a relationship between the properties of the piece of metal being tested and the size of the crystals which compose the piece. In an entirely different category are the studies of austenite grain-size in steel, as exemplified by the widely used McQuaid-Ehn test. Here the steel is examined not for its actual grain-size while being tested but to discover what its grain-size had been previously, namely when it consisted of crystals of austenite, at high temperature.
- “The size of these austenite grains in any individual piece of steel may be varied in a number of different ways, and so may have little or nothing to do with the size of the grains in the piece after it has cooled and transformed to ferrite.”

E.S. Davenport and E.C. Bain:

- “The grain structure must be well marked out in a specimen at ordinary temperature, and therefore no longer austenitic, which can be examined under the microscope, for clearly it is impracticable to examine the specimen at the heating temperature just prior to cooling when the austenite...is actually established. This is not entirely easy, for in some cases, the structure formed during the transformation occasioned by cooling does not in any way reflect the grain-boundaries of the parent austenite.”
- “In certain slowly-cooled, low carbon steels the ferrite-pearlite patchwork seen in the microscope bears no apparent relation to the pattern of the austenitic grains. Nevertheless, in practically any steel the austenite grain structure can, with some expenditure of effort, be marked out in an unequivocal manner.”

Prior austenite grain boundary delineation

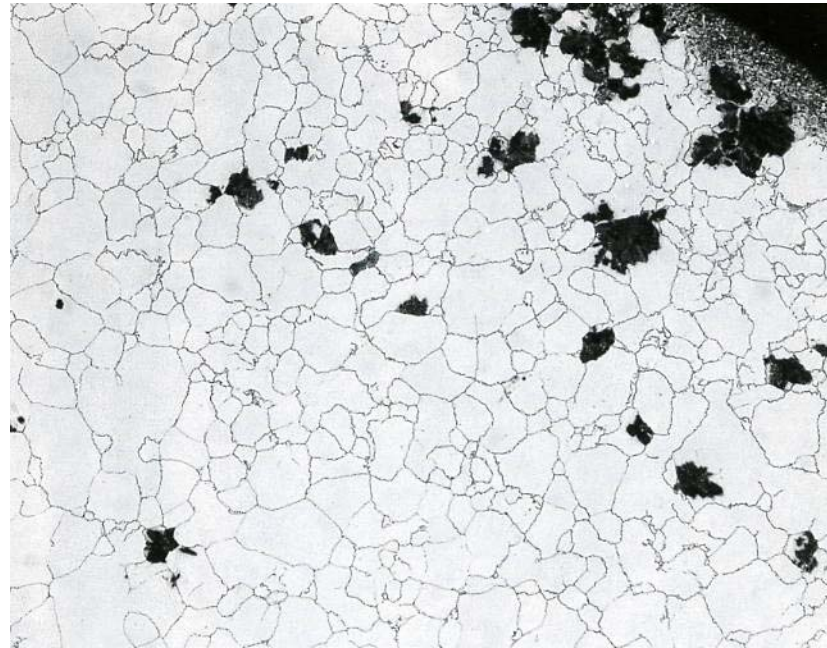
- **Air-melted steels** – typically higher in residual elements such as phosphorus and sulfur, which tend to segregate to grain boundaries in austenite. These segregated boundaries can be revealed after austenitic transformation by suitable etching techniques.
- **Vacuum-melted steels** – typically lower in segregating residual elements than air-melted steels, due to more stringent specifications. Non-segregated prior austenite grain boundaries can leave very little evidence of their former location in transformed room-temperature microstructures. Direct observation of prior austenite grain boundaries in medium-carbon and low-carbon steels can be difficult.

- For many steel grades, good prior austenite grain boundary delineation can be obtained using the carburizing procedure known as the McQuaid-Ehn test for lower-carbon steels, and the Oxidation procedure for medium-carbon steels, as described in the ASTM E 112 standard.
- In higher carbon bearing and tool steels, prior austenite grain boundaries can be readily observed in the transformed room temperature microstructures, usually by etching in nital.

McQuaid-Ehn Test

- Has been in use for at least 90 years. Commonly used on low-carbon steels and carburizing steels.
- Specimens are typically pack-carburized; on cooling, the austenite grain boundaries are outlined by precipitated pro-eutectoid carbides.
- Requires the development of a hypereutectoid carbon level in the carburized case.
- Point to consider: Is the austenitic grain structure and grain-coarsening behavior in the high-carbon case equivalent to that in the lower-carbon core? Is this important?

McQuaid-Ehn Test – 9310 steel:



Oxidation Test

- Per ASTM E 112, specimens are polished on one face, heated in air at 1575F for one hour, then water quenched to room temperature.
- Surface oxide scale is removed by re-polishing, leaving more heavily oxidized prior austenite grain boundaries on the re-polished surface.
- Observation: The heavily-oxidized surface can also heavily decarburized. In most instances, the grain size determination is therefore obtained from a localized region that has a different carbon concentration than the underlying material. Does this matter?

Grain size testing experience with two vacuum-melted steels:

- Nitralloy 135M – nitriding steel

<u>C</u>	<u>Mn</u>	<u>Si</u>	<u>Cr</u>	<u>Mo</u>	<u>Al</u>
.40	.60	.30	1.60	.35	1.20

- Lesco TM 53 – carburizing steel*

<u>C</u>	<u>Mn</u>	<u>Si</u>	<u>Cr</u>	<u>Ni</u>	<u>Mo</u>	<u>V</u>	<u>Cu</u>
.11	.40	.90	1.00	2.00	3.25	.10	2.00

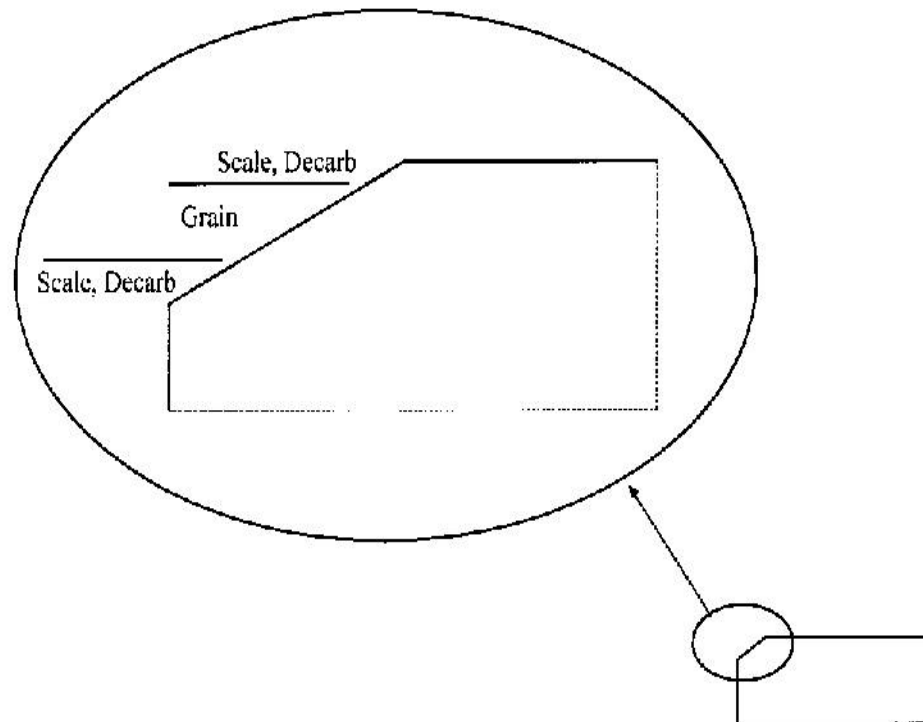
*aka – Pyrowear [®] 53, AMS 6308, X-53

Nitralloy 135M – Oxidation Test Experience

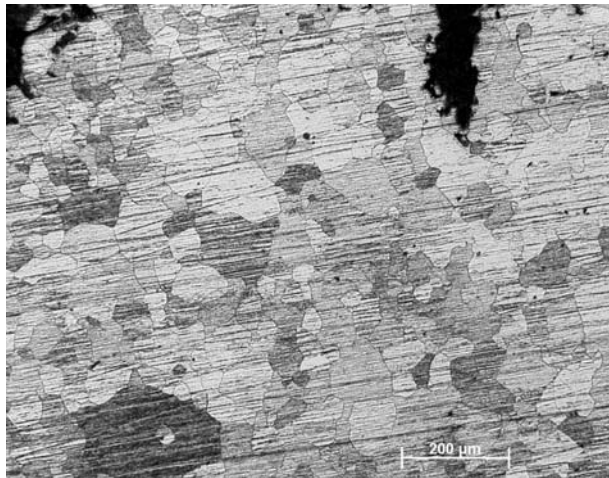
- Difficult to produce a grain size that meets the specification requirement of ASTM #5 by a comfortable margin.
- Alloy decarburizes heavily.
- Prior austenite grain size appears finer in interior regions compared with decarburized surface region.
- Polishing technique can affect grain size results.
- Starting microstructure/heat treat condition can affect grain size results.

Polishing techniques

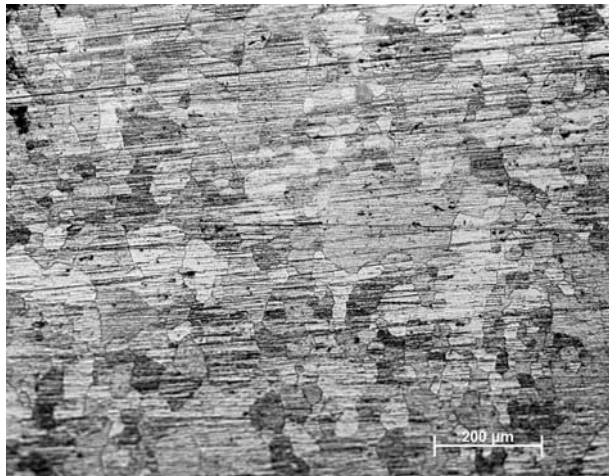
- ASTM E 112 recommends re-polishing the oxidized face of the pre-polished specimen.
- Latrobe Steel has, for many years, used an alternative polishing technique, which we call “feather” polishing:



Effect of oxidizing time - Trials were run using different times at 1575F to see if this could affect the oxidized grain size. A strong trend was not observed.



- Specimen oxidized for 60 minutes at 1575F. Feather-polished, nital etched, 100X magnification.



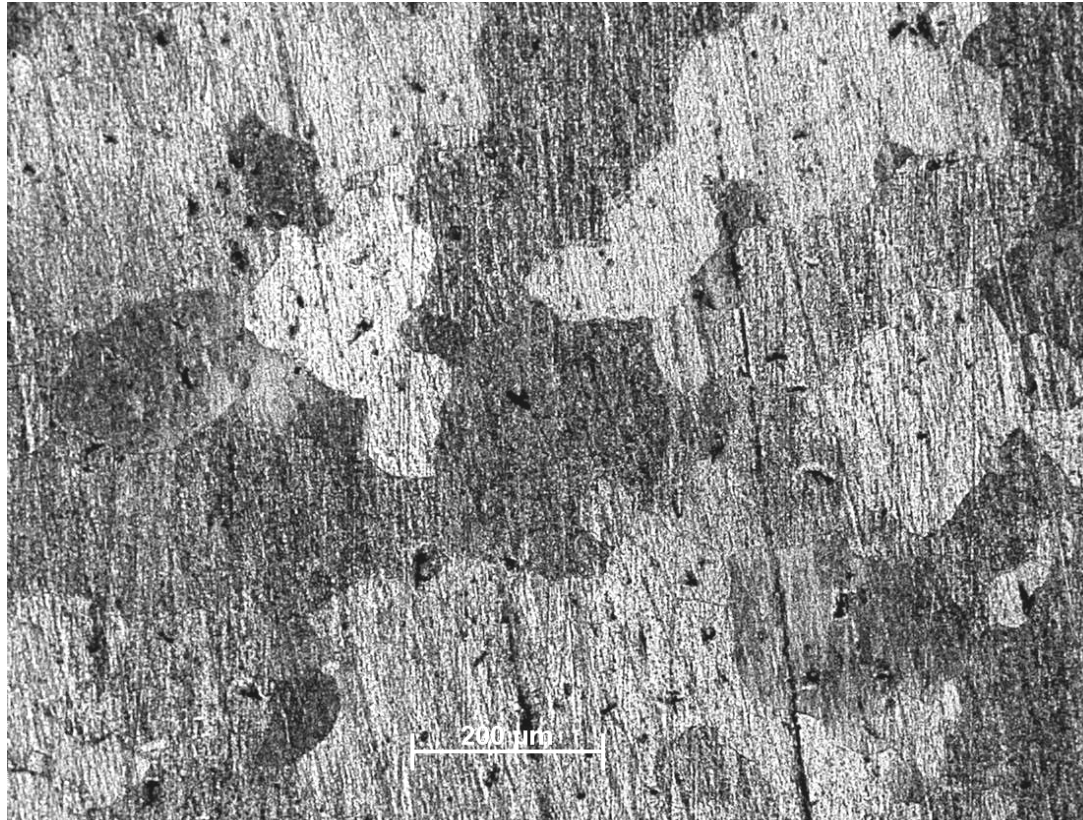
- Specimen oxidized for 15 minutes at 1575F. Feather-polished, nital etched, 100X magnification.

Decarburization effect



- Specimen oxidized for 15 minutes at 1575F. Plane of polish is perpendicular to surface. Nital etched.

Effect of polishing technique:

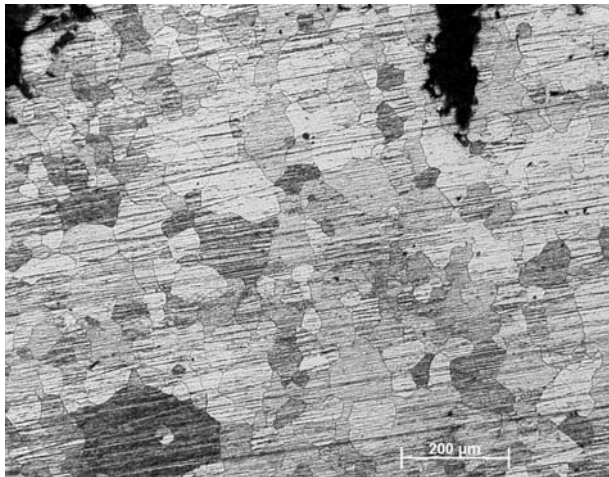


- Pre-polished specimen oxidized for 15 minutes at 1575F. Re-polished flat, nital etched. Plane of polish is within heavily decarburized near-surface region.

Attempt to reduce decarburization:

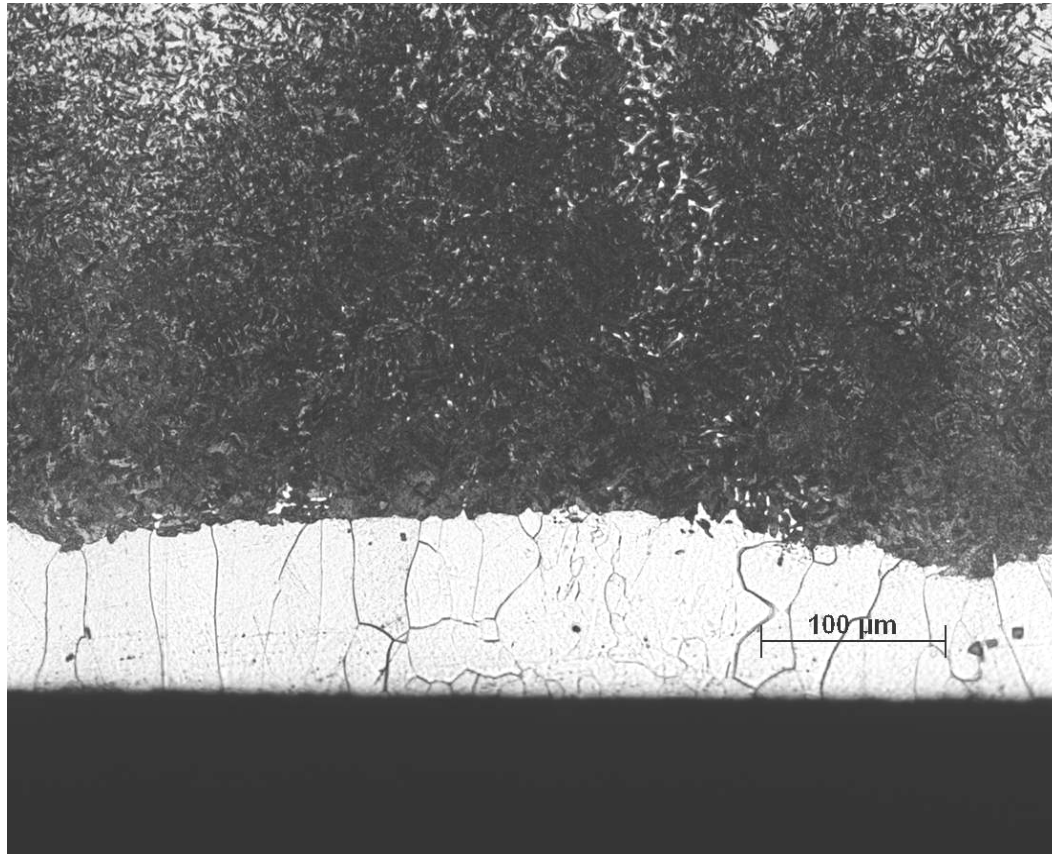


- Specimen coated with alumina-based slurry intended to reduce decarburization and scaling. Oxidized at 1575F for 60 minutes.



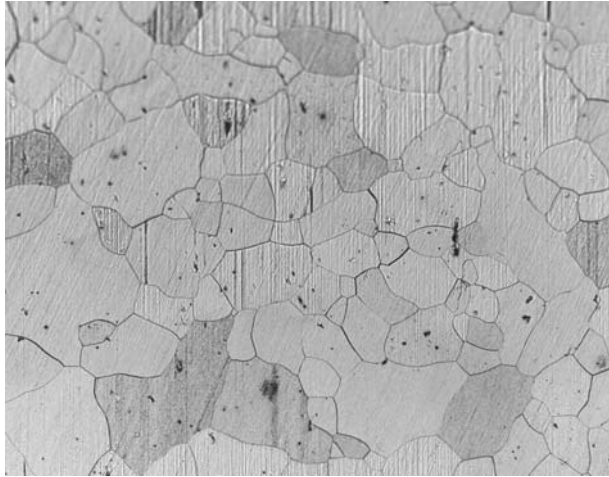
- Specimen oxidized at 1575F for 60 minutes, without alumina coating.

Slurry-coated specimen

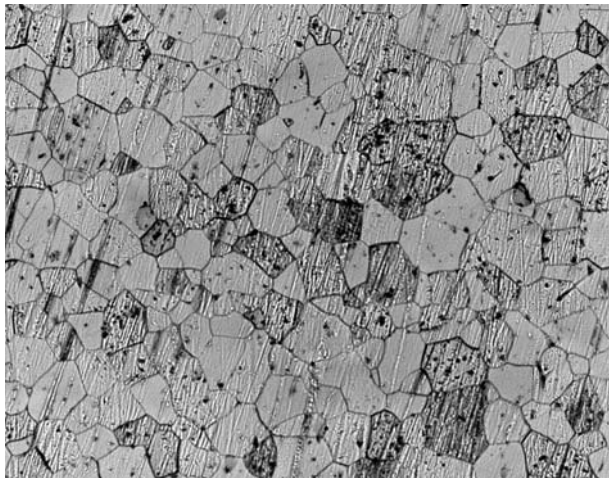


- Specimen coated with alumina slurry and oxidized for 60 minutes at 1575F. Plane of polish is perpendicular to surface. Nital etched.

Effect of starting microstructure/heat treat condition - non-annealed versus annealed:

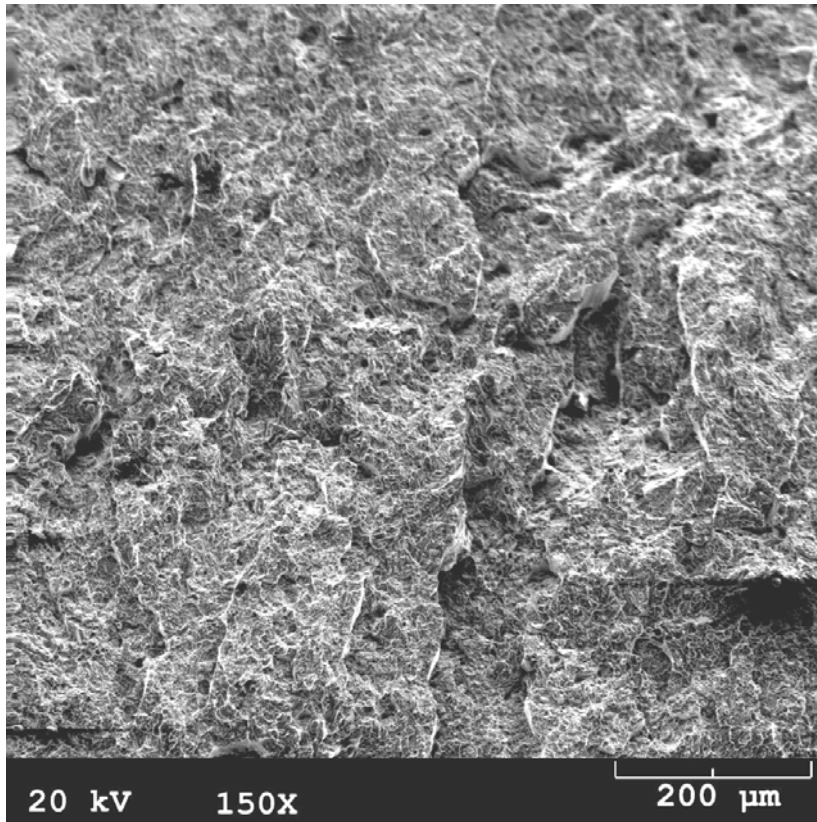


- Oxidized grain structure of non-annealed Nitralloy 135M billet material. Feather-polished, nital etched.



- Same billet material as above, but annealed prior to oxidation heat treatment. Feather-polished, nital etched.

Shepherd fracture grain size – Nitralloy 135M:



- Specimen hardened from 1575F, tempered at 400F for 2 hours, cooled in liquid nitrogen and fractured.
- Estimated grain size:
ASTM #2 – #6 (duplex).

Grain size testing of Lesco 53:

- McQuaid-Ehn carburizing test results: Experience to date has generally not been too successful in revealing prior austenite grain boundaries in carburized case region, regardless of etching technique.
- Better results have been obtained in revealing grain boundaries in the non-carburized interior regions.
- More work is needed to develop a robust, universally acceptable method of determining the grain size in this steel. Use of this alloy is increasing, and it will likely replace carburized 9310 in many aircraft drivetrain applications in the future.

McQuaid-Ehn test results:

- Grain boundaries are rarely visible in the carburized case region.
- Hypothesis was that cooling conditions may affect the precipitation of pro-eutectoid carbides at austenite grain boundaries. To investigate this, a series of thermal treatments was planned.
- It was then discovered that the case carbon content of pack-carburized specimens was surprisingly low. OES analysis indicated that carbon levels in the case were in the range of 0.20% to 0.35%.
- The lack of pro-eutectoid grain boundary carbide precipitation is therefore no longer a mystery.

Lesco 53:

- Due to the lack of success in revealing grain boundaries in the case region, the focus has shifted to delineating grain boundaries in the non-carburized core region.

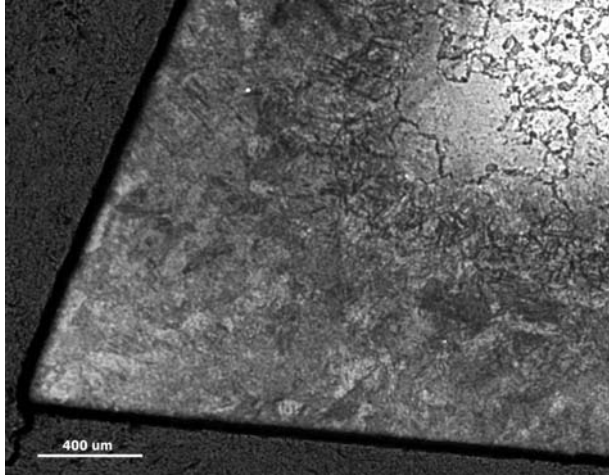
- Need to decorate austenite grain boundaries with an austenite decomposition product that can be revealed by etching.
 - Thermal treatment development.
 - Etching technique development.

- Several labs have been involved with this effort, with varying amounts of success.

Current Latrobe Steel procedure:

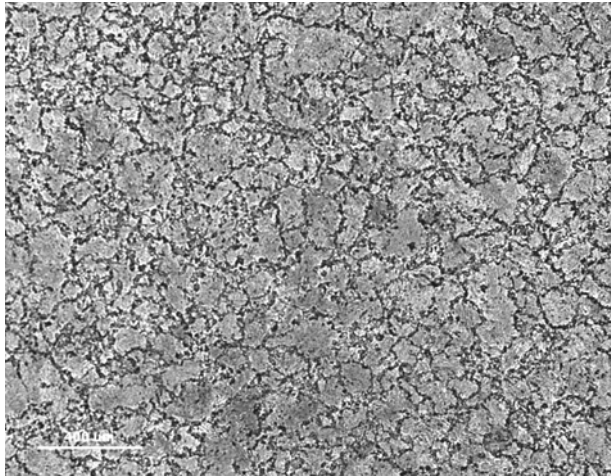
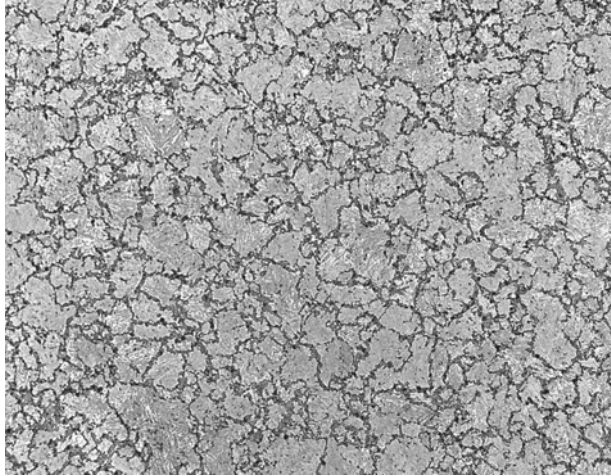
- Pack-carburize in charcoal at 1700F for 4 hours, transfer to furnace at 1300F, hold for 1 hour at temperature, air cool to room temperature.
- Rough grind, polish through 3 micron diamond.
- Etch with alcohol solution of 9% HCL, 3% HNO₃, + a few drops of H₂O₂. Submerge for ~10 minutes until very dark.
- Lightly re-polish by hand with 3 micron diamond to partially remove etch.
- Etch with 4% nital.

Latrobe Steel results:



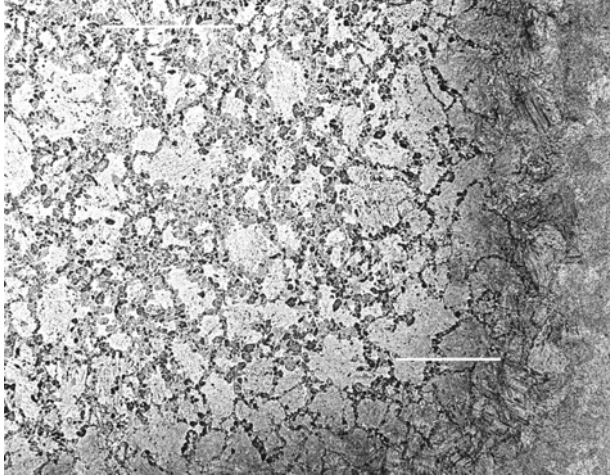
- Carburized case regions show little or no evidence of grain boundary delineation.

Latrobe Steel results:

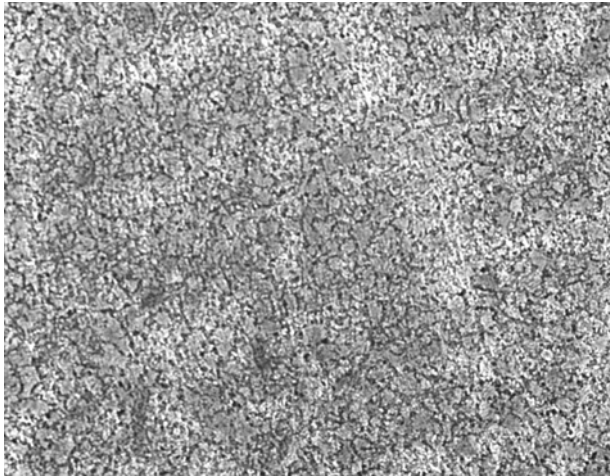


- Non-carburized core regions show better grain boundary delineation. These images represent our best efforts to date.

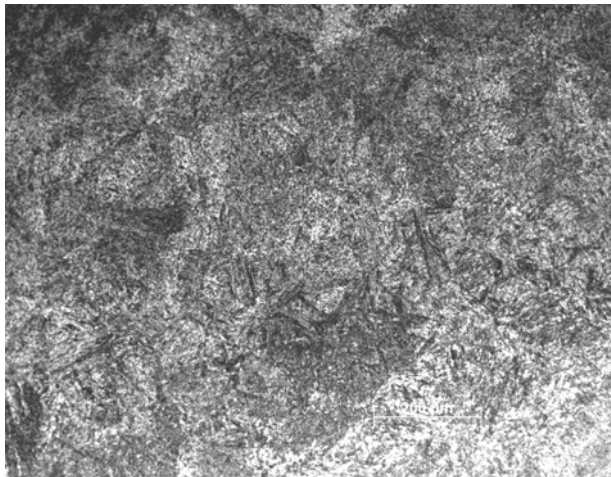
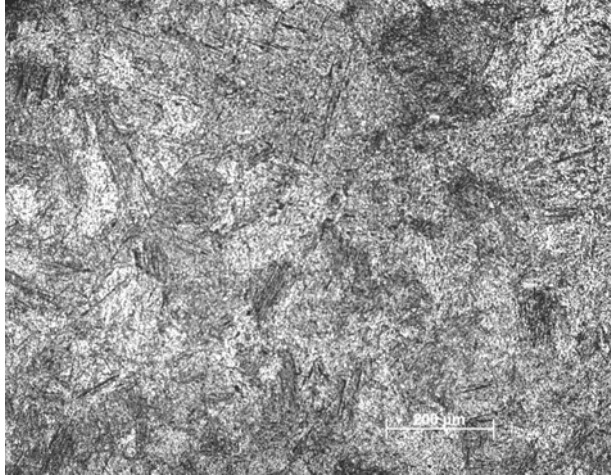
Latrobe Steel results:



- We also get images like these.

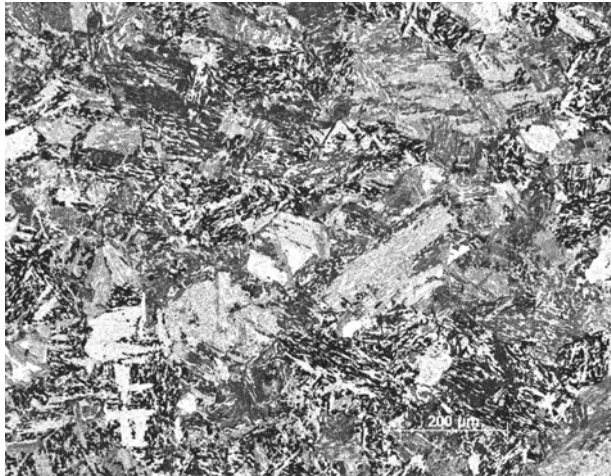
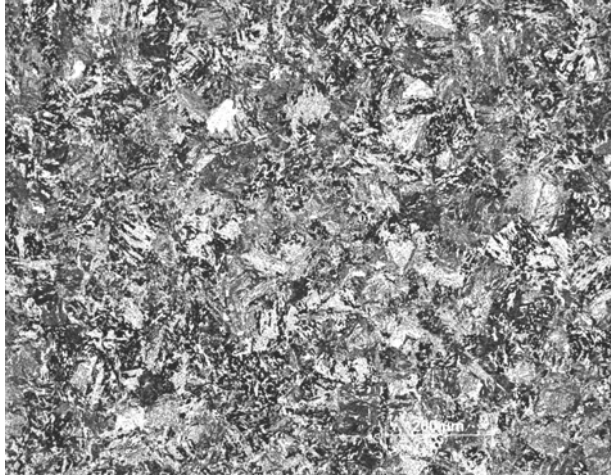


Latrobe Steel results:



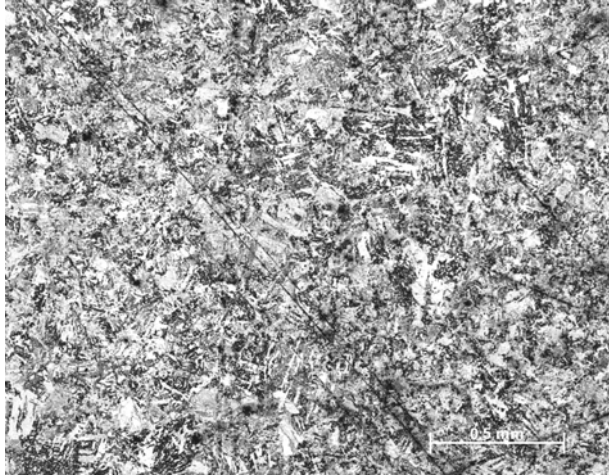
- Or even like these, unfortunately.

Results from Laboratory “X”



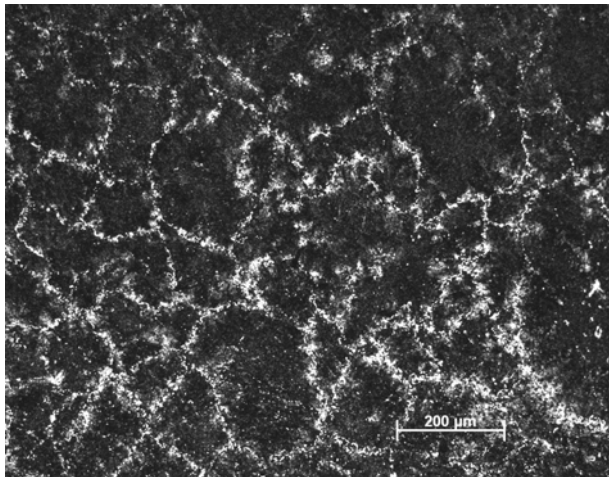
- Round robin test results - specimens were prepared by another lab, as follows:
 - Condition – “as-received,” not carburized.
 - Swabbed in Vilella’s reagent for 5 seconds, followed by 3 second swab in Stainless #2 etch.
- Vilella’s: 1 g picric acid, ml HCl, 100 ml methanol.
- Stainless #2: 50 g FeCl, 50 ml HCl, 100 ml methanol.

Results from Laboratories “Y” and “Z”:



Thermal treatment and etching procedures were not provided.

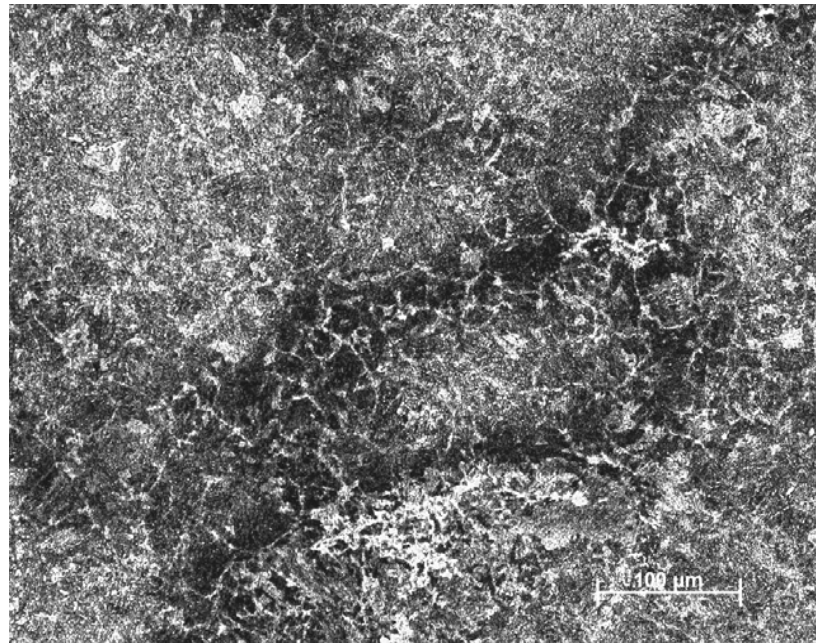
- Lab “Y”



- Lab “Z”

Recent Latrobe result:

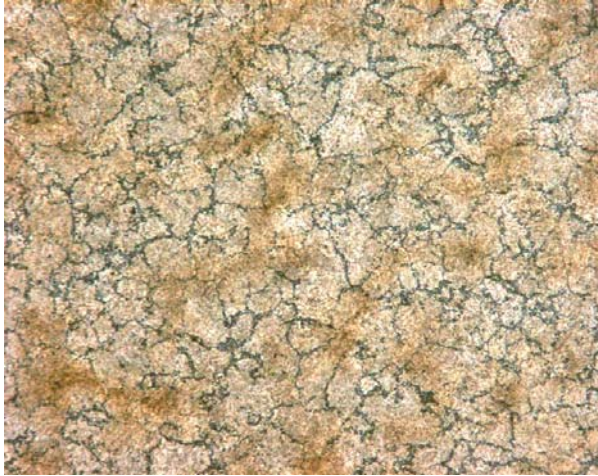
- Goal is to outline prior austenite grain boundaries with ferrite. Heat treatment trials involved holding at 1675F for one hour, cooling to 1300F and holding at 1300F for various times to obtain ferrite transformation, followed by air cooling to room temperature.
- Specimen below was held at 1300F for 1 hour. A second phase, likely ferrite, is visible at grain boundaries. Shorter holding times at 1300F did not produce the grain boundary phase.



George Vander Voort's work on Lesco 53:

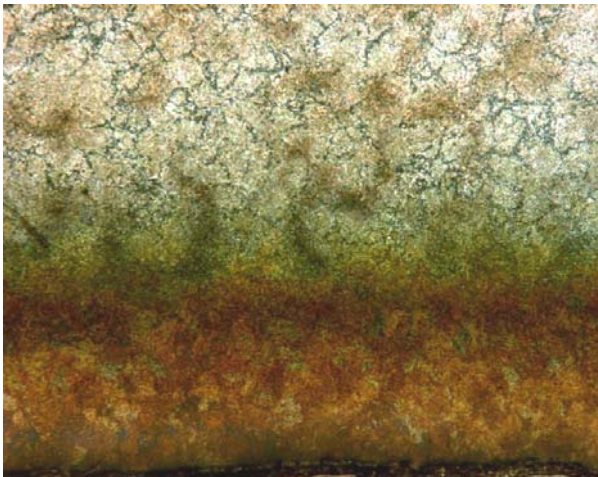
- Latrobe Steel submitted pack-carburized specimens to George for grain size determination.
- Promising results obtained by electrolytic etching.

George Vander Voort's results on Lesco 53:



- Etchant: 10% Ammonium persulfate. 6 V dc, 10 seconds.

- Interior



- Case/interior

George Vander Voort's results on Lesco 53:



- Etchant: 10% Ammonium persulfate. 6 V dc, 10 seconds.

George Vander Voort's results on Lesco 53:



- Etchant: Lucas' reagent.
 - 50 ml Lactic acid
 - 150 ml HCl
 - 3 g Oxalic acid
- 1.6 V dc, 8 seconds.

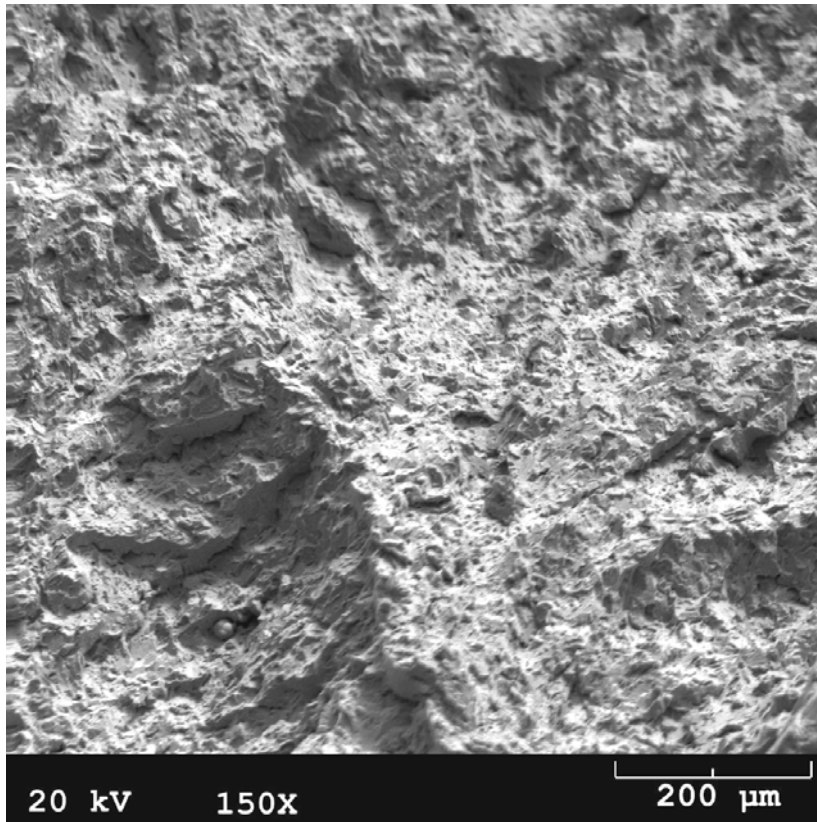
George Vander Voort's work on Lesco 53:



- Etchant: Lucas' reagent.
 - 50 ml Lactic acid
 - 150 ml HCl
 - 3 g Oxalic acid

- 1.6 V dc, 8 seconds.

Shepherd fracture grain size – Lesco 53:



- Specimen hardened from 1675F, tempered at 500F for 2 hours, cooled in liquid nitrogen and fractured.
- Estimated grain size:
ASTM #5.5 – #6.

Some things to consider:

- **Dirty steel can be the metallographer's friend when it comes to revealing prior austenite grain boundaries.**
- **Room temperature, transformed microstructures can bear little resemblance to the parent austenite grain structures.**
- **Heavily decarburized, oxidized surface regions may show evidence of coarser prior austenite grain size than the interior regions.**

More things to consider:

- **Success with McQuaid-Ehn testing requires sufficient carbon content in the carburized case.**
- **Different steel grades react differently to the various grain size test methods. Customized approaches based on steel grade are necessary.**
- **Shepherd fracture grain size test may not be all that bad!**